NONDESTRUCTIVE TESTING HANDBOOK

Third Edition

Volume 1

Leak Testing

Technical Editors
Charles N. Jackson, Jr.
Charles N. Sherlock

Editor
Patrick O. Moore

American Society for Nondestructive Testing

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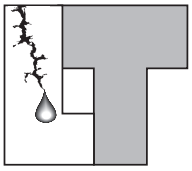

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Library of Congress Cataloging-in-Publication Data

Leak Testing / technical editors, Charles N. Jackson, Jr., Charles N. Sherlock ; editor, Patrick O. Moore. -- 3rd ed.
p. cm. — (Nondestructive testing handbook ; v. 1)
Includes bibliographic references and index.
ISBN-13 978-1-57117-071-2
ISBN-10 1-57117-071-5
1. Leak detectors. 2. Gas leakage. I. Jackson, Charles N. II. Sherlock, Charles N. III. Moore, Patrick O. IV. American Society for Nondestructive Testing. V. Series: Nondestructive testing handbook (3rd ed.) ; v. 1.
TA165.L34 1998 98-10437
620.1'127--dc21 CIP

ISBN-13: 978-1-57117-071-2 (print)
ISBN-13: 978-1-57117-038-5 (CD)
ISBN-13: 978-1-57117-289-1 (ebook)

Errata

You can check for errata for this and other ASNT publications at

<https://www.asnt.org/errata>.

First printing 05/98
Second printing with revisions 12/04
Third printing 09/07
Fourth printing 03/11
ebook 07/13

Published by the American Society for Nondestructive Testing
PRINTED IN THE UNITED STATES OF AMERICA

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In memory of

Charles N. Sherlock

(1932–1997)

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President's Foreword

This book is the first volume of the third edition of the *Nondestructive Testing Handbook*. The existence of books such as *Leak Testing* is testimony to the dedication of the American Society for Nondestructive Testing (ASNT) to its missions of providing technical information and instructional materials and of promoting nondestructive testing technology as a profession. The series documents advances in the various nondestructive testing methods and provides reference materials for nondestructive testing educators and practitioners in the field. ASNT's hope is that the third edition will build on the successes of the past and surpass them by providing current information about our rapidly evolving technology.

Leak Testing was written and reviewed under the guidance of ASNT's Handbook Development Committee. The collaboration between the volunteers and staff in the this volume has made productive use of ASNT's volunteer resources. Scores of authors and reviewers have donated thousands of hours to this volume. A special note of thanks is extended to Handbook Development Director Gary Workman, to Leak Testing Committee Chair Gary Elder, to Technical Editors Charles Sherlock and Charles Jackson, to Handbook Coordinators John Keve and Stuart Tison and to Handbook Editor Patrick Moore for their dedicated efforts and commitment in providing this significant book.

Hussein M. Sadek
ASNT National President (1997–98)

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Foreword

The Aims of a Handbook

The volume you are holding in your hand is the first in the third edition of the *Nondestructive Testing Handbook*. Now, with the beginning of a new series, is a good time to reflect on the purposes and nature of a handbook.

Handbooks exist in many disciplines of science and technology, and certain features set them apart from other reference works. A handbook should ideally give the basic knowledge necessary for an understanding of the technology, including both scientific principles and means of application.

The typical reader may be assumed to have completed three years of college toward a degree in mechanical engineering or materials science and hence has the background of an elementary physics or mechanics course. Occasionally an engineer may be frustrated by the difficulty of the discussion in a handbook. That happens because the assumptions about the reader vary according to the subject in any given section. Computer science requires a different sort of background from nuclear physics, for example, and it is not possible for the handbook to give all the background knowledge that is ancillary to nondestructive testing.

A handbook offers a view of its subject at a certain period in time. Even before it is published, it starts to get obsolete. The authors and editors do their best to be current but the technology will continue to change even as the book goes to press.

Standards, specifications, recommended practices and inspection procedures may be discussed in a handbook for instructional purposes, but at a level of generalization that is illustrative rather than comprehensive. Standards writing bodies take great pains to ensure that their documents are definitive in wording and technical accuracy. People writing contracts or procedures should consult real standards when appropriate.

Those who design qualifying examinations or study for them draw on handbooks as a quick and convenient way of approximating the body of knowledge. Committees and individuals who write or anticipate questions are selective in what they draw from any source. The parts of a handbook that give scientific background, for instance, may have little bearing on a practical examination. Other parts of a handbook are specific to a certain industry. Although a handbook does not pretend to offer a complete treatment of its subject, its value and convenience are not to be denied.

The present volume is a worthy beginning for the third edition. The editors, technical editors and many contributors and reviewers worked together to bring the project to completion. For their scholarship and dedication I thank them all.

Gary L. Workman
Handbook Development Director

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Preface

Unfortunately, too many people still have the impression that leak testing involves little more than finding a hole in a flat tire. The development of the helium mass spectrometer in the days of the Manhattan Project during the 1940s was the initial quantum leap in leak testing. With miniaturization and technological advances in electronics and hardware, leak testing has grown into a technology of great sophistication.

In 1982, the American Society for Nondestructive Testing (ASNT) published *Leak Testing,* the first volume of the second edition *Nondestructive Testing Handbook*. Since then, 3000 copies of that book have been sold, providing many leak testing personnel, both technicians and managers, with a ready source of reference information.

In May 1990, to determine the general location of apparent leakage, the National Aeronautics and Space Administration had to develop a combination of remote hydrogen sensors and a multiple channel mass spectrometer connected to a computer for numeric readouts during liquid hydrogen fueling. This illustrates the versatility of the mass spectrometer and also points out the need for more research and development to improve leak testing monitoring systems.

It is good to have aspirations about space travel, but the pressing reality of the moment is the environmental damage we continue to inflict on our space home, Earth. We are rapidly destroying the environment in which we live through contamination of the air we breathe, the water we drink and the soil in which we grow our food.

One of the problems today is the many storage tanks and ponds that have been leaking contaminants (all sorts of petrochemical and petroleum products) into the ground for years with no effective continuous leakage monitoring. Many of these structures were not adequately leak tested at the time they were fabricated and, until recently, were not closely monitored for leakage that passed into the ground, contaminating the soil and water supply.

What does leak testing have to do with all of this? It is the one nondestructive testing method that can be used to determine the total leakage rate (quantity or mass) of undesirable products escaping from their containers into the environment. A combination of pressure change and mass flow in one form or another has been used for this purpose for many decades. A good example is the integrated leakage rate testing of nuclear containment systems. The existence of these containment systems and the tests that proved their total leakage to be within acceptable limits helped reduce the environmental damage from the incident at Three Mile Island. Without these safeguards, that incident would have been an environmental catastrophe such as occurred at Chernobyl in the Ukraine.

Many combinations of volume change, tracer gas testing with detector probes, liquid displacement, ultrasound etc. are used to test storage tanks. Needed now are quantitative test techniques sensitive enough to detect all fluid leakage and yet reasonably economical for construction of tank configurations and products. It is time for development of better leak testing systems and procedures for these structures.

More training, qualification and certification for leak testing personnel will be implemented when management realizes that nondestructive testing can save money and when codes and standards include such requirements. The impetus to make it happen will have to come from the nondestructive testing community and organizations like ASNT.

The Technical Editors would like to thank all the ASNT staff and volunteers — contributors, reviewers and committee members — who made this book possible.

Charles N. Jackson, Jr.
Charles N. Sherlock

Technical Editors

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Editor's Preface

The third edition of the *Nondestructive Testing Handbook* begins as the second edition did, with the volume *Leak Testing*. This third edition volume is indebted to the preceding edition's volume in many ways. Much of the text is the same, despite significant additions and alterations.

Published in 1959 by the American Society for Nondestructive Testing (ASNT), the first edition of the *Nondestructive Testing Handbook* did not cover leak testing at all. In 1982, the second edition's *Leak Testing* volume was groundbreaking. Aside from the *Leakage Testing Handbook* (1968), written by J.W. Marr for the National Aeronautics and Space Administration, there had been no comprehensive books on the subject. Although parts of *Leak Testing* drew on Marr's work, on standards published by sister societies and on literature provided by equipment manufacturers, *Leak Testing* was a highly original contribution to technical literature. For this reason, the second edition *Leak Testing* contained very few references to other publications.

The technical content of this third edition volume differs in several ways from that of the second. (1) New technology is represented, including infrared thermography and counterflow mass spectrometry. (2) Pages have been added to cover new applications, such as the inspection of storage tanks. (3) The text reflects the fact that, for reasons of environment, fluorocarbon tracer gases have been regulated. (4) A comprehensive glossary is provided. (5) An extensive bibliography lists leak testing publications, more than some leak testing practitioners might have expected.

The greatest setback during the preparation of this volume was the death in February 1997 of Technical Editor Charles Sherlock. He contributed many pages to this volume and edited the first half through the galley stage. His good humor and willingness to give freely of his time and knowledge endeared him to many ASNT members. The technical community will continue to miss him for many years.

After his passing, the task of editing for technical accuracy was undertaken by Charles Jackson. ASNT is very fortunate that he was willing to devote his technical expertise to this project.

ASNT is likewise indebted to Handbook Coordinators Stuart Tison and John Keve and to the technical experts listed at the end of this foreword. (Please note that people listed as contributors were also reviewers but are listed only once, as contributors.)

It is difficult to overstate the contributions of staff members Hollis Humphries-Black and Joy Grimm to the art, layout and text of the book. I would also like to thank Publications Manager Paul McIntire for his support during design and production.

Patrick O. Moore
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CHAPTER 1

Introduction to Leak Testing

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PART 1. Nondestructive Testing

Nondestructive testing (NDT) has been defined as comprising those test methods used to examine or inspect a part or material or system without impairing its future usefulness.[1] The term is generally applied to nonmedical investigations of material integrity.

Strictly speaking, this definition of nondestructive testing includes noninvasive medical diagnostics. X-rays, ultrasound and endoscopes are used by both medical and industrial nondestructive testing. Medical nondestructive testing, however, has come to be treated by a body of learning so separate from industrial nondestructive testing that today most physicians never use the word *nondestructive*.

Nondestructive testing is used to investigate specifically the material integrity of the test object. A number of other technologies — for instance, radio astronomy, voltage and amperage measurement and rheometry (flow measurement) — are nondestructive but are not used specifically to evaluate material properties. Radar and sonar are classified as nondestructive testing when used to inspect dams, for instance, but not when they are used to chart a river bottom.

Nondestructive testing asks "Is there something wrong with this material?" Various performance and proof tests, in contrast, ask "Does this component work?" This is the reason that it is not considered nondestructive testing when an inspector checks a circuit by running electric current through it. Hydrostatic pressure testing is another form of proof testing and may destroy the test object.

Another gray area that invites various interpretations in defining nondestructive testing is *future usefulness*. Some material investigations involve taking a sample of the inspected part for testing that is inherently destructive. A noncritical part of a pressure vessel may be scraped or shaved to get a sample for electron microscopy, for example. Although future usefulness of the vessel is not impaired by the loss of material, the procedure is inherently destructive and the shaving itself — in one sense the true "test object" — has been removed from service permanently.

The idea of future usefulness is relevant to the quality control practice of sampling. Sampling (that is, the use of less than 100 percent inspection to draw inferences about the unsampled lots) *is* nondestructive testing if the tested sample is returned to service. If the steel is tested to verify the alloy in some bolts that can then be returned to service, then the test is nondestructive. In contrast, even if spectroscopy used in the chemical testing of many fluids is inherently nondestructive, the testing is destructive if the samples are poured down the drain after testing.

Nondestructive testing is not confined to crack detection. Other discontinuities include porosity, wall thinning from corrosion and many sorts of disbonds. Nondestructive material characterization is a growing field concerned with material properties including material identification and microstructural characteristics — such as resin curing, case hardening and stress — that have a direct influence on the service life of the test object.

Nondestructive testing has also been defined by listing or classifying the various methods.[1-3] This approach is practical in that it typically highlights methods in use by industry.

Purposes of Nondestructive Testing

Since the 1920s, the art of testing without destroying the test object has developed from a laboratory curiosity to an indispensable tool of production. No longer is visual examination of materials, parts and complete products the principal means of determining adequate quality. Nondestructive tests in great variety are in worldwide use to detect variations in structure, minute changes in surface finish, the presence of cracks or other physical discontinuities, to measure the thickness of materials and coatings and to determine other characteristics of industrial products. Scientists and engineers of many countries have contributed greatly to nondestructive test development and applications.

The various nondestructive testing methods are covered in detail in the literature but it is always wise to consider objectives before plunging into the details of a method. What is the use of nondestructive testing? Why do

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thousands of industrial concerns buy the testing equipment, pay the subsequent operating costs of the testing and even reshape manufacturing processes to fit the needs and findings of nondestructive testing?

Modern nondestructive tests are used by manufacturers (1) to ensure product integrity and, in turn, reliability; (2) to avoid failures, prevent accidents and save human life; (3) to make a profit for the user; (4) to ensure customer satisfaction and maintain the manufacturer's reputation; (5) to aid in better product design; (6) to control manufacturing processes; (7) to lower manufacturing costs; (8) to maintain uniform quality level; and (9) to ensure operational readiness.

These reasons for widespread profitable use of nondestructive testing are sufficient in themselves, but parallel developments have contributed to its growth and acceptance.

Increased Demand on Machines

In the interest of greater speed and rising costs of materials, the design engineer is always under pressure to reduce weight. This can sometimes be done by substituting aluminum or magnesium alloys for steel or iron, but such light alloy parts are not of the same size or design as those they replace. The tendency is also to reduce the size. These pressures on the designer have subjected parts of all sorts to increased stress levels. Even such commonplace objects as sewing machines, sauce pans and luggage are also lighter and more heavily loaded than ever before. The stress to be supported is seldom static. It often fluctuates and reverses at low or high frequencies. Frequency of stress reversals increases with the speeds of modern machines and thus parts tend to fatigue and fail more rapidly.

Another cause of increased stress on modern products is a reduction in the safety factor. An engineer designs with certain known loads in mind. On the supposition that materials and workmanship are never perfect, a safety factor of 2, 3, 5 or 10 is applied. Because of other considerations though, a lower factor is often used, depending on the importance of lighter weight or reduced cost or risk to consumer.

New demands on machinery have also stimulated the development and use of new materials whose operating characteristics and performance are not completely known. These new materials create greater and potentially dangerous problems. As an example, there is a record of an aircraft's being built from an alloy whose work hardening, notch resistance and fatigue life were not well known. After relatively short periods of service some of these aircraft suffered disastrous failures. Sufficient and proper nondestructive tests could have saved many lives.

As technology improves and as service requirements increase, machines are subjected to greater variations and to wider extremes of all kinds of stress, creating an increasing demand for stronger materials.

Engineering Demands for Sounder Materials

Another justification for the use of nondestructive tests is the designer's demand for sounder materials. As size and weight decrease and the factor of safety is lowered, more and more emphasis is placed on better raw material control and higher quality of materials, manufacturing processes and workmanship.

An interesting fact is that a producer of raw material or of a finished product frequently does not improve quality or performance until that improvement is demanded by the customer. The pressure of the customer is transferred to implementation of improved design or manufacturing. Nondestructive testing is frequently called on to deliver this new quality level.

Public Demands for Greater Safety

The demands and expectations of the public for greater safety are apparent everywhere. Review the record of the courts in granting higher and higher awards to injured persons. Consider the outcry for greater automobile safety, as evidenced by the required use of auto safety belts and the demand for air bags, blowout proof tires and antilock braking systems. The publicly supported activities of the National Safety Council, Underwriters Laboratories, the Environmental Protection Agency and the Federal Aviation Administration in the United States, and the work of similar agencies abroad, are only a few of the ways in which this demand for safety is expressed. It has been expressed directly by the many passengers who cancel reservations immediately following a serious aircraft accident. This demand for personal safety has been another strong force in the development of nondestructive tests.

Rising Costs of Failure

Aside from awards to the injured or to estates of the deceased and aside from costs to the public (e.g. evacuation due to chemical leaks), consider briefly other

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factors in the rising costs of mechanical failure. These costs are increasing for many reasons. Some important ones are (1) greater costs of materials and labor; (2) greater costs of complex parts; (3) greater costs due to the complexity of assemblies; (4) greater probability that failure of one part will cause failure of others due to overloads; (5) trend to lower factors of safety; (6) probability that the failure of one part will damage other parts of high value; and (7) part failure in an automatic production machine, shutting down an entire high speed, integrated, production line. When production was carried out on many separate machines, the broken one could be bypassed until repaired. Today, one machine is tied into the production of several others. Loss of such production is one of the greatest losses resulting from part failure.

Applications of Nondestructive Testing

Nondestructive testing is a branch of the materials sciences that is concerned with all aspects of the uniformity, quality and serviceability of materials and structures. The science of nondestructive testing incorporates all the technology for detection and measurement of significant properties, including discontinuities, in items ranging from research specimens to finished hardware and products. By definition, nondestructive techniques are the means by which materials and structures may be inspected without disruption or impairment of serviceability. Using nondestructive testing, internal properties of hidden discontinuities are revealed or inferred by appropriate techniques.

Nondestructive testing is becoming an increasingly vital factor in the effective conduct of research, development, design and manufacturing programs. Only with appropriate use of nondestructive testing techniques can the benefits of advanced materials science be fully realized. However, the information required for appreciating the broad scope of nondestructive testing is available in many publications and reports.

Classification of Methods

In a report, the National Materials Advisory Board (NMAB) Ad Hoc Committee on Nondestructive Evaluation adopted a system that classified methods into six major categories: visual, penetrating radiation, magnetic-electrical, mechanical vibration, thermal and chemical-electrochemical.[3] Each method can be completely characterized in terms of five principal factors: (1) energy source or medium used to probe object (such as X-rays, ultrasonic waves or thermal radiation); (2) nature of the signals, image and/or signature resulting from interaction with the object (attenuation of X-rays or reflection of ultrasound, for example); (3) means of detecting or sensing resultant signals (photoemulsion, piezoelectric crystal or inductance coil); (4) method of indicating and/or recording signals (meter deflection, oscilloscope trace or radiograph); and (5) basis for interpreting the results (direct or indirect indication, qualitative or quantitative and pertinent dependencies).

The objective of each method is to provide information about the following material parameters:

1. discontinuities and separations (cracks, voids, inclusions, delaminations etc.);
2. structure or malstructure (crystalline structure, grain size, segregation, misalignment etc.);
3. dimensions and metrology (thickness, diameter, gap size, discontinuity size etc.);
4. physical and mechanical properties (reflectivity, conductivity, elastic modulus, sonic velocity etc.);
5. composition and chemical analysis (alloy identification, impurities, elemental distributions etc.);
6. stress and dynamic response (residual stress, crack growth, wear, vibration etc.); and
7. signature analysis (image content, frequency spectrum, field configuration etc.).

Terms used in this block are defined in Table 1 with respect to specific objectives and specific attributes to be measured, detected and defined.

The limitations of a method include conditions required by that method: conditions to be met for technique application (access, physical contact, preparation etc.) and requirements to adapt the probe or probe medium to the object examined. Other factors limit the detection and/or characterization of discontinuities, properties and other attributes and limit interpretation of signals and/or images generated.

Classification Relative to Test Object

Nondestructive testing methods may be classified according to how they detect indications relative to the surface of a test object. Surface methods include liquid penetrant testing, visual testing, grid and moiré testing, holography and shearography. Surface/near-surface methods include tap, potential drop,

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magnetic particle and electromagnetic testing. When surface or surface/near-surface methods are applied during intermediate manufacturing processes, they provide preliminary assurance that volumetric methods performed on the completed object or component will reveal few if any rejectable discontinuities, that is, flaws. Volumetric methods include radiography, ultrasonic testing, acoustic emission testing, certain infrared thermographic techniques and less familiar methods such as acoustoultrasonic testing and magnetic

TABLE 1. Objectives of nondestructive testing methods.

Objectives	Attributes Measured or Detected
Discontinuites and separations	
Surface anomalies	roughness; scratches; gouges; crazing; pitting; inclusions and imbedded foreign material
Surface connected anomalies	cracks; porosity; pinholes; laps; seams; folds; inclusions
Internal anomalies	cracks; separations; hot tears; cold shuts; shrinkage; voids; lack of fusion; pores; cavities; delaminations; disbonds; poor bonds; inclusions; segregations
Structure	
Microstructure	molecular structure; crystalline structure and/or strain; lattice structure; strain; dislocation; vacancy; deformation
Matrix structure	grain structure, size, orientation and phase; sinter and porosity; impregnation; filler and/or reinforcement distribution; anisotropy; heterogeneity; segregation
Small structural anomalies	leaks (lack of seal or through-holes); poor fit; poor contact; loose parts; loose particles; foreign objects
Gross structural anomalies	assembly errors; misalignment; poor spacing or ordering; deformation; malformation; missing parts
Dimensions and metrology	
Displacement; position	linear measurement; separation; gap size; discontinuity size, depth, location and orientation
Dimensional variations	unevenness; nonuniformity; eccentricity; shape and contour; size and mass variations
Thickness; density	film, coating, layer, plating, wall and sheet thickness; density or thickness variations
Physical and mechanical properties	
Electrical properties	resistivity; conductivity; dielectric constant and dissipation factor
Magnetic properties	polarization; permeability; ferromagnetism; cohesive force
Thermal properties	conductivity; thermal time constant and thermoelectric potential
Mechanical properties	compressive, shear and tensile strength (and moduli); Poisson's ratio; sonic velocity; hardness; temper and embrittlement
Surface properties	color; reflectivity; refraction index; emissivity
Chemical composition and analysis	
Elemental analysis	detection; identification, distribution and/or profile
Impurity concentrations	contamination; depletion; doping and diffusants
Metallurgical content	variation; alloy identification, verification and sorting
Physiochemical state	moisture content; degree of cure; ion concentrations and corrosion; reaction products
Stress and dynamic response	
Stress; strain; fatigue	heat treatment, annealing and cold work effects; residual stress and strain; fatigue damage and life (residual)
Mechanical damage	wear; spalling; erosion; friction effects
Chemical damage	corrosion; stress corrosion; phase transformation
Other damage	radiation damage and high frequency voltage breakdown
Dynamic performance	crack initiation and propagation; plastic deformation; creep; excessive motion; vibration; damping; timing of events; any anomalous behavior
Signature analysis	
Electromagnetic field	potential; strength; field distribution and pattern
Thermal field	isotherms; heat contours; temperatures; heat flow; temperature distribution; heat leaks; hot spots
Acoustic signature	noise; vibration characteristics; frequency amplitude; harmonic spectrum and/or analysis; sonic and/or ultrasonic emissions
Radioactive signature	distribution and diffusion of isotopes and tracers
Signal or image analysis	image enhancement and quantization; pattern recognition; densitometry; signal classification, separation and correlation; discontinuity identification, definition (size and shape) and distribution analysis; discontinuity mapping and display

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resonance imaging. Through-boundary methods described include leak testing, some infrared thermographic techniques, airborne ultrasonic testing and certain techniques of acoustic emission testing. Other less easily classified methods are material identification, vibration analysis and strain gaging.

No one nondestructive testing method is all-revealing. That is not to say that one method or technique of a method cannot be adequate for a specific object or component. However, in most cases it takes a series of test methods to do a complete nondestructive test of an object or component. For example, if surface cracks must be detected and eliminated and the object or component is made of ferromagnetic material, then magnetic particle would be the obvious choice. If that same material is aluminum or titanium, then the choice would be liquid penetrant or electromagnetic testing. However, for either of these situations, if internal discontinuities were to be detected, then ultrasonics or radiography would be the selection. The exact technique in either case would depend on the thickness and nature of the material and the type or types of discontinuities that must be detected.

Value of Nondestructive Testing

The contribution of nondestructive testing to profits has been acknowledged in the medical field and computer and aerospace industries. However, in industries such as heavy metals, though nondestructive testing may be grudgingly promoted, its contribution to profits may not be obvious to management. Nondestructive testing is sometimes thought of as a cost item only. One possible reason is industry downsizing. When a company cuts costs, two vulnerable areas are quality and safety. When bidding contract work, companies add profit margin to all cost items, including nondestructive testing, so a profit should be made on the nondestructive testing. However, when production is going poorly and it is anticipated that a job might lose money, it seems like the first corner that production personnel will try to cut is nondestructive testing. This is accomplished by subtle pressure on nondestructive testing technicians to accept a product that does not quite meet a code or standard requirement. The attitude toward nondestructive testing is gradually improving as management comes to appreciate its value.

Nondestructive testing should be used as a control mechanism to ensure that manufacturing processes are within design performance requirements. It should never be used in an attempt to obtain quality in a product by using nondestructive testing at the end of a manufacturing process. This approach will ultimately increase production costs. When used properly, nondestructive testing saves money for the manufacturer. Rather than costing the manufacturer money, nondestructive testing should add profits to the manufacturing process.

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PART 2. Management and Applications of Leak Testing[4,5]

Functions of Leak Testing

Leak testing is a form of nondestructive testing used in either pressurized or evacuated systems and components for detection and location of leaks and for measurement of fluid leakage. The word *leak* refers to the physical hole that exists and does not refer to the quantity of fluid passing through that hole. A leak may be a crack, crevice, fissure, hole or passageway that, contrary to what is intended, admits water, air or other fluids or lets fluids escape (as with a leak in a roof, gas pipe or ship). The word *leakage* refers to the flow of fluid through a leak without regard to physical size of the hole through which flow occurs. *Fluid* denotes any liquid *or gas* that can flow.

Surface nondestructive testing methods or volumetric nondestructive testing methods often reveal through-wall leaks to a nondestructive testing technician. However, it would not be economical to perform a complete surface liquid penetrant test of an object or component in order to detect existing leaks. Many of the penetrant indications would not be leaks through the wall. Applying the liquid penetrant to one surface and the developer to the opposite surface would increase the probability that only leaks would be detected, but this liquid penetrant technique is a leak test. This complete dependency only on capillary action to reveal leaks still would not necessarily be proof that all leaks were revealed. Adding even a small differential pressure to aide that capillary action would further enhance this leak testing technique's sensitivity.

Surface methods such as magnetic particle would be of little value in revealing leaks because they indicate linear discontinuities such as cracks or nonfusion, not through-wall leaks. Volumetric methods such as radiography or ultrasonic testing might be useful in revealing the exact location of a difficult-to-pinpoint leak, but only after that leak is detected and known to exist. A volumetric method such as acoustic emission has leak testing techniques useful in pinpointing leaks but such techniques have rather limited test sensitivity. Infrared thermography is another method whose techniques are directly related to leak testing. Other more specialized nondestructive testing methods previously mentioned would be of little use in detecting or pinpointing leaks. In the environment of high vacuum technology for things such as computer chip production, X-ray tubes, linear accelerators for both high voltage X-rays and physics research for gravitational waves and quarks, the main applicable nondestructive testing method is leak testing. Thus, leak testing and methods and techniques of leak testing must be included as a part of the nondestructive testing field.

When the specification for the manufacture of an object or component has a required minimum leak size that must be detected and/or has a required maximum total leakage rate that must be proven, then a leak testing method or technique of a leak testing method must be performed to comply with that specification requirement. No other nondestructive testing method could be substituted to fulfill that requirement.

Reasons for Leak Testing

Leaks are special types of anomalies that can have tremendous importance where they influence the safety or performance of engineered systems. The operational reliability of many devices is greatly reduced if enough leakage exists. Leak testing is performed for three basic reasons: (1) to prevent material leakage loss that interferes with system operation; (2) to prevent fire, explosion and environmental contamination hazards or nuisances caused by accidental leakage; and (3) to detect unreliable components and those whose leakage rates exceed acceptance standards.

The purposes of leak testing are to ensure reliability and serviceability of components and to prevent premature failure of systems containing fluids under pressure or vacuum. Nondestructive methods for rapid leak testing of pressurized or evacuated systems and of sealed components are thus of great industrial and military importance.

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Relationship of Leak Testing to Product Serviceability

Most types of nondestructive tests are designed to aid in evaluating serviceability of materials, parts and assemblies. Tests are used for determining integrity of structure, measuring thickness or indicating the presence of internal and surface anomalies. For most nondestructive test methods evaluation is indirect; the quantities measured have to be properly correlated to the serviceability characteristics of the material in question. Thus, the use of indirect tests depends on the interpretation of the test results. Leak testing procedures, on the other hand, facilitate direct evaluation. The measured leakage rate represents the physical effect of a faulty condition and thus requires no further analysis for practical assessment.

Determination of Overall Leakage Rates through Pressure Boundaries

Many leak tests of large vessels or systems are concerned with the determination of the rate at which a liquid, gas or vapor will penetrate through their pressure boundaries. Leakage may occur from any location within a component, assembly or system to points outside the boundary, or from external regions to points within a volume enclosed by a pressure boundary. When a fluid flows through a small leak, the leakage flow rate depends on (1) the geometry of the leak, (2) the nature of the leaking fluids and (3) the prevailing conditions of fluid pressure, temperature and type of flow. For purposes of leak testing, an easily detectable gas or liquid tracer fluid may be used, rather than air or the system operating fluid. Leakage typically occurs as a result of a pressure differential between the two regions separated by the pressure boundary.

The term minimum detectable leakage refers to the smallest fluid flow rate that can be detected. The leakage rate is sometimes referred to as the mass flow rate. In the case of gas leakage, the leakage rate describes the number of molecules leaking per unit of time, if the gas temperature is constant, regardless of the nature of the tracer gas used in leak testing. When the nature of the leaking gas and the gas temperature are known, it is possible to use the ideal gas laws to determine the actual mass of the leakage.

Measuring Leakage Rates to Characterize Individual Leaks

The flow of fluid through a leak typically results from a pressure differential or a concentration differential of a gaseous constituent that acts across the pressure boundary. The flow characteristics of a leak are often described in terms of the conductance of the leak. The leak represents a physical hole with some equivalent length and internal cross-sectional area or diameter. However, because a leak is not manufactured intentionally into a product or system, the leak hole dimensions are generally unknown and cannot be determined by nondestructive tests. Therefore, in leak testing, the quantity used to describe the leak is the measured leakage rate.

The leakage rate depends on the pressure differential that forces fluid through the leak passageway. The higher this pressure difference, the greater the leakage rate through a given leak. Therefore, leakage measurements of the same leak under differing pressure conditions can result in differing values of mass flow rate. The leak conductance is defined both by the leakage rate and the pressure differential across the leak. Thus, conductance or leakage rate at a given pressure for a particular tracer fluid should always be specified in reporting and interpreting the results of a leak test.

Ensuring System Reliability through Leak Testing

One important reason for leak testing is to measure the reliability of the system under test. Leak testing is not a direct measure of reliability, but it might show a fundamental fault of the system by a higher than expected leakage rate measurement. A high rate of leakage from mechanical connections might indicate that a gasket is improperly aligned or missing. In the same manner, a high leakage value might show the presence of a misaligned or misthreaded flange. Therefore, it is possible to detect installation errors by high leakage values. (However, the absence of high leakage does not necessarily indicate the absence of improperly installed components.) Leakage measurements to detect installation errors need not be extremely sensitive, because the leakage rates to be expected from serious error will be relatively large (10^{-1} to 10^{-5} Pa·m^3·s^{-1} or 1 to 10^{-4} std cm^3·s^{-1}). Thus, leak locations can usually be detected easily.

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For practical discussions, a *small* leak is often defined as having a low leakage rate, that is, less than that which ensures water tightness, about 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}). Leaks greater than 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) are considered *large.*

Leak Testing to Detect Material Flaws

Many leaks are caused by material anomalies such as cracks and fissures. Some of these can be detected by measurement of leakage rates. Other leaks can be detected by discontinuity detection techniques that identify leak locations. However, neither of these two leak testing technique categories will detect all anomalies. Leak testing is therefore complementary to other nondestructive testing methods used to find and evaluate basic material anomalies.

Because service reliability is not necessarily a direct function of the leakage in a system, it is difficult to establish an acceptance level for leakage rate. The decision may be influenced by the fact that increased leak testing sensitivity may detect only a small number of additional leaks at considerable added cost. This is because most leaks in welded, brazed and mechanical joints tend to be relatively large. This is partly due to the clogging of smaller leaks by water vapor and liquids that occurs in parts exposed to industrial processes or to the atmosphere. The only case where very small leaks of less than 10^{-8} Pa·m^3·s^{-1} (10^{-7} std cm^3·s^{-1}) are encountered is in parts that receive special clean room treatment during manufacture.

Specifying Desired Degrees of Leak Tightness

In industry, the term *leaktight* has taken on a variety of meanings. A water bucket is *tight* if it does not allow easily detectable quantities of water to leak out. A high vacuum vessel is tight if the rate of apparent leakage into the system cannot be indicated with the equipment on hand. One might even consider that a gravel truck is leaktight so long as there are no openings in the truck bed large enough to allow the smallest nugget to escape. The degree of leak tightness depends on the individual situation. Leak tightness requires that the leakage flow be too small to be detected. However, leak tightness is a relative term. Therefore, it becomes a necessity to establish a practical level of leak testing sensitivity for any given component under test. Thus, nothing is leaktight except by comparison to a standard or specification. Even then, the measured degree of leak tightness can be ensured only at the time of leak testing and under specific leak testing conditions. Later operation at higher pressures or temperatures might open leaks.

Avoiding Impractical Specifications for Leak Tightness

Aiming at absolute tightness is an academic endeavor. In practice, all that can be asked for is a more or less stringent degree of tightness selected according to the application requirements. Nothing made by man can truly be considered to be absolutely leaktight. Even in the absence of minute porosities, the permeation of certain gases through metals, crystals, polymers and glasses still exists.

Thus, it is necessary to establish a practical leakage rate that is acceptable for a given component under test. A preliminary decision has to be made concerning the definition of leak tightness for the particular situation. Because leak tightness is a relative term and has no absolute meaning, the sensitivity of the available leak testing equipment is a practical guide to attainable levels of leak testing sensitivity. Any increase in required sensitivity of leak testing increases the time required for leak testing and increases test cost. This increase in cost of leak testing reaches a maximum when the leakage specification is given in such impractical terms as *no detectable leakage, no measureable leakage, no leakage* and *zero leakage.*

Impractical leak testing specifications are expensive to implement. They are also very confusing unless the leak testing method is precisely described. With specifications in impractical terms, the leak testing operator is always working against background instrument noise. He must then decide whether the leakage reading obtained is caused by the random fluctuations of test instruments or by the actual detection of specific leakage. It is much easier to discriminate whether a measured leakage rate is above or below a given standard than to discriminate leakage from random instrument noise. It is therefore suggested that, when specified, zero leakage be defined as a measurable quantitative value of leakage rate that is insignificant in the operation of the system. Such a definition allows the system or the measurement sensitivity to be compared with a flow through a standard physical leak. In this way, a

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qualification of the system performance acceptability can be made during the test operation.

Specifying Leak Testing Requirements to Locate Every Leak

Occasionally it is desirable to locate every existing leak irrespective of size for the following reasons.

1. Stress leaks have a habit of growing, i.e., very small leaks may become very troublesome later, after repeated stressing.
2. High temperature leaks may be very small at test temperature but may have higher leakage rates at system operating temperatures.
3. Temperature cycling to either high or cryogenic levels usually creates stress that results in change of leakage rates.

The criterion whereby a decision is made whether or not to seek greater reliability should be the ratio of cost of the leak testing procedure to the number of leaks found. For example, improving leak testing reliability from 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}) to a reliability of 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}) may not be justified. The cost of obtaining the small increase in reliability may be prohibitive in relation to the value of the increase in detection reliability.

The expected leak tightness of sealing operations that will be used to isolate the system during leak testing must also be considered. The leak testing specification should be written with advice from an experienced engineer who makes a judgment of the reasonable value of allowable leakage rate. Factors to be considered include the leak testing method and technique; type, size and complexity of the system under test; and the service requirements and operating conditions under which the tested system will be used.

Specifying Sensitivity of Leak Testing for Practical Applications

In specifying the sensitivity of the leak testing technique, an optimum leakage sensitivity value should be sought first. Large deviations from this optimum value could increase the cost and the difficulty of measuring the leakage rate. Secondly, any increase in the sensitivity specified for a particular leakage test automatically increases the cost of leak testing. Therefore, a compromise has to be reached between testing cost and leakage tolerance. Thirdly, the sensitivity required in leak testing depends on the particular effects of leakage that must be controlled or eliminated, as illustrated in the following examples. Finally, the language in which the leak testing specification is written should be easy to interpret and to implement in testing, to ensure that management's goals are achieved by the leak test.

Specifying Tightness Required to Control Material Loss by Leakage

The first consideration in specifying the leak tightness required of a fluid containment system is to ensure that the system does not leak sufficient material to cause system failure during the operational life of the system. Then the largest leakage rate is the allowable total leakage divided by the operational life of the system. Of course, conversion might have to be made between numerical values for the tracer gas leakage during leak testing and those for the material leakage under system operation conditions.

Specifying Tightness Required to Control Environmental Contamination by Leakage

Contamination failure of a system might cause environmental damage, personnel hazard or degraded appearance. The environmental damage to a system may be caused by material leaking either into or out of the system. For example, system damage may be caused to a liquid rocket motor when the oxidizer leaks out of the storage tank and reacts with parts of the motor. On the other hand, electronic components can fail when air or water vapor enters a hermetically sealed protective container.

It is sometimes difficult to calculate the very small amount of material necessary to cause a contamination failure to occur. However, in most cases, such calculations are not impossible if the failure can be defined. For example, if some decision can be made as to the allowable amount of reaction between the oxidizer and the rocket engine parts, the maximum acceptable rate of total leakage of oxidizer from the storage tank can be defined. Similarly, in an electronic component, if failure results from adsorption of a monolayer of leaking molecules on the surface, then knowing that 10^{15} molecules form one monolayer on a square centimeter of surface makes it possible to calculate the allowable leakage rate for this particular component. If failure results from a pressure rise, then the maximum allowable pressure, the planned

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system operation time and system volume are all that are necessary for calculation of the allowable leakage rate.

Specifying Tightness Required to Avoid Personnel Hazard Caused by Fluid Leakage

Material leakage can cause personnel hazard during system operation. If the tolerable concentrations are known, and these are often reported in literature, it is again quite easy to calculate the maximum tolerable equipment leakage rate.

Specifying Tightness Required to Avoid Undesirable Appearance Caused by Leakage

An appearance specification is a specification for maximum leakage that is made because leakage of a higher value will spoil the appearance of the system. Appearance is often specified when no more stringent specification is necessary. A specification for leakage of oil out of the oil pan of a new car is a good example. This leakage specification may not be caused by concern that too much oil will be lost or that damage to the car motor will occur; instead, it is specified because the prospective buyer would not be inclined to buy a car that is dripping oil onto the showroom floor.

Specifying Tightness Required to Ensure Continuing System Operation

When appearance sets the allowable leakage of the system, the leakage is often only a nuisance. However, even leaks that are largely a nuisance may alter the effectiveness of the total system. For example, during the East Coast power blackout in the United States on November 9, 1965, a large steam generator failed during the shutdown because the auxiliary steam supply used for lubrication purposes was not available. This steam supply had been shut off earlier by workers who were bothered by excessive leakage of steam through some valve packing. This steam leakage was not critical, but it was enough of a nuisance that the system was shut down for repair. The repair did not take place in time and the bearings of the generator burned out during emergency shutdown of the system.

Definition of Leak Detector and Leak Test Sensitivity

A leak detector's sensitivity is a measure of the concentration or flow rate of tracer gas that gives a minimum measureable leak signal. Sensitivity depends on the minimum detectable number of tracer gas molecules entering the detector. The sensitivity of a leak detector is independent of the pressure in the system being tested, provided that time is ignored as a test factor.

Leak test sensitivity refers to the minimum detectable amount of leakage that will occur in a specific period of time under specified leak test conditions. It is necessary to state both the leakage rate and the prevailing test conditions to properly define leak test sensitivity in terms of the smallest physical size leak that can be detected. To avoid confusion, a set of standard leak test conditions is required.

Standard Conditions for Leak Testing

The set of conditions most commonly accepted as standard for pressure measurement is that of dry air at 25 °C (77 °F), for a pressure differential between one standard atmosphere and a vacuum (a standard atmosphere is roughly 100 kPa or precisely 101.325 kPa). For practical purposes, the vacuum need be no better than 0.01 of an atmosphere or 1 kPa (0.15 $lb_f \cdot in.^{-2}$). When a leak is being described and only the leakage rate is given, it is assumed that the leakage rate refers to leakage at standard conditions. The sensitivity of a leak testing instrument is synonymous with the minimum detectable leakage or minimum flow rate the instrument can detect. These minima are independent of leak testing conditions. When the instrument is applied to a test, the leak testing sensitivity depends on existing conditions of pressure differential, temperature and fluid type in addition to the instrument sensitivity. However, the leak test instrument should be more sensitive by at least a factor of 2 than the minimum leakage to be detected, to ensure reliability and reproducibility of measurements.

Example of Sensitivity and Difficulty of Bubble Leak Testing

Each modification of a leak testing procedure has an optimum sensitivity value at which it is most readily used.

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Deviation from this optimum value of sensitivity makes it more difficult to perform the measurement and decreases confidence in the results. Figure 1 shows the influence of leak testing sensitivity level on the ease of operation of test equipment. In most cases, after reaching a plateau, further increase of sensitivity rapidly decreases the ease of operation. Bubble testing by immersion in water is an example of how the optimum value affects the ease of performing the test.

The bubble testing sensitivity range extends from 10^{-2} to 10^{-5} Pa·m^3·s^{-1} (10^{-1} to 10^{-4} std cm^3·s^{-1}). In measuring for 10^{-2} Pa·m^3·s^{-1} (10^{-1} std cm^3·s^{-1}) leaks, a component may be placed in water and observed quickly. Bubbles may emerge from the pressurized component at such a rapid rate that there is no question of the existence of a leak. When checking for leaks in the range of 10^{-3} to 10^{-4} Pa·m^3·s^{-1} (10^{-2} to 10^{-3} std cm^3·s^{-1}), the operator must be sure that the test object or component is submerged long enough for any bubbles coming from crevices to have a chance to collect and rise. When locating leaks in the 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) range, the component, after being immersed, has to be completely stripped of attached air bubbles so that the bubble formed by leaking gas may be detected. The 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) leakage range is near the limit of detectability of the bubble technique, although longer waiting periods theoretically could obtain higher sensitivity. Longer waiting periods become impractical when the rate of bubble evolution approaches the rate at which tracer gas is dissolving in the test fluid.

Specifying sensitivity much greater than 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) makes bubble testing exceedingly difficult. For instance, bubble testing could be used at higher sensitivity by saturating the immersion liquid with the tracer gas used in leak testing. However, it would be better to change to a different leak testing method that is more effective at that higher sensitivity. Bubble testing to detect leaks greater than 10^{-2} Pa·m^3·s^{-1} (10^{-1} std cm^3·s^{-1}) becomes difficult because of rapid gas evolution and rapid decay of pressure in the system under test. However, difficulties in the less sensitive test range are usually not so great as in the more stringent sensitivity range.

Relation of Test Costs to Sensitivity of Leak Testing

Leak testing instrumentation costs increase as required test sensitivity increases, as sketched in Fig. 2.[5] The test equipment investment for determining a leakage rate of 10^{-4} Pa·m^3·s^{-1} (10^{-3} std cm^3·s^{-1}) is negligible compared with that for a sensitivity of 10^{-13} Pa·m^3·s^{-1} (10^{-12} std cm^3·s^{-1}), whose cost is 10 000 times higher. Even after a test technique has been selected, raising leak sensitivity requirements within this technique will result in an increase in measurement cost. This increase is usually caused by greater complexity of leak tests with increased sensitivity. Cost increases become particularly drastic when the required sensitivity is higher than the optimum operating range shown in Fig. 1.

FIGURE 1. Ease of test operation as a function of leak testing sensitivity.

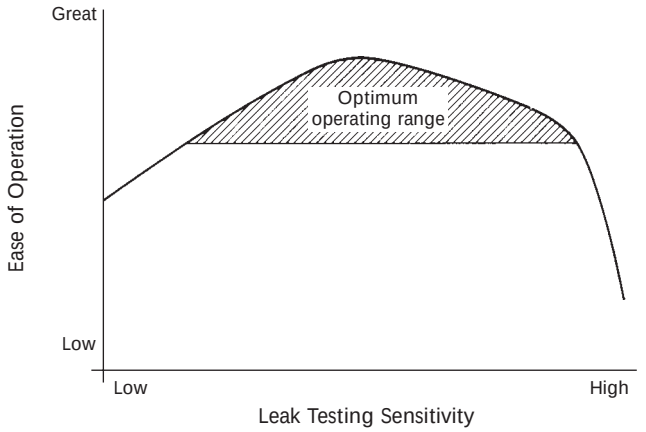

TABLE 2. Leak testing methods and techniques.

Methods	Techniques
Bubble solution	immersion; film solution
Ultrasonic/acoustic	sonic/mechanical flow; sound generator
Voltage discharge	voltage spark; color change
Pressure	hydrostatic; hydropneumatic; pneumatic
Ionization	photo ionization; flame ionization
Conductivity	thermal conductivity; catalytic combustible
Radiation absorption	infrared; ultraviolet; laser
Chemical based	chemical penetrants; chemical tracer gases
Halogen detector	halide torch; electron capture; halogen diode
Radioisotope	krypton-85
Pressure change	absolute; reference; pressure rise; flow measurement; pressure decay; volumetric
Mass spectrometer	helium or argon; tracer probe location; hooding total leakage; detector probe location; sealed objects; residual gas analyzer

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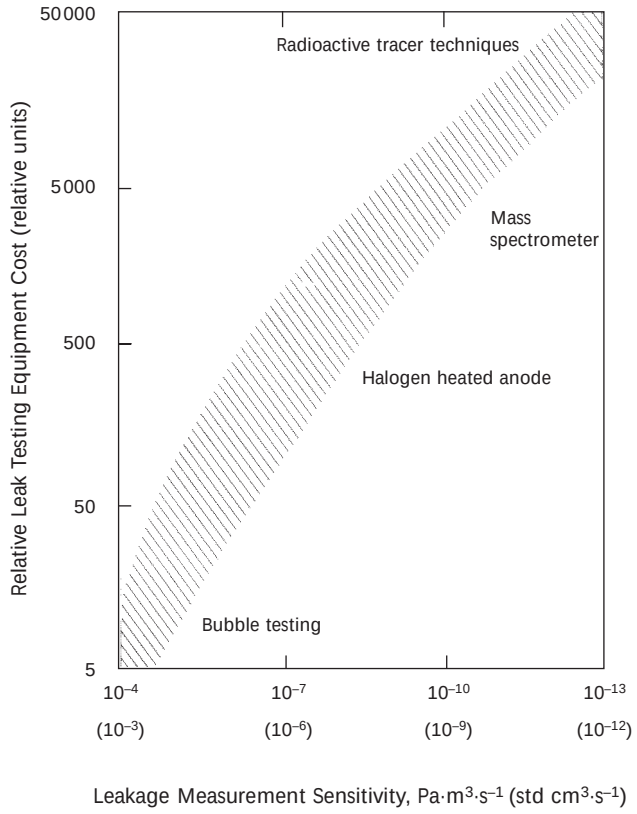
Selection of Specific Leak Testing Technique for Various Applications[6]

Figure 3 provides a graphical guide to selection of leak testing methods and techniques for various applications. It shows a *decision tree* with which the choice of a leak testing method becomes a step-by-step process. The selection processes suggested by Fig. 3 serve as a basic guide.[5] Further consideration of specific leak testing requirements may suggest other methods or techniques for test selection or cause the test engineer to modify leak testing procedures. See also Table 2. The final selection of the leak testing method will typically be made from perhaps only three or four possible test methods. The special conditions under which tests must be made can become a major factor in this final test selection.

The first question to be asked when choosing the best leak testing method, or technique of a method, is "Should this test reveal the presence of a suspected leak, or is its purpose to show the location of a known leak?" The second question to be answered is, "Is it necessary to measure the rate of leakage at the specific leak?" If leakage measurement is essential, use of calibrated or reference leaks or other means to provide quantitative leakage measurement is required. In the *decision tree* of Fig. 3, the first branch (or decision point) answers the preceding questions and determines if the purpose or requirements of the test lead to the upper branch of leak location only or to the lower branch of leakage rate measurements.

FIGURE 2. Effect of required sensitivity on leak detection equipment cost.



Basic Categories of Leak Testing

Types of Fluid Media Used in Leak Testing

Leak testing can be divided into three main categories: (1) leak detection, (2) leak location and (3) leakage measurement. Each technique in all categories involves a fluid leak tracer and some means for establishing a pressure differential or other means for causing fluid flow through the leak or leaks. Possible fluid media include gases, vapors and liquids or combinations of these physical states of fluid probing media. Selection of the desired fluid probing medium for leak testing depends on operator or engineering judgment involving factors such as: (1) type and size of test object or system to be tested; (2) typical operating conditions of test object or system; (3) environmental conditions during leak testing; (4) hazards associated with the probing medium and the pressure conditions involved in testing; (5) leak testing instrumentation to be used and its response to the probing medium; (6) the leakage rates that must be detected and the accuracy with which measurements must be made; and (7) compatibility of test probing medium with test object and content (to avoid corrosion etc.).

Gases and vapors are generally preferred to liquid media where high sensitivity to leakage must be attained; however, liquid probing media are used for leak testing in many specific applications.

Selection of Tracer Gas Technique for Leak Location Only

As shown on the upper branch of the *decision tree* of Fig. 3, tracer gas tests whose purpose is leak location only can be divided into a tracer probe technique

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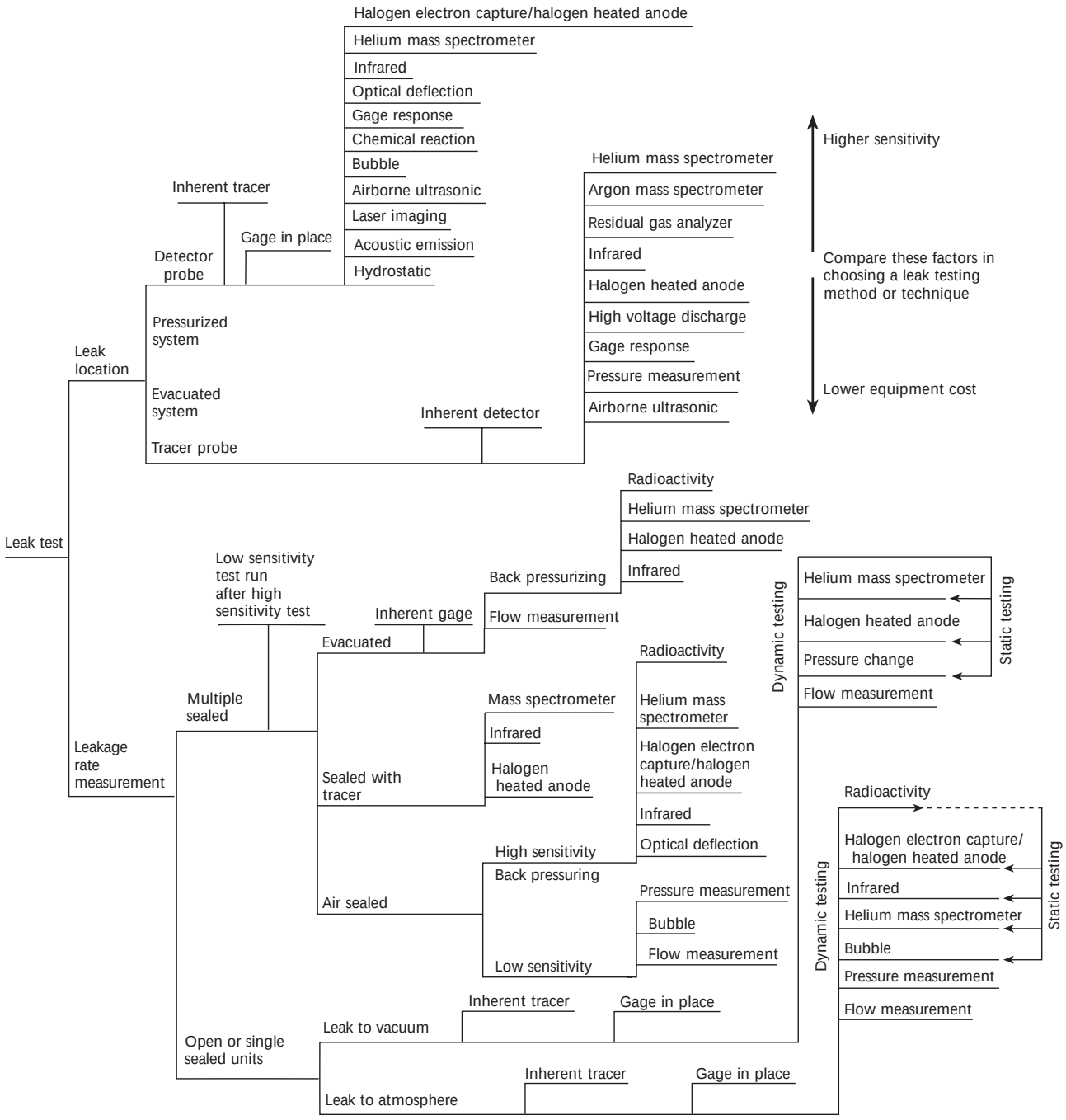
and a detector probe technique (see Fig. 4).[5] When choosing either technique, it is important that leak location be attempted only after the presence of a leak has been ascertained. The tracer probe technique is used when the test system is evacuated and the tracer gas is applied to the outside of the pressure boundary of the test system. The detector probe technique is selected when the test system is pressurized with gases including the tracer gas (if used) and the sniffing or sampling of the leaking gas is being done at atmospheric pressure in the ambient air. This selection corresponds to the second decision point in the upper branch of the *decision tree* of Fig. 3.

FIGURE 3. Graphical decision tree for step-by-step selection of leak testing methods.



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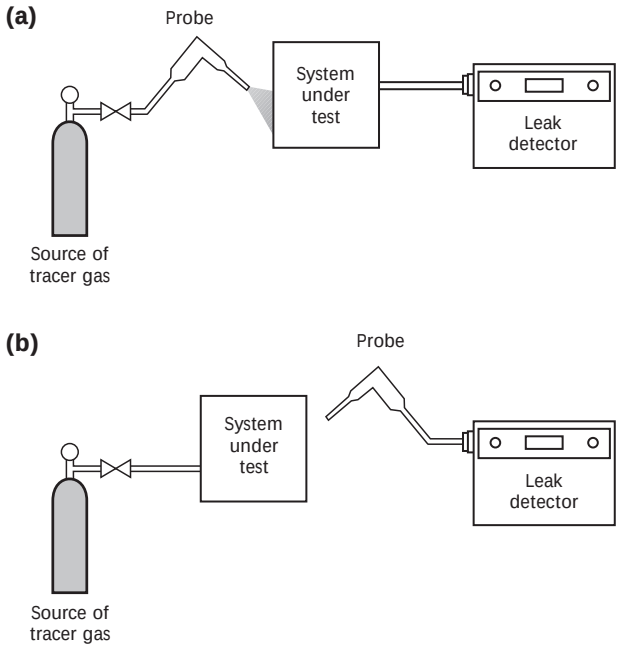
Factors Influencing Choice between Detector Probe and Tracer Probe Tests

One of the most difficult and important decisions is the choice of which leak testing method should be used. A correct choice will optimize sensitivity, cost and reliability of the leak testing procedure. Choice of an incorrect test method makes leak testing less sensitive and less reliable, while adding to the difficulty of testing. One simplified way to choose is to rank various leak testing methods by means of their leakage sensitivity. If this were sufficient, the test engineer would only need to decide what degree of sensitivity is required and then to select the test method from among those offering adequate sensitivity for the specific test application. However, each leak testing technique can have a different test sensitivity under different operating conditions. For example, a mass spectrometer leak detector is 10 000 times more sensitive than a heated anode halogen vapor detection instrument when used for leak location in the tracer probe leak location test of an evacuated vessel. However, if these two instruments are used for leak detection on a pressurized test system, the halogen leak detector is 100 times more sensitive. The reason for this apparent discrepancy becomes obvious on close examination of the operating characteristics of these two instruments. The mass spectrometer is designed for operation under vacuum conditions, whereas the halogen leak detector is designed for operation in air at atmospheric pressure.

As another example, a helium mass spectrometer leak detector may have a leakage sensitivity of 10^{-12} Pa·m^3·s^{-1} (10^{-11} std cm^3·s^{-1}) during routine leak testing with dynamic leakage measurement techniques. On very small systems, this optimum sensitivity may be increased to 10^{-15} Pa·m^3·s^{-1} (10^{-14} std cm^3·s^{-1}), a gain of 1000×, by using the static accumulation leakage measurement technique. However, the static leakage measurement technique is not the standard method of using the mass spectrometer leak detector. Therefore, the last sensitivity stated above is subject to some question. It must be recognized that each method of leak detection or measurement is usually optimized for one particular type of leak testing. Therefore, it can be a mistake to compare sensitivities of various leak testing methods under the same conditions, if each test is not designed to operate under these same conditions.

FIGURE 4. Tracer gas probing for locating leaks with sensitive electronic leak detection instruments; (a) tracer probe technique; (b) detector probe technique.



Leak Location Technique with Detector Probe Operating at Atmospheric Pressure

When testing a pressurized system that is leaking into the atmosphere, the next decision point is whether or not the leaking fluid can be used as a tracer (this decision point lies along the top branch of the tree of Fig. 3). For example, most refrigeration and air conditioning systems are charged with a refrigerant gas (refrigerant-22 or -134a) that is a fluorocarbon to which the heated anode halogen vapor detector is specifically highly sensitive. When searching for leaks in operating systems of this type, the inherent tracer dictates the use of the halogen leak testing method. Because of potential environmental effects from fluorocarbons, some current systems are being charged with refrigerant-134a gas or sulfur hexafluoride for use, respectively, with modified residual gas analyzer halogen leak detectors or electron capture halogen leak detectors.

If the pressurized test system contains ammonia gas, a chemical type of leak detector might prove to be optimum. In certain cases where the mass spectrometer leak detector is to be used, the presence of a specific gas (such as argon, helium or neon) within the system provides an excellent inherent tracer. Alternative procedures involve pressurizing the test system with such a tracer gas or a mixture of air with tracer gas.

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Some other methods for leak location do not depend on the specific nature of the leaking gas; among these are the ultrasonic leak detector and bubble testing. In some cases, the tracer gas might be suitable for use with more than one testing method, e.g., helium could be used for bubble testing for large leaks or for mass spectrometer testing for small leaks or quantitative leakage measurements.

The detector probe leak testing methods, in order of increasing leak sensitivity, time and costs, are ultrasonic, bubble, chemical, pressure or flow gage response, infrared gas detector, mass spectrometer leak detector and halogen vapor detector. These relative sensitivity ratings apply for detector probes searching with the detector inlet probe or sniffer searching in air at atmospheric pressure. These alternative leak test methods are listed vertically at the right end of the top branch of the *decision tree* of Fig. 3. The lowest cost, highest speed, simplest leak tests are at the bottom of this list. The slower, more costly, higher sensitivity test methods appear at the top of the list shown to the right of the top branch of the *decision tree* of Fig. 3.

Leak Location Technique with Tracer Probe outside an Evacuated System

When testing an evacuated system that has in-leakage from the ambient atmosphere or from a tracer probe, the first consideration in selection of a test method is whether there is an inherent detector within the system. the inherent detector might be a pressure gage of an electronic type or, more desirably, a gage that is specifically responsive to the partial pressure of a specific tracer gas. Vacuum systems often contain one or more types of vacuum gages. In Fig. 3, this point appears in the second main line from the top, for tracer probe testing of evacuated systems, and is labeled *inherent detector*.

If a vacuum gage does not exist within the evacuated system under test, other test methods must be examined individually to determine their limitations and advantages for leak testing of this system. The tracer probe leak testing methods, in order of increasing leak sensitivity, time and cost, are ultrasonic, pressure change gage response, high voltage electrical discharge, heated anode halogen detector, infrared gas detector and mass spectrometer helium leak detector (highest in list). These methods are listed vertically at the right end of the second horizontal branch in Fig. 3.

The methods shown in the upper half of Fig. 3 for leak location are those in primary or most common usage. Other methods, such as those using radioactive tracer gases, are not generally used because of safety and other operating problems associated with their use. However, if none of the leak location methods described for detector probe or tracer probe leak tests in the preceding discussion is satisfactory for a specific application, more complicated leak testing methods may be considered during selection of an appropriate leak testing method.

Selection of Technique for Leakage Measurement

The lower half of the *decision tree* diagram of Fig. 3 is a guide for step-by-step selection of optimum techniques for leakage measurements. Leakage measurements can be divided into two different types based on the nature of the test objects whose leakage is to be measured. The first decision is based on the accessibility of test surfaces on the pressure boundaries of the test object. Test objects are classified by accessibility into two groups.

1. Open units are accessible on both sides of the pressure boundary, for tracer probes or detector probes.
2. Sealed units are accessible only on external surfaces.

The second category usually consists of mass produced items such as transistors, relays, ordnance components and sealed instruments. In the lower portion of Fig. 3, this choice is indicated first on the decision path for leakage measurement.

Practical Measurement of Leakage Rates with Gaseous Tracers

Principles of Leakage Measurement

All leak detection with tracer gases involved their flow from the high pressure side of a pressure boundary through a presumed leak to the lower pressure side of the pressure boundary. When tracer gases are used in leak testing, instruments sensitive to tracer gas presence or concentration are used to detect outflow from the low pressure side of the leak in the pressure boundary. Where leak tests involve measurements of change in pressure or change in volume of gas within a pressurized enclosure, the loss of internal gas pressure or volume indicates that leakage has occurred through the

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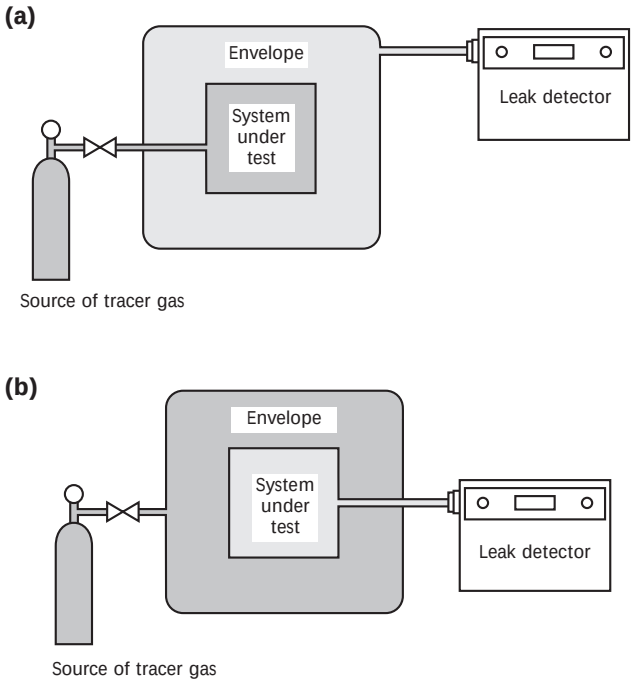
pressure boundary (or temporary seals placed on openings of the pressure boundary). When evacuated or low pressure test systems or components are surrounded by higher pressure media such as the earth's atmosphere, or a hood or test chamber containing gases at higher pressures, leakage can be detected by loss of pressure in the external chamber or by rise in pressure within the lower pressure system under test.

Classification of Techniques of Leakage Measurement with Tracer Gases

Leakage rate measurement techniques involving the use of tracer gases fall into two other classifications known as (1) static leak testing and (2) dynamic leak testing. In static leak testing, the chamber into which tracer gas leaks and accumulates is sealed and is not subjected to pumping to remove the accumulated gases. In dynamic leak testing, the chamber into which tracer gas leaks is pumped continuously or intermittently to draw the leaking tracer gas through the leak detector instrumentation, as sketched in Fig. 5.[5] The leakage rate measurement procedure consists of first placing tracer gas within or around the whole system being tested. A pressure differential across the system boundary is established either by pressurizing the one side of the pressure boundary with tracer gas or by evacuating the other side. The concentration of tracer gas on the lower pressure side of the pressure boundary is measured to determine leakage rates.

FIGURE 5. Leakage measurement dynamic leak testing using vacuum pumping: (a) pressurized system mode for leak testing of smaller components; (b) pressurized envelope mode for leak testing of larger volume systems.



Leakage Measurements of Open Test Objects Accessible on Both Sides

When test objects have pressure boundaries accessible on both sides, the second decision in the selection of a leakage measurement test method is whether the unit can or should be evacuated during leak testing. This decision will determine if the leak test is performed with the tracer probe or detector probe. If one side of the pressure boundary can be evacuated so that leakage occurs to vacuum and the leak detector is placed in the vacuum system, more sensitive leak testing will usually result. In vacuum, the tracer gases can reach the detector quickly, particularly with dynamic tests in which the evacuated test volume is pumped rapidly and continuously. In this case, there is little possibility of stratification of tracer gases.

However, evacuation does not always produce the most sensitive and reliable leakage measurements. If the test volume is extremely large, high pumping speed is necessary to reduce response time. Such auxiliary pumping will cause split flow, thus reducing the amount of tracer gas reaching the leak detector. This, in turn, can reduce signal levels and leakage sensitivity. Other restraints may prevent evacuation of the test system to a sufficiently low absolute pressure. Conventional helium mass spectrometer leak detectors, for example, should be operated at vacuum levels of 0.1 Pa (1 mtorr) or lower. Conventional helium mass spectrometers can operate with manifold vacuums of 2 Pa (20 mtorr) or lower whereas counterflow helium mass spectrometers can operate with manifold vacuums of 10 Pa (0.1 torr) and higher. The structure of the equipment under test (particularly if thin walls not intended to withstand external pressure are involved) may prevent use of leakage rate measurement techniques in which the leak detector must operate within a vacuum. In Fig. 3, the lowest branch leading to the junction of the *leak to vacuum* path and the *leak to atmosphere* path represents the point of decision discussed in this paragraph.

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Selecting Specific Method for Leak Testing of Evacuated Test Units or Systems

As indicated along the next-to-bottom decision path at the center of Fig. 3, the first approach to selecting leak test methods for units that can be evacuated is to determine whether or not there is an inherent tracer in the test system while in operation. For example, if in normal operation the system under test contains one of the specific tracer gases such as helium or halogenated hydrocarbons, a test method sensitive to that specific tracer gas might be preferred. In this way, considerable savings in test time and cost can be realized if there is no need to fill the system under test with a tracer gas.

If there is no inherent tracer gas within the system under test, the next decision step might be to determine if there is a pressure or flow gage already present in the evacuated system to be leak tested. If so, this gage might be used for leakage measurement in place of some additional type of leak detector. This internally available gage might be a simple vacuum dial, thermocouple or ionization gage or, in some fortunate cases, a mass spectrometer that is incorporated into the system as a part of its analytical instrumentation or controls. Consideration need not be limited to those types of gages commonly used for leak testing. Any gas concentration measuring equipment that happens to be available may be used for leakage measurement and is accurate enough and sensitive enough for the required results. This decision point is that labeled *gage in place* in the two bottom decision pathways shown in Fig. 3.

Methods of Leakage Measurement in Evacuated Systems with No Inherent Tracer

If there is no inherent tracer or adequate gage present within an evacuated test system, other vacuum mode leak testing methods must be considered. Methods for leak testing of evacuated systems, in order of increasing leak sensitivity and cost of leak testing equipment, include gas flow measurement, pressure change measurement, heated anode halogen vapor leak detection and mass spectrometer helium leak detection. These methods, listed vertically at the end of the next-to-bottom decision line in Fig. 3, should each be considered individually and evaluated in terms of their advantages and limitations. In most cases, all of the possible leak testing methods should be considered. Selection depends on pertinent factors. For example, a more sensitive leak testing method might involve higher initial costs for equipment and test setups but, on the other hand, it might result in great cost savings during testing programs or provide greater reliability in leak testing results.

Once the basic vacuum leak testing method has been selected, a second consideration involves selection between static and dynamic test techniques. It is usually preferable to perform leak tests using a dynamic testing technique (tests involving pumping of the vacuum system throughout the test period). However, static techniques of leakage rate measurement should also be considered. Static tests involving rise or loss in pressure, or accumulation of tracer gases over prolonged leak periods, are slower than typical dynamic leak tests. However, higher sensitivity can be achieved in static tests if the volume under test is not excessive; this may be worth the extra effort.

Selection of Test Methods for Systems Leaking to Atmospheric Pressure

The choice of pressure mode testing methods — i.e., for test systems leaking to atmospheric pressure — should be made by following the same type of decision pattern as for leak testing of evacuated systems. The decision path for this case appears at the bottom of Fig. 3. The leak testing methods applicable to testing of systems leaking to atmosphere, in order of increasing test sensitivity, are flow measurement, pressure measurement (for larger volume systems), immersion bubble testing, infrared gaseous leak testing, heated anode and electron capture halogen leak testing, mass spectrometer helium leak testing and leak testing using radioactive tracer gases. A dynamic leak testing method should be used wherever possible. After various dynamic leak test methods have been considered and those whose limitations are unacceptable have been rejected, a static leak testing method should also be considered. Although a static technique will increase leak testing time, it will also increase leak testing sensitivity.

Leak Testing to Locate Individual Leaks

Leak testing for the purpose of locating individual leaks is required when it is necessary to detect, locate and evaluate each leak; unacceptable leaks then can be repaired and total leakage from a vessel or system brought within acceptable limits.

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Methods for detecting and locating individual leaks are generally quantitative only in the sense that the lower limit of detectable leak size is determined by the sensitivity of the leak detecting indicators and test method used. Thus, only rather crude overall leakage rate information could be approximated by adding the leakage rates measured for the leaks that are detectable. Numerous different leak detecting, locating and measuring techniques and devices are available. The selection of test equipment, tracer gas and leak detection method is influenced by the following factors: (1) size of the leaks to be detected and located; (2) nature and accuracy of leak test information required; (3) size and accessibility of the system being tested; (4) system operating conditions that influence leakage; (5) hazards associated with specific leak location methods; (6) quantity of parts to be tested; and (7) ambient conditions under which leak location tests are required to be carried out (wind or lack of air circulation and stratification effects can influence test sensitivity and personnel).

Classification of Techniques for Locating and Evaluating Individual Leaks

Techniques for location and evaluation of individual leaks can be categorized in various ways, including by types of leak tracer used in the detection, location and possible measurements of individual leaks. A primary classification is that between the use of liquid tracers and the use of more sensitive gaseous tracers. Leak location techniques that depend on tracer gas properties are listed below in general categories, in order of increasing leak testing sensitivity and complexity of test methods:

1. leak location techniques independent of any characteristic properties of the tracer gas (use of candles, liquid and chemical penetrants, bubble testing and sonic or ultrasonic leak tests, for example);
2. leak location techniques using tracer gases with easily detectable physical or chemical properties (gases with thermal conductivities or chemical properties differing from those of the pressurizing gas, gaseous halogen compounds and gases having characteristic radiation absorption bands in the ultraviolet or infrared spectral ranges); and
3. leak location techniques involving the use of tracer gases with atomic or nuclear properties providing easily detectable leak signals (helium and other inert gases having specific charge-to-mass properties that permit their sensitive detection by mass spectrometers and gaseous radioactive isotopes detectable with particle counters and radiation detectors).

Tables 3 and 4 list some typical leak detection systems and give their leakage sensitivities.

Techniques for Locating Leaks with Electronic Detector Instruments

Figure 4 shows arrangements of two basic techniques for locating leaks with electronic instruments that detect gas flow or presence of specific tracer gases: (1) the detector probe probe technique and (2) the tracer technique. With either, it is important that leak location pinpointing be attempted only after the presence of a leak has been ascertained. When choosing between the pressure test technique and the vacuum test technique, both of the alternative techniques listed above must be considered when the test object will

TABLE 3. Sensitivity limits of various methods of leak testing.

Method	Minimum Detectable Leakage Rate $Pa \cdot m^3 \cdot s^{-1}$ (std $cm^3 \cdot s^{-1}$)		Comments
Mass loss	time limited		pressure change; generally limited to sizable leaks; good overall quantitative measure; no information on leak location; time consuming
Ultrasonics	0.05	(0.5)	leak location only; fast; no cleanup; can detect from distance; large leaks only
Penetrants	$\leq 10^{-4}$	($\leq 10^{-3}$)	simple to use; location only; may plug small leaks; requires cleanup
Bubbles	10^{-5}	(10^{-4})	for leak location; fluids may plug small leaks; requires cleanup
Thermal conductivity	10^{-6}	(10^{-5})	simple; compact; portable; inexpensive; sensitive to various gases; operates in air
Halogen	10^{-10}	(10^{-9})	operates in air; sensitive (10^{-12} claimed with sulfur hexafluoride); portable; requires cleanup; loses sensitivity with use; sensitive to ambient halide gases
Mass spectrometer	10^{-13}	(10^{-12})	most accurate for vacuum testing; expensive; relatively complex; not as portable as halogen detectors; much less sensitive when used in detector probing

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withstand either pressure or vacuum. If a satisfactory choice of one technique has been made, it is a good idea to compare it with a satisfactory choice of the other technique, to see if reduced cost or an easier test method might be possible.

The detector probe leak location technique is used when the system under test is pressurized and testing is done at ambient atmospheric pressure. The tracer probe technique is usually used when the system under test is evacuated and the tracer gas comes from outside this system. The tracer probe technique is usually the most rapid test because the tracer gas travels more rapidly in vacuum and so reaches the leak detector in a shorter time. On the other hand, a higher pressure differential can be used with the detector probe.

Coordinating Overall Leakage Measurements with Leak Location Tests

Leakage rate measurement techniques do not provide information on the number and locations of individual leaks. The latter can only be determined by leak location test techniques. However, use of the leak location techniques alone cannot give reliable assurance that no leaks exist or that tests have revealed all leaks that exist. Without prior assurance that leaks do exist, leak location test techniques become arbitrary in application.

In practice, preliminary leakage testing is often done first by less sensitive methods to permit detection, location and rectification of gross leaks. Next, the operator can determine if any additional leakage exists by an overall leakage measurement of the entire test vessel, system or component. Then each individual leak should be discovered by sensitive leak location techniques and repaired if feasible, until all detectable leak locations have been identified and their leaks rectified. For final assurance that the test object or system meets leakage specification requirements, it may be necessary to repeat the overall leakage rate measurement to determine whether the total leakage rate falls within the acceptable limits.

TABLE 4. Relative ultimate leakage sensitivities of leak testing methods under ideal conditions with very high concentrations of tracer gases.[a]

Test Method	Minimum Detectable Leakage Rate $Pa \cdot m^3 \cdot s^{-1}$	Minimum Detectable Leakage Rate (std $cm^3 \cdot s^{-1}$)
Liquid pressure drop	——[b]	——[b]
Gas pressure drop	——	——
Pressure rise	——[c]	——[c]
Ultrasonic leak detector	10^{-2}	(10^{-1})
Volumetric displacement [d]	10^{-3}	(10^{-2})
Gas discharge	10^{-3}	(10^{-2})
Ammonia and phenolphthalein	10^{-3} to 10^{-4}	$(10^{-2}$ to $10^{-3})$
Ammonia and bromocresol purple	10^{-3} to 10^{-4}	$(10^{-2}$ to $10^{-3})$
Ammonia and hydrochloric acid	10^{-3} to 10^{-4}	$(10^{-2}$ to $10^{-3})$
Ammonia and sulfur dioxide	10^{-3} to 10^{-4}	$(10^{-2}$ to $10^{-3})$
Halide torch	10^{-4}	(10^{-3})
Air bubbles in water	10^{-4} to 10^{-5}	$(10^{-3}$ to $10^{-4})$
Air and soap or detergent	10^{-4} to 10^{-5}	$(10^{-3}$ to $10^{-4})$
Thermal conductivity	10^{-5}	(10^{-4})
Infrared	6×10^{-5} to 6×10^{-7}	$(6 \times 10^{-4}$ to $6 \times 10^{-6})$
Hydrogen Pirani	10^{-7}	(10^{-6})
Hot filament ionization gage	10^{-7} to 10^{-8}	$(10^{-6}$ to $10^{-7})$
Mass spectrometer detector probe	10^{-6} to 10^{-7}	$(10^{-5}$ to $10^{-6})$
Halogen diode detector	10^{-7} to 10^{-9}	$(10^{-6}$ to $10^{-8})$
Hydrogen bubbles in alcohol	5×10^{-7}	(5×10^{-6})
Paladium barrier detector	10^{-8} to 10^{-9}	$(10^{-7}$ to $10^{-8})$
Mass spectrometer envelope test	10^{-10}	(10^{-9})
Radioactive isotopes	10^{-9} to 10^{-13}	$(10^{-8}$ to $10^{-12})$

a. Numbers not to be used as guides in practical leak testing.
b. Depends on volume tested and pressure range of gage.
c. Depends on volume tested.
d. Gas type flow meters.

Training of Leak Testing Personnel[7]

Because of the many leak testing techniques and the multiple variations of each, leak testing could require more training and knowledge than any of the other nondestructive testing methods. Successful execution of many of these techniques by inspection personnel is highly dependent on knowledge and skill. Nevertheless, there are fewer instruction and training materials available for leak testing than for other methods.

Leak testing may be divided into four methods: bubble testing, pressure change testing, halogen diode leak testing and mass spectrometer leak testing (see Table 2), to which may be added acoustic methods. The outline for the Level I leak testing methods course in *Recommended Practice No. SNT-TC-1A* expands this list of four methods to a total of 12 techniques.[8]

The 34 variations in Table 2 reveal the complex nature of leak testing and may also be the reason why such a small percentage of ASNT membership is qualified to Level III in the leak test method. At Level I, proficiency in one or two techniques is possible, but it would be very difficult to meet the training and experience guidelines that are recommended by ASNT for more than two or three techniques. A brief listing for each technique may make you aware of your weaknesses. Variations of each technique may require familiarity with different test equipment and tracers.

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Many inspection people are also confused, when choosing a technique, by the disadvantages and limitations in sensitivity for each technique.

Inspection personnel often have difficulty understanding how extremely small some leaks are that they will try to find. This also makes it difficult to realize that some leaks may be temporarily sealed by foreign material such as oil, grease, water even cleaning solvents or even moisture in air. Improper handling after cleaning may temporarily prevent location of leaks that will reappear at a later time. A comparison of leakage rates in three different ways (Table 5) may help to visualize the size.

When leak testing is performed with equipment capable of locating and measuring leaks smaller than 10^{-9} Pa·m^3·s^{-1} (10^{-8} std cm^3·s^{-1}), tracer gas permeation through the test object materials of construction may appear as a leak indication several seconds to hours after application of the tracer. This may require a knowledge of those materials that allow permeation by the tracer being used.

Many Level II or III inspection personnel establish reject specifications that are unrealistically small with respect to the expected life of the product being tested. As a result, many tested objects with leaks that are 10 to 100 times smaller than an acceptable level are rejected for repair or destruction. This creates unnecessary cost and loss of profits. Some examples of leaks that may affect certain products are as follows: chemical process equipment, 10^{-2} to 10^{-1} Pa·m^3·s^{-1} (10^{-1} to 1 std cm^3·s^{-1}); torque converter, 10^{-4} to 10^{-5} Pa·m^3·s^{-1} (10^{-3} to 10^{-4} std cm^3·s^{-1}); beverage can end, 10^{-6} to 10^{-7} Pa·m^3·s^{-1} (10^{-5} to 10^{-6} std cm^3·s^{-1}); vacuum process system, 10^{-7} to 10^{-8} Pa·m^3·s^{-1} (10^{-6} to 10^{-7} std cm^3·s^{-1}); integrated circuit package, 10^{-8} to 10^{-9} Pa·m^3·s^{-1} (10^{-7} to 10^{-8} std cm^3·s^{-1}); pacemaker, 10^{-10} Pa·m^3·s^{-1} (10^{-9} std cm^3·s^{-1}).

Another reason training must be emphasized is that many leak testing hazards may exist that cause injury to inspection personnel, damage to test equipment or damage to the product being tested. The following examples illustrate numerous hazards: flammable/toxic solvents for cleaning, flammable/toxic/explosive tracers, asphyxiation by vapors or tracer gases, access difficult on large objects, pneumatic and hydrostatic pressure, radioactive tracer gases, compressed gas cylinders/regulators and structural stress.

To summarize the need for leak testing methods training, there are eleven reasons to expand this training: choice of many techniques, sensitivity of various techniques, advantages and limitations of each technique, dependence of techniques on testing skills and experience, leakage location versus measurement, factors affecting measurement accuracy, employers' cutting cost by hiring entry level people and minimizing training time, hazards to personnel and products, few courses available that offer skills training, limited available training materials and the small number of qualified Level III personnel.

TABLE 5. Comparison of leak rates.

Measurement[a] std cm^3·s^{-1}	Equivalent[b]	Bubble Equivalent[b,c]
10^{-2}	1 std cm^3/10 s	steady stream
10^{-3}	1 std cm^3/100 s	10 s^{-1}
10^{-4}	3 std cm^3/h	1 s^{-1}
10^{-5}	1 std cm^3/3 h	0.1 s^{-1}
10^{-6}	1 std cm^3/24 h	—— [d]
10^{-7}	1 std cm^3/2 wk	—— [d]
10^{-8}	3 std cm^3/yr	—— [d]
10^{-9}	1 std cm^3/3 yr	—— [d]
10^{-10}	1 std cm^3/30 yr	—— [d]
10^{-11}	1 std cm^3/300 yr	—— [d]
10^{-12}	1 std cm^3/3000 yr	—— [e]

a. 1 std cm^3·s^{-1} = 0.1 Pa·m^3·s^{-1}.
b. Approximate.
c. Assuming bubble of 1 mm^3 (6.1×10^{-5} in.^{3}) volume.
d. Bubbles too infrequent to observe or partially dissolved.
e. Smallest detectable leak by mass spectroscopy.

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PART 3. History of Leak Testing[9]

According to modern accounts, making a vacuum was generally considered impossible until the mid-1600s. However, leaks have concerned technologists for thousands of years.

Despite the importance of leaks for ship construction, nothing on methods of caulking is to be found in reference works in the history of ancient technology. Leak testing, up to the era of vacuum, depended solely on the eye and was so commonplace as to escape attention. At any event, references to leak testing are hard to find until well into the 1800s.

Ruhmkorff and Tesla Coils as Leak Detector

Although Nollet in Paris observed the electric discharge in an exhausted vessel in 1740, it was not until a century later that substantial investigation of this low pressure discharge took place. Michael Faraday, in 1831, had enunciated the principle of the induction coil and had studied discharges in gases by 1839.

By about 1850, Ruhmkorff and others had made substantial improvements in Faraday's coil. Presumably, development of the Ruhmkorff induction coil and the Tesla coil greatly facilitated investigation of the high voltage vacuum discharge.

By 1859, there were reports by Gassiot and others of the changing nature of the discharge with pressure. Moreover, it was observed that the color of the discharge depended on the gas in the discharge tube as well as on the pressure.

It seems likely that, soon after 1860, high voltage was applied to glass systems to determine the presence of leakage. Besides being sensitive to pressure and chemistry, the discharge tends to enter the system through the leaks, the air in the leak offering a low resistance path.

Nineteenth Century Leak Testing

In previous centuries, in the absence of precise instrumentation for measurement of flow, pressure or chemical concentrations, leak testing had to rely on methods that emphasized detection of gross leak by making the leaking substance more conspicuous and hence making the leakage easier to find.

Natural Gas Pipe Leak Testing

In the 1880s, inventor George Westinghouse patented a means of detecting leakage of fossil gas through gas pipelines. The idea was essentially to encase or sheathe one pipe within another. The zone between the two pipes could then be monitored to detect gas leaking from the interior pipe. As principal owner of utilities and gas delivery systems based in western Pennsylvania, Westinghouse had a strong commercial interest in leak testing.[10]

Smoke Tracer

A leak detection device has a role in the story "A Scandal in Bohemia" in the *Adventures of Sherlock Holmes* (1892) by Arthur Conan Doyle. Sherlock Holmes assumes a disguise and gains admittance to a woman's lodgings to recover love letters compromising to his client. At a prearranged moment, Dr. Watson throws a smoke bomb, called a *plumber's smoke rocket*, in through a window and calls "fire." The lady promptly goes to rescue the love letters, thereby revealing their hiding place. Not *rockets* at all in the modern sense, smoke bombs were used by plumbers who would ignite and put them in piping and ductwork so that smoke would reveal leaks.

Pressure Gages

After the invention of the high voltage sparker in the mid-1800s, no advances in leak detection methods are documented until after the turn of the century. In 1906, Pirani described his hot wire *manometer*, the well known *Pirani* gage. The resistance of an electrically heated wire was measured continuously to determine the temperature of the wire, the temperature increasing with decrease in pressure.

That same year, W. Volge published a description of a hot wire manometer known as the *thermocouple gage* in which the temperature of the wire was indicated by the output of a thermocouple welded to the wire. Both the Pirani and thermocouple gages are affected by the

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residual gases in the vacuum. Accordingly, exposing the system to a gas such as hydrogen, or painting suspected leakage points with liquids such as alcohol or acetone, results in changes of gage output when a leak has actually been covered.

Hot Cathode Gage

There are many gages that can be used as leak detectors because their outputs are functions of the system residual gases. But the most sensitive is the hot cathode ionization gage because it measures the lowest pressures. This was described (but not illustrated) by Oliver E. Buckley in 1916.

It is to be noted that Adolf von Bäyer, in 1909, used both a diode and a triode to measure ionization currents but did not suggest their use as pressure gages. McLeod invented the gage (named after him) in 1874. This gage, and several other gages earlier than the Buckley ionization gage, are not used for leak testing either because they do not have a continuous output or because they are difficult to manufacture and/or use.

Helium Mass Spectrometer Leak Detector

Developed in 1910, the mass spectrometer had as its first achievement the positive confirmation of the existence of isotopes, specifically those of neon. The instrument was improved rapidly so that it became a tool for precision determination of particle mass and relative isotopic abundance. Perhaps its most familiar application is the quantitative and qualitative analysis of chemical compounds and mixtures. However, one of the earliest and presently the largest single application of mass spectrometers is that of the location and measurement of extremely fine leaks.

During the Second World War, the Manhattan Project had been formed in the United States Corps of Engineers to build atomic bombs. An essential part of its assignment was to separate substantial quantities of radioactive uranium-235 from uranium-238, with which it occurs in ores. One approach to this separation was embodied in the diffusion plant built in Oak Ridge, Tennessee.

The plant was to operate on uranium in the form of uranium hexafluoride (UF_6) in the vapor state, and it was realized early on that the process equipment would have to be free from leaks. The lowest pressure in the system was to be about 10 Pa (0.1 atm), so that loss of vacuum was not a concern. First of all, there was the possible outflow of uranium hexafluoride, which is corrosive and poisonous and which would include loss of precious uranium-235. But the real fear, amounting to a nightmare, was the possible inflow of moist air.

The Oak Ridge plant was to consist of acres of diffusion barrier, and the barrier was to be a membrane containing billions of holes of diameters less than 10 nm (4×10^{-7} in.), the mean free path of uranium hexafluoride being about 100 nm (4×10^{-6} in.). Moist air would react with uranium hexafluoride to form uranium oxide in the form of finely divided powder. Conceivably, in the first day of operation of the plant, this powder could clog all the barrier pores, and the most expensive and important war project the United States had ever undertaken would be unsuccessful.

Consequently, a subgroup was set up to determine or develop a suitable hole detection device. The group was headed by Robert B. Jacobs, who was given the task of developing the most sensitive detection system he and his group could devise.

A number of approaches were tried, including the use of a variety of trapped vacuum gages and an optical spectrometer, all of which lacked either the necessary sensitivity and/or selectivity. Jacobs was aware that A.O.C. Nier of the University of Minnesota, Minneapolis, was doing work with a relatively simple type of mass spectrometer of his own design — a 60 degree sector instrument. Nier had used his spectrometer to obtain the first samples of uranium-235 separated from uranium-238.

At Jacobs' behest, Nier devised a leak detector, based on a simplified mass spectrometer gas analyzer, that used a hot filament cathode and was designed to detect helium as a search gas. Helium had been chosen as the leak probe gas because of its very low concentration — one part per 200 000 — in atmospheric air. In theory the spectrometer was selective but actually at the time there were some interferences.

Leak Testing for Efficiency Improvement

The helium leak detector is by far the most sensitive device of its kind. In 1945, its sensitivity was in the neighborhood of 10^{-7} Pa·m^3·s–1 (10^{-6} std cm^3·s^{-1}). This was 100 or more times more sensitive than an ionization gage, the next most sensitive device. Today's mass spectrometer leak detectors can detect flows of 10^{-12} Pa·m^3·s^{-1} (10^{-11} std cm^3·s^{-1}), i.e., leaks 10^5 times smaller than the original models.

While waiting for the mass spectrometer's delivery, a number of

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accessories essential to the reliable use of the instrument were being developed. These included calibrated leaks of the flattened tube type, portable setups for preparing helium-air mixtures of low, known helium concentration, vacuum tight metallic quick connects and pump stations.

When the first few mass spectrometers finally arrived, it was found that each spectrometer was made of glass and included a glass mercury high vacuum pump. The electron emitting filament was fused into the glass tube. Whenever a filament burned out, an expert glassblower was required to crack the filament out of the tube and fit in another with precisely the right orientation. Nevertheless, the units were tested for sensitivity (about 1 part helium in 100 000 parts of air mixture) and sent to project contractors such as Chrysler Corporation.

Although mass spectrometers were typically made of glass then, the leak testing personnel at manufacturing plants during the war were continually burning out the filaments and accidentally breaking the glass tubes. After being chided several times, they finally threatened to quit.

Jacobs was asked to resolve this crisis and came up with the idea of an all metal system that included the spectrometer tube. Nier's reaction was negative because in his experience metal had never been used for the mass spectrometer tube and he could think of a number of reasons why it wouldn't work. At Jacobs' urging, however, the project was undertaken by Nier and his University of Minnesota group.

In a few months, a first model was constructed and worked as well as the original glass one. Moreover, the filament was now mounted into a standard glass male taper. It was a relatively simple job to align this in a companion metal taper mounted on the metal mass spectrometer tube and seal it with vacuum wax. And so the Nier-Keller-General Electric leak detector (Fig. 6) was born. The Nier-Keller prototype was given to General Electric to reengineer and manufacture, and General Electric supplied all the detectors used for diffusion plant testing.

The diffusion plant equipment was designed and constructed along lines laid down by Jacobs' group, to facilitate leak testing. The plant worked, substantial quantities of uranium-235 were prepared, and the leak detector successfully performed its mission. However, rumor had it that leak tightness of the plant did not have to be as extreme as originally thought.

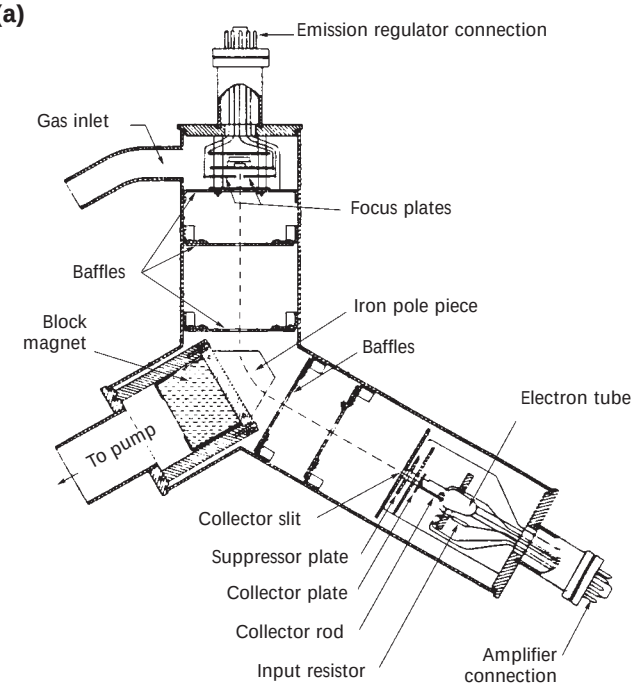
Immediately after the war, leak detectors were being offered to the public and found immediate and widespread application to the testing of electron tubes and to atomic work, the age of the particle accelerator having begun.

Contemporary Leak Detectors

In the years since 1945, the helium detector has undergone somewhat spectacular improvement, although the

FIGURE 6. Nier's helium mass spectrometer leak detector (circa 1944): (a) schematic; (b) photograph.

(a)



(b)

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change may be typical of what happens with any new instrument. In 1945, the sensitivity for helium was about 10 parts helium in 1 million parts of air mixture, or about 10^3 Pa·m^3·s^{-1} (10^4 std cm^3·s^{-1}). By the late 1950s, this figure had gone to about 10^{-9} or 10^{-10} Pa·m^3·s^{-1} (10^{-8} or 10^{-9} std cm^3·s^{-1}). For a number of years now, commercial units have been providing sensitivities better than 10^{-11} Pa·m^3·s^{-1} (10^{-10} std cm^3·s^{-1}). The equivalent parts-per-million figure is 100 to 10 nL·L^{-1}. Obviously, helium in air can now easily be detected.

Size has been reduced even though an extra mechanical pump for roughing has been included in the instrument cabinet. In recent years, several mobile units have been made available. The weight reduction in these units is accomplished in part by eliminating the cold trap and by using a small mechanical pump that acts as both a diffusion pump backer and a test line roughing pump.

The Oak Ridge detector had manually controlled valves. Operator error frequently resulted in admission of atmospheric pressure to the unit, with attendant casualties to the mass spectrometer filament, the pump oil and the system. Models in the 1990s automatically monitor gas admission to the detector and give automatic, digital readout of the leak rate of the defect being probed. Some units require only the depressing of a button to start the detecting task. So-called *industrial* leak testing systems are available for testing mass produced components. The operator needs only to place the test object into a rack and press a *start* button. The system operates automatically and flashes a *go* or *no-go* signal at the end of the test.

Helium mass spectrometer leak detectors became commercially available in the United States in the late 1940s. The versatility of mass spectrometer instruments has led to a wide variety of applications. Presently, thousands of these sensitive leak detectors are in use throughout the world. Leak detectors are found in almost every university, industrial or government physics laboratory.

Thanks to these historic developments, a tremendous amount of time has been saved in leak testing operations. Whereas days and even weeks were spent in finding leaks in laboratory high vacuum setups, the helium detector made it possible to locate the leaks in hours or minutes. Nier will probably be most remembered in the annals of physics for his work in mass spectroscopy but the scientific world is more in his debt for the leak detector.

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PART 4. Units of Measure for Nondestructive Testing

Origin and Use of the SI System

In 1960 the General Conference on Weights and Measures devised the International System of Units. *Le Systeme Internationale d'Unites* (SI) was designed so that a single set of interrelated measurement units could be used by all branches of science, engineering and the general public. Without SI, this *Nondestructive Testing Handbook* volume could have contained a confusing mix of Imperial units, obsolete centimeter-gram-second (cgs) metric system version units and the units preferred by certain localities or scientific specialties.

SI is the modern version of the metric system and ends the division between metric units used by scientists and metric units used by engineers and the public. Scientists have given up their units based on centimeter and gram and engineers made a fundamental change in abandoning the kilogram-force in favor of the newton. Electrical engineers have retained their amperes, volts and ohms but changed all units related to magnetism. The main effect of SI has been the reduction of conversion factors between units to *one* (1) — in other words, to eliminate them entirely.

Table 6 lists seven base units. Table 7 lists derived units with special names. Table 8 gives examples of conversions to SI units. In SI, the unit of time is the second (s) but hour (h) is recognized for use with SI.

For more information, the reader is referred to the information available through national standards organizations and specialized information compiled by technical organizations.[11-13]

TABLE 6. Base SI units.

Quantity	Unit	Symbol
Length	meter	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Temperature[a]	kelvin	K
Amount of substance	mole	mol
Luminous intensity	candela	cd

a. Kelvin can be expressed in degrees celsius (°C = K – 273.15).

Multipliers

Very large or very small numbers with units are expressed by using the SI multipliers, prefixes of 10^3 intervals (Table 9) in science and engineering. The multiplier becomes a property of the SI unit. For example, a millimeter (mm) is 0.001 meter (m). The volume unit cubic centimeter (cm^3) is $(0.01)^3$ or 10^{-6} m^3. Unit submultiples such as the centimeter, decimeter, dekameter (or decameter) and hectometer are avoided in scientific and technical uses of SI because of their variance from the 10^3 interval. However, dm^3 and cm^3 are in use specifically because they represent a 10^3 variance.

TABLE 7. Derived SI units with special names.

Quantity	Units	Symbol	Relation to Other SI Units[a]
Frequency (periodic)	hertz	Hz	$1 \cdot s^{-1}$
Force	newton	N	$kg \cdot m \cdot s^{-2}$
Pressure (stress)	pascal	Pa	$N \cdot m^{-2}$
Energy	joule	J	$N \cdot m$
Power	watt	W	$J \cdot s^{-1}$
Electric charge	coulomb	C	$A \cdot s$
Electric potential[b]	volt	V	$W \cdot A^{-1}$
Capacitance	farad	F	$C \cdot V^{-1}$
Electric resistance	ohm	Ω	$V \cdot A^{-1}$
Conductance	siemens	S	$A \cdot V^{-1}$
Magnetic flux	weber	Wb	$V \cdot s$
Magnetic flux density	tesla	T	$Wb \cdot m^{-2}$
Inductance	henry	H	$Wb \cdot A^{-1}$
Luminous flux	lumen	lm	$cd \cdot sr$
Illuminance	lux	lx	$lm \cdot m^{-2}$
Plane angle	radian	rad	1
Radioactivity	becquerel	Bq	$1 \cdot s^{-1}$
Radiation absorbed dose	gray	Gy	$J \cdot kg^{-1}$
Radiation dose equivalent	sievert	Sv	$J \cdot kg^{-1}$
Solid angle	steradian	sr	1
Time	hour	h	60 s
Volume[c]	liter	L	dm^3

a. Number one expresses dimensionless relationship.
b. Electromotive force.
c. The only prefixes that may be used with liter are milli (m) and micro (μ).

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Note that 1 cm^3 is not equal to 1/100 m^3. Also, in equations, submultiples such as centimeters (cm) or decimeters (dm) should be avoided because they disturb the convenient 10^3 or 10^{-3} intervals that make equations easy to manipulate.

In SI, the distinction between upper and lower case letters is meaningful and should be observed. For example, the meanings of the prefix m (milli) and the prefix M (mega) differ by nine orders of magnitude.

SI Units for Leak Testing

Pressure

The pascal (Pa), equal to one newton per square meter (1 $N \cdot m^{-2}$), is used to measure pressure, stress etc. It is used in place of units of pound force per square inch ($lb_f \cdot in.^{-2}$), atmosphere, millimeter of mercury (mm Hg), torr, bar, inch of mercury (in. Hg), inch of water (H_2O) and other units (see Table 10). The text must indicate whether *gage, absolute* or *differential* pressure is meant. Negative pressures might be used in heating duct technology and in vacuum boxes used for bubble testing, but in vacuums as used in tracer leak testing absolute pressures are used.

Volume

The cubic meter (m^3) is the only volume measurement unit in SI. It takes the place of cubic foot, cubic inch, gallon, pint, barrel and more. In SI, the liter (L) is also approved for use. The liter is a special name for cubic decimeter (1 L = 1 dm^3 = 10^{-3} m^3). Only the *milli* (m) and *micro* (μ) prefixes may be used with liter.

The fundamental units of time, temperature, pressure and volume are expressed every time a leakage is measured.

Units for Measurement of Radioactive Tracer Gases

The original curie was simply the radiation of one gram of radium. Eventually all equivalent radiation from any source was measured with this same unit. The original roentgen was the quantity of radiation that would ionize one cubic centimeter of air to one electrostatic unit of electricity of either sign. It is now known that a curie is equivalent to 3.7×10^{10} disintegrations per second and a roentgen is equivalent to 258 microcoulomb per kilogram (258 $\mu C \cdot kg^{-1}$) of air. This corresponds to 1.61×10^{15} ion pairs per kilogram of air that has absorbed 8.8 millijoule (mJ) or 0.88 rad.

In SI, radiation units have been given established physical foundations and new names where necessary. The unit for radioactivity (formerly curie) is the becquerel (Bq), defined as one disintegration per second.

Derived SI Units

Gas Quantity. Pascal cubic meter ($Pa \cdot m^3$). The quantity of gas stored in a container or which has passed through a leak is described by the derived SI unit of pascal

TABLE 8. Examples of conversions to SI units

Quantity	Measurement in Non-SI Unit	Multiply by	To Get Measurement in SI Unit
Area	square inch ($in.^2$)	645	square millimeter (mm^2)
Distance	angstrom (Å)	0.1	nanometer (nm)
	inch (in.)	25.4	millimeter (mm)
Energy	British thermal unit (BTU)	1.055	kilojoule (kJ)
	calorie (cal), thermochemical	4.184	joule (J)
	British thermal unit per hour ($BTU \cdot h^{-1}$)	0.293	watt (W)
Specific heat	British thermal unit per pound per degree Fahrenheit ($BTU \cdot lb_m^{-1} \cdot °F^{-1}$)	4.19	kilojoule per kilogram per kelvin ($kJ \cdot kg^{-1} \cdot K^{-1}$)
Force (torque, couple)	foot-pound ($ft\text{-}lb_f$)	1.36	joule (J)
Force or pressure	pound force per square inch ($lb_f \cdot in.^{-2}$)	6.89	kilopascal (kPa)
Frequency (cycle)	cycle per minute	1/60	hertz (Hz)
Illuminance	footcandle (ftc or fc)	10.76	lux (lx)
Luminance	candela per square foot ($cd \cdot ft^{-2}$)	10.76	candela per square meter ($cd \cdot m^{-2}$)
	candela per square inch ($in. \cdot ft^{-2}$)	1 550	candela per square meter ($cd \cdot m^{-2}$)
	footlambert	3.426	candela per square meter ($cd \cdot m^{-2}$)
	lambert	3 183 (= 10 000/π)	candela per square meter ($cd \cdot m^{-2}$)
Radioactivity	curie (Ci)	37	gigabecquerel (GBq)
Ionizing radiation exposure	roentgen (R)	0.258	millicoulomb per kilogram ($mC \cdot kg^{-1}$)
Mass	pound (lb_m)	0.454	kilogram (kg)
Temperature (difference)	degree fahrenheit (°F)	0.556	degree celsius (°C)
Temperature (scale)	degree fahrenheit (°F)	(°F – 32)/1.8	degree celsius (°C)
			(°F – 32)/1.8) + 273.15 kelvin (K)

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cubic meter, the product of pressure and volume. To be strict, the temperature should be specified for the gas quantity or leakage measurement to define the gas quantity (sometimes loosely described as the mass of gas) more precisely. Often, gas quantity is defined for standard temperature and pressure, typically the standard atmospheric pressure of 100 kPa (1 atm) and a temperature of 0 °C (32 °F). Temperature corrections are usually required if temperature varies significantly during leak testing. However, small changes in temperature may sometimes be insignificant compared with many orders of magnitude of change in gas pressure or leakage quantity.

Gas Leakage Rate. Pascal cubic meter per second ($Pa \cdot m^3 \cdot s^{-1}$). The leakage rate is defined as the quantity (mass) of gas leaking in one second. The unit in prior use was the standard cubic centimeter per second (std $cm^3 \cdot s^{-1}$). Use of the word *standard* in units such as std $cm^3 \cdot s^{-1}$ requires that gas leakage rate be converted to standard temperature and pressure conditions (293 K and 101.325 kPa), often even during the process of collecting data during leakage rate tests. Leakage rates given in SI units of $Pa \cdot m^3 \cdot s^{-1}$ can be converted to units of std $cm^3 \cdot s^{-1}$ at any time by simply multiplying the SI leakage rate by 10 or (more precisely) by 9.87.

Gas Permeation Rate. Pascal cubic meter per second per square meter per meter $(Pa \cdot m^3 \cdot s^{-1})/(m^2 \cdot m^{-1})$. Permeation is the leakage of gas through a (typically solid) substance that is not impervious to gas flow. The permeation rate is larger with an increased exposed area, a higher pressure differential across the substance (membrane, gasket etc.) and is smaller with an increasing thickness of permeable substance. In vacuum testing, the pressure differential is usually considered to be one atmosphere (about 100 kPa). One sometimes finds units of permeation rate where the gas quantity is expressed in units of mass and where the differential pressure is expressed in various units. Equation 1 expresses an equivalence for conversion of measurements:

$$\textbf{(1)} \qquad 1.0 \frac{\text{std cm}^3 \cdot \text{s}^{-1}}{\text{cm}^2 \cdot \text{cm}^{-1}} \cong 0.1 \frac{\text{Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}}{\text{m}^2 \cdot \text{m}^{-1}}$$

Rounding

Many tables and graphs were obtained from researchers and scientists who did their work in the English system. In the

TABLE 9. SI multipliers.

Prefix	Symbol	Multiplier
yotta	Y	10^{24}
zetta	Z	10^{21}
exa	E	10^{18}
peta	P	10^{15}
tera	T	10^{12}
giga	G	10^{9}
mega	M	10^{6}
kilo	k	10^{3}
hecto[a]	h	10^{2}
deka (or deca)[a]	da	10
deci[a]	d	10^{-1}
centi[a]	c	10^{-2}
milli	m	10^{-3}
micro	μ	10^{-6}
nano	n	10^{-9}
pico	p	10^{-12}
femto	f	10^{-15}
atto	a	10^{-18}
zepto	z	10^{-21}
yocto	y	10^{-24}

a. Avoid these prefixes (except in dm^3 and cm^3) for science and engineering.

TABLE 10. Conversion factors for pressure values.

To Convert From	To	Multiply by
pascal (Pa)	$lb_f \cdot in.^{-2}$	1.4504×10^{-4}
	$kg \cdot mm^{-2}$	1.0197×10^{-7}
	atm	9.8692×10^{-6}
	in. Hg	2.9530×10^{-4}
	torr	7.5006×10^{-3}
pound per square inch ($lb_f \cdot in.^{-2}$)	Pa	6.8948×10^{3}
	$kg \cdot mm^{-2}$	7.0307×10^{-4}
	atm	6.8046×10^{-2}
	in. Hg	2.0360
	torr	51.715
kilogram per square millimeter ($kg \cdot mm^{-2}$)	Pa	9.8066×10^{5}
	$lb_f \cdot in.^{-2}$	1.4223×10^{3}
	atm	96.784
	in. Hg	2.8959×10^{3}
	torr	7.3556×10^{4}
atmosphere (atm)	Pa	1.01325×10^{5}
	$lb_f \cdot in.^{-2}$	14.696
	$kg \cdot mm^{-2}$	1.0332×10^{-2}
	in. Hg	29.921
	torr	760.0
inch mercury (in. Hg)	Pa	3.3864×10^{3}
	$lb_f \cdot in.^{-2}$	4.9115×10^{-1}
	$kg \cdot mm^{-2}$	3.4532×10^{-4}
	atm	3.3421×10^{-2}
	torr	25.40
torr	Pa	1.3332×10^{2}
	$lb_f \cdot in.^{-2}$	1.9337×10^{-2}
	$kg \cdot mm^{-2}$	1.3595×10^{-5}
	atm	1.3158×10^{-3}
	in. Hg	3.9370×10^{-2}

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conversion, some numbers have been rounded drastically but some were left as irrational numbers, especially where quotes were made to specific entries.

Quantitative Description of Leakage Rates

The significant quantitative measurement resulting from leak testing is the volumetric leakage rate or mass flow rate of fluid through one or more leaks. Leakage rate thus has dimensions equivalent to pressure times volume divided by time. The units used previously for volumetric leakage rate were standard cubic centimeter per second (std $cm^3 \cdot s^{-1}$). The *Nondestructive Testing Handbook* uses the international standard SI nomenclature. In SI units, the quantity of gas is measured in units of pascal cubic meter ($Pa \cdot m^3$). The leakage rate is measured in pascal cubic meter per second ($Pa \cdot m^3 \cdot s^{-1}$). For this SI leakage rate to be a mass flow, the pressure and temperature must be at standard values of 101 kPa (760 torr) and 0 °C (32 °F). Table 11 gives factors for conversion of leakage rates in various common units, past and present. Table 12 provides leakage rate comparisons that permit a better understanding of the quantities involved, when leakage rates are specified.

TABLE 11. Mass flow conversion factors for leakage rate.

To Convert from	To	Multiply by
Pascal cubic meter per second ($Pa \cdot m^3 \cdot s^{-1}$)	std $cm^3 \cdot s^{-1}$	9.87 (≅ 10)
	$mol \cdot s^{-1}$	4.40×10^{-4}
	$torr \cdot L \cdot s^{-1}$	7.50
	$mb \cdot L \cdot s^{-1}$	1.00×10^{1}
	std $ft^3 \cdot h^{-1}$	1.25
Standard cubic centimeter per second (std $cm^3 \cdot s^{-1}$)	$Pa \cdot m^3 \cdot s^{-1}$	1.01×10^{-1}
	$mol \cdot s^{-1}$	4.46×10^{-5}
	$torr \cdot L \cdot s^{-1}$	7.60×10^{-1}
	$mb \cdot L \cdot s^{-1}$	1.01
	std $ft^3 \cdot h^{-1}$	1.27×10^{-1}
Mole per second ($mol \cdot s^{-1}$)	$Pa \cdot m^3 \cdot s^{-1}$	2.27×10^{3}
	std $cm^3 \cdot s^{-1}$	2.24×10^{4}
	$torr \cdot L \cdot s^{-1}$	1.70×10^{4}
	$mb \cdot L \cdot s^{-1}$	2.27×10^{5}
	std $ft^3 \cdot h^{-1}$	2.85×10^{3}
Torr liter per second ($torr \cdot L \cdot s^{-1}$)	$Pa \cdot m^3 \cdot s^{-1}$	1.33×10^{-1}
	std $cm^3 \cdot s^{-1}$	1.32
	$mol \cdot s^{-1}$	5.87×10^{-5}
	$mb \cdot L \cdot s^{-1}$	1.33
	std $ft^3 \cdot h^{-1}$	1.67×10^{-1}
Millibar liter per second ($mb \cdot L \cdot s^{-1}$)	$Pa \cdot m^3 \cdot s^{-1}$	1.00×10^{-1}
	std $cm^3 \cdot s^{-1}$	9.87×10^{-1}
	$mol \cdot s^{-1}$	2.27×10^{4}
	$torr \cdot L \cdot s^{-1}$	7.50×10^{-1}
	std $ft^3 \cdot h^{-1}$	1.26×10^{-1}
Standard cubic foot per hour (std $ft^3 \cdot h^{-1}$)	$Pa \cdot m^3 \cdot s^{-1}$	0.80
	std $cm^3 \cdot s^{-1}$	7.87
	$mol \cdot s^{-1}$	3.51×10^{-4}
	$torr \cdot L \cdot s^{-1}$	5.99
	$mb \cdot L \cdot s–1$	7.94

Leakage is not simply the volume of air entering the vacuum chamber. Instead, the critical factor is the number of gaseous molecules entering the vacuum system. This number of molecules, in turn, depends on the external pressure, temperature and the volume of gas at this pressure that leaks into the vacuum system. The leakage rate is expressed in terms of the product of this pressure difference multiplied by the gas volume passing through the leak, per unit of time. Thus, the leakage rate is directly proportional to the number of molecules leaking into the vacuum system per unit of time.

The molecular unit of mass flow used for gas by the National Institute of Standards and Technology is mole per second ($mol \cdot s^{-1}$), a mass flow unit measured at standard atmospheric pressure and standard temperature of 0 °C (32 °F). A common unit of gas is the standard cubic meter (std m^3). This unit is equivalent to one million units given as atmospheric cubic centimeter (atm cm^3). Both units indicate the quantity of gas (air) contained in a unit volume at average sea level atmospheric pressure at a temperature of 0 °C (32 °F). The average atmospheric pressure at sea level is 101.3 kPa (760 mm Hg or 760 torr). The SI unit of pressure, the pascal (Pa), is equivalent to newton per square meter ($N \cdot m^{-2}$).

Non-SI Units Used Earlier for Measurement of Leakage

Various units have been used for measurement of leakage, including many related to English units commonly used in engineering in the United States. Justification for prior use of this diversity of units lies in the relative ease with which these common units can be adapted for many practical engineering problems. For example, suppose that an operator has a gas cylinder with a pressure gage calibrated in units of pound-force per square inch ($lb_f \cdot in.^{-2}$). With daily gage readings, it is convenient for the operator to express leakage as the gage pressure change multiplied by cylinder volume, divided by the leakage time period (days). This simple calculation results in leakage rate measurement in units of $lb_f \cdot in.^{-2}$ ft^3 per day. This leakage rate has dimensions of (pressure) × (volume) ÷ (time). To have expressed the leakage merely as the

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volume of gas lost is insufficient because the volume of gas that leaves daily at high cylinder pressure will be considerably larger than the volume leaking to the atmosphere each day when the internal pressure of the cylinder is lower. Many combinations of units for pressure, volume and time are possible. The SI volumetric leakage rate unit pascal cubic meter per second ($Pa \cdot m^3 \cdot s^{-1}$) is used in this book.

Units for Leakage Rates of Vacuum Systems

Suppose that leakage of air into a vacuum system has an undesired effect on the pressure within the vacuum system. The operator of the vacuum system can read absolute pressures in pascal or torr from gages permanently installed in the system. (The pressure unit known as a torr is defined as 1/760th of a standard atmosphere and differs only by one part in seven million from the well known barometric pressure unit of millimeter mercury.) In the past, the leakage rate in vacuum systems was measured in torr liter per second. If the volume of the vacuum chamber had been measured in cubic meter, the operator might find it easier to measure leakage rate in units of pascal cubic meter per day or per second.

Leakage is not simply the volume of air entering the vacuum chamber. Instead, the critical factor is the number of gaseous molecules entering the vacuum system. This number of molecules, in turn, depends on the external pressure, temperature and the volume of gas at this pressure that leaks into the vacuum system. The leakage rate is expressed in terms of the product of this pressure difference multiplied by the gas volume passing through the leak, per unit of time. Thus, the leakage rate is directly proportional to the number of molecules leaking into the vacuum system per unit of time.

TABLE 12. Leakage rates expressed in various units

$Pa \cdot m^3 \cdot s^{-1}$	std $cm^3 \cdot s^{-1}$	$mol \cdot s^{-1}$
1	10	4.40×10^{-4}
10^{-1}	1	4.40×10^{-5}
10^{-2}	10^{-1}	4.40×10^{-6}
10^{-3}	10^{-2}	4.40×10^{-7}
10	10^{-3}	4.40×10^{-8}
10^{-5}	10^{-4}	4.40×10^{-9}
10^{-6}	10^{-5}	4.40×10^{-10}
10^{-7}	10^{-6}	4.40×10^{-11}
10^{-8}	10^{-7}	4.40×10^{-12}
10^{-9}	10^{-8}	4.40×10^{-13}
10^{-10}	10^{-9}	4.40×10^{-14}

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2

CHAPTER

Tracer Gases in Leak Testing[1]

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PART 1. Introduction to Properties of Tracer Gases for Leak Testing

Fluid Media Used in Leak Testing

Leak testing can be divided into three categories: (1) leak detection, (2) leak location and (3) leakage measurement. Each involves a fluid leak tracer and some means for establishing a pressure differential or other means to make fluid flow through the leak or leaks. Possible fluid probing media include gases, vapors, liquids or combinations of these. Selection of the desired fluid probing medium for leak testing depends on operator or engineering judgment and involves factors such as:

1. type and size of test object or system to be tested;
2. typical operating conditions of test object or system;
3. environmental conditions during leak testing;
4. hazards associated with the probing medium and the pressure involved in testing;
5. leak testing instrumentation and its response to the probing medium; and
6. leakage rates that must be detected and the accuracy with which measurements must be made.

Where high sensitivity to leakage must be attained, gases and vapors are generally preferred to liquid media. The present discussion is devoted specifically to gaseous tracers used in leak testing. Special gaseous tracers are discussed elsewhere in this volume. Liquid probing media are used for leak testing in many applications.

Volumes Occupied by Gases and Liquids

The volume of any substance is the space occupied by that substance. For gases, the volume of a sample of gas is the same as the volume of the container within which the gas is held. The volume occupied by liquids or by solids does not change very much with a change in pressure or temperature. Therefore, to describe the amount of a solid or of a liquid, it is usually sufficient to specify only the volume of the sample. However, this cannot be done with gases. For example, 1 m^3 of gaseous helium at a certain temperature and pressure will have a different gas density and mass than would 1 m^3 of gaseous helium at different temperature and pressure conditions. To determine the quantity of a given volume of gas, it is necessary to know its pressure and temperature. When liquids are mixed together, the total volume is roughly equal to the sums of the original volumes. However, this is not necessarily true for mixtures of gases. Gases can mix in any proportions and still fill the volumes within which they are mixed.

Pressures Exerted by Gases or Liquids

Fluid pressure is defined as a force per unit area. In liquids and gases, the pressure at a given point is the same in all directions. In general, for all gases and liquids, the greater the depth of immersion, the greater the internal pressure. These effects can be illustrated by considering a swimmer under water. At a given depth, the pressure exerted on the body is the same no matter how the swimmer turns. This is due to the pull of gravity on the water above. The body is subject to pressure because it must support the weight of water above the swimmer.

The earth is surrounded by a blanket of air several hundred kilometers thick. People live at the bottom of this ocean of air, which exerts atmospheric pressure. The force per unit area exerted on the earth's surface is equal to the weight of the column of air above it, 100 kPa (14.7 $lb_f \cdot in.^{-2}$). This pressure also corresponds to the weight of a column of mercury 760 mm high, or 760 torr. The mercury barometer balances the weight of its column of mercy against the weight per unit area of the earth's atmosphere. At sea level, the pressure is typically near 100 kPa (14.7 $lb_f \cdot in.^{-2}$). The pressure is reduced as the altitude increases, so the barometer can also be used as an altimeter. The atmospheric pressure also changes from day to day as cold, dense air masses are replaced by less dense warm air masses and vice versa. Thus, care must be taken to exclude the effects of local changes in atmospheric pressure from leak testing measurements or to correct for their effects.

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Pressures can be measured in atmospheres (atm) with respect to zero pressure (absolute pressures) or normal atmospheric pressure (gage pressures). In general, gas pressure is a measure of the work done to compress gas into a unit volume. The change in energy W stored in gas under pressure within a container is related to the product P of its pressure and its volume V, as in Eq. 1:

$$W = PV \quad (1)$$

where P is absolute pressure of gas (pascal), V is volume of gas (cubic meter) and W is stored energy (joule).

Boyle's Law Relating Pressure and Volume of Gases at Constant Temperature

A characteristic property of gases is that they are easily compressed. This behavior is described by Boyle's law (1662), which states that, at constant temperature, a fixed mass of gas occupies a volume inversely proportional to the pressure exerted on it. If the pressure is doubled, the volume becomes half as large (Fig. 1). Boyle's law is expressed by Eq. 2:

$$P_i V_i = P_f V_f \quad (2)$$

In Eq. 2, the subscripts i and f denote the initial and final conditions, respectively, of the fixed quantity or mass of gas.

Charles' Law Relating Temperature and Volume of Gases under Constant Pressure

Like most substances, gases increase in volume when their temperature is raised. This increase in volume with increasing temperature can be observed experimentally with the arrangement sketched in Fig. 2. If the force on top of the piston is constant, the gas sample remains at constant pressure P. If the gas is heated, the piston moves out and the volume V of gas beneath it increases. This behavior is expressed by Charles' law (1787), which states that, at constant pressure, the volume occupied by a fixed mass of gas is directly proportional to the absolute (kelvin) temperature of the gas. Mathematically, Charles's law is expressed by Eq. 3:

$$\frac{V_i}{V_f} = \frac{T_i}{T_f} \quad (3)$$

where the subscripts i and f denote the initial and the final conditions, respectively. In Eq. 3, the temperature T must always be expressed in units of absolute temperature (kelvin or degree rankine).

Variations of temperature of contained gases during leak testing could lead to erroneous interpretations of leak testing data if the effects of Charles's law were ignored. Thus, it is desirable to make leak tests during periods of reasonably constant temperature, if possible, and to correct for test temperature variations during data analysis to ensure valid interpretations and measurements of leakage.

Dalton's Law of Partial Pressures of Mixed Gases

The behavior observed when two or more gases are placed within the same container is summarized in Dalton's law

FIGURE 1. Boyle's law experiment showing volume decrease of gas when pressure increases, at constant temperature.

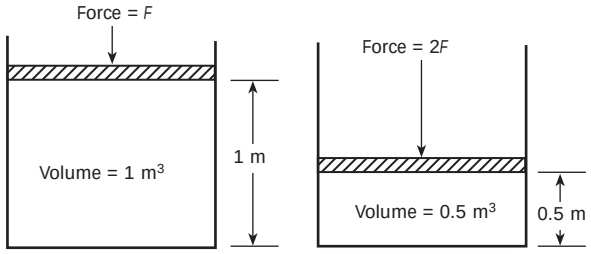
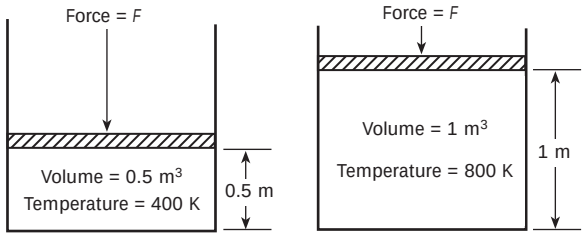


FIGURE 2. Charles' law experiment showing volume increase with temperature, in gas at constant pressure.



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of partial pressures (1801), which states that the total pressure exerted by a mixture of gases is equal to the sum of the partial pressures of the various gases. The partial pressure of a gas in a mixture is defined as the pressure the specific gas would exert if it were alone in the container. The meaning of Dalton's law is indicated by the sketch of Fig. 3. One cubic meter (1.0 m^3 or 35 ft^3) of nitrogen at a pressure of 50 kPa (7.25 $lb_f \cdot in^{-2}$) and 1.0 m^3 (35 ft^3) of oxygen at a pressure of 70 kPa (10.15 $lb_f \cdot in^{-2}$) would exert a total pressure of 120 kPa (17.40 $lb_f \cdot in^{-2}$) if the two gases were mixed and contained within a volume of 1.0 m^3 (35 ft^3). For the general case, Dalton's law can be expressed by Eq. 4:

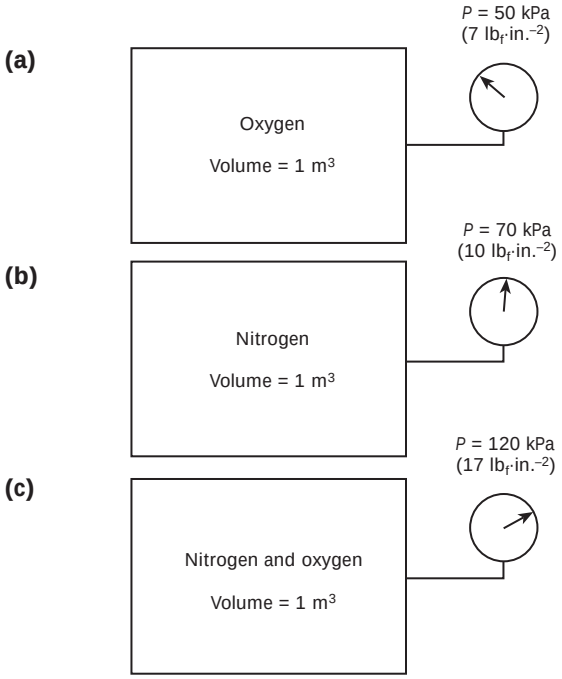
$$P_{total} = P_1 + P_2 + P_3 + \ldots P_n \quad (4)$$

where the numerical subscripts indicate the partial pressures due to each gas constituent.

Avogadro's Principle Describing Number of Gas Molecules in a Volume

Amedeo Avogadro in 1811 was the first to propose the principle now known as Avogadro's principle. It states that equal volumes of gases at the same temperature and pressure contain equal numbers of gas molecules. Through modern techniques it has been possible to make the following observation concerning the average number of gas molecules in one mole of gas. A mole is the amount of gas whose weight in gram equals its atomic mass. Avogadro's number of 6×10^{23} molecules (a mole) is the number of gas molecules that would occupy a volume of 22.4 L (0.79 ft^3) at standard temperature and pressure. Standard temperature is designated at 0 °C (32 °F), the freezing point of water; standard pressure is defined as 100 kPa (1 atm). This standard pressure was originally based on the atmospheric pressure that will support a column of mercury 760 mm in height, which corresponds to the mean atmospheric pressure at sea level. According to Avogadro's principle, the volume that a gas sample occupies at standard temperature and pressure is directly proportional to the number of gas molecules within that gas sample.

FIGURE 3. Dalton's law experiment showing total pressure to equal sum of partial pressures of mixed gases injected into a fixed volume: (a) oxygen; (b) nitrogen; (c) combined pressure of same quantitites of nitrogen and oxygen combined.



General Gas Law Applicable to All Ideal Gases and Mixtures of Ideal Gases

Boyle's law, Charles' law and Avogadro's principle can be combined to give a general relationship between volume V, pressure P, temperature T and the number of moles of gas m in a gas sample. The general gas law can be applied without the necessity of maintaining one of these variables constant. Boyle's law states that the volume occupied by a gas is inversely proportional to the gas pressure. Charles' law states that the gas volume is directly proportional to the gas temperature. Avogadro's principle states that the volume is directly proportional to the total number of gas molecules contained in that volume (regardless of the species of the individual molecules). These relationships are summarized in Eqs. 5 through 8, in which the symbol ≅ means "is proportional to": Boyle's law,

$$V \cong \frac{1}{P} \quad (5)$$

with constant T and m; Charles' law,

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(6) $V \cong T$

with constant P and m; Avogadro's principle,

(7) $V \cong m$

with constant T and P; and a General relationship,

(8) $V \cong \frac{mT}{P}$

without restriction.

The general relationship of Eq. 8 combines the individual relationships of Eq. 5, 6 and 7. This can be seen by imagining that any two of the variables, such as T and m, are constant and noting the relation of the other two variables.

The general ideal gas law (applicable to all ideal gases) can be written in the form of Eq. 9:

(9) $PV = mRT$

Here, R (in $J \cdot mol^{-1} \cdot K^{-1}$) is the universal gas constant found from known values of P, V, m and T by Avogadro's principle, by use of EQ. 10:

(10) $R = \frac{PV}{mT} = 8.314$

The individual gas constant R_i ($J \cdot kg^{-1} \cdot K^{-1}$) is obtained by dividing the universal gas constant R (joule) by the molecular mass M (kilogram) of the specific gas involved, by use of Eq. 11:

(11) $R_i = \frac{R}{M} = \frac{PV}{mMT}$

The numerical value of the individual gas constant for several common tracer gases is given in Table 1. The behavior of real gases conforms closely to the Ideal gas law of Eq. 9 under a wide range of conditions. It begins to deviate from this ideal gas law only as gas densities become much higher than those usually used in leak testing.

However, the behavior of vapors, including several types of vapors used in leak testing, can deviate significantly from the relation of the Ideal gas law. Thus, care is required in computing leakage rates by the ideal gas law relationship when the pressurizing gas or leak tracer is a vapor or contains a large proportion of vapor constituent. (A vapor is the gaseous form of any substance that usually exists in the form of a liquid or a solid, such as water vapor. A pure liquid in equilibrium with its own vapor will have two phases, liquid and vapor, which coexist at a specific partial pressure known as the vapor pressure. Because condensation or evaporation can occur, vapor molecules can enter or leave the gaseous phase. This changes the number of molecules of that vapor species that will be present within a particular gas volume.) These vapor effects are not included in the general gas law relationship of Eqs. 9 to 11.

Graham's Law for Diffusion of Gases

A gas expands to occupy the volume within which it is contained. If a bottle of ammonia is opened at one end of a room, it is soon detected by odor at the other end of the room. This spreading of a gas constituent through other gaseous

TABLE 1. Physical properties of typical gases and vapors at 15 °C (59 °F).

Gas	Chemical Symbols	Molecular Weight	Molecular Diameter (pm)	Viscosity (μPa·s)	Gas Constant, ($J \cdot kg^{-1} \cdot K^{-1}$)
Air		29.00		18.0	287
Ammonia	NH_3	17.03	297.0	9.7	488
Argon	Ar	39.94	288.0	22.0	208
Carbon dioxide	CO_2	44.01	334.0	14.5	189
Dichlorodifluoromethane	CCl_2F_2	120.93		12.7	68.8
Helium	He	4.00	190.0	19.2	2079
Hydrochloric acid	HCl	36.50		14.0	228
Hydrogen	H_2	2.02	240.0	8.6	4116
Krypton	Kr	83.80		24.6	9.92
Methane	CH_4	16.04		10.7	518
Neon	Ne	20.18		31.0	412
Nitrogen	N_2	28.01	315.0	17.3	297
Nitrous oxide	N_2O	44.00		14.3	189
Oxygen	O_2	31.99	298.0	19.9	260
Sulfur dioxide	SO_2	64.00		12.3	130
Water vapor	H_2O	18.02	460.0	9.3	461

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constituents within a volume is called diffusion. Under fixed conditions, it is found that lighter gases diffuse more rapidly than the heavier gases. Graham's law of diffusion states, The rates of diffusion of different gases are inversely proportional to their individual molecular masses. Graham's law can be written mathematically in the form of Eq. 12:

$$\frac{D_1}{D_2} = \frac{M_1}{M_2} \quad (12)$$

where D_1 and D_2 are the rates of diffusion of gases 1 and 2 and where M_1 and M_2 are the respective molecular masses of these two different gases.

A leak testing tracer gas with low diffusivity provides an advantage in detector probe leak detection techniques because the concentration of the tracer gas builds up at the leak exit. This allows detection by a probe to locate the site of a leak. With low diffusivity, the tracer gas does not leave the leak location rapidly.

A tracer gas of high diffusivity is needed for internal pressurization where it is necessary to fill cul-de-sacs or blind passageways within a reasonable soak time before testing. A low diffusion rate would not allow a tracer gas to traverse a tortuous leak passage, thus making leak detection by tracer gas an unreliable procedure. Table 2 lists the diffusivities of typical tracer gases in air at standard

TABLE 2. Diffusivities of tracer gases in air at standard temperature of 0 °C (32 °F) and standard pressure of 100 kPa (760 torr). (Diffusion coefficient values are calculated from an empirical equation, after Slattery.[2])

Gas	Formula	Molecular Mass ($g \cdot mol^{-1}$)	Difffusion Coefficient	
			$mm^2 \cdot s^{-1}$	$(ft^2 \cdot h^{-1})$
Acetylene	C_2H_2	26.0	14.2	(0.55)
Ammonia	NH_3	17.0	17.0	(0.66)
Argon	Ar	39.9	14.7	(0.61)
Benzene	C_6H_6	78.1	7.7	(0.30)
Butane	C_4H_{10}	58.1	8.5	(0.33)
Carbon dioxide	CO_2	44.0	13.4	(0.52)
Carbon disulfide	CS_2	76.1	9.3	(0.36)
Carbon monoxide	CO	28.0	17.3	(0.67)
Carbon tetrachloride	CCl_4	154.0	7.2	(0.28)
Dichloromethane	CH_2Cl_2	84.93	7.4	(0.29)
Ethane	C_2H_6	30.1	12.6	(0.49)
Ethyl alcohol	C_2H_5OH	46.1	9.8	(0.38)
Ethylene	C_2H_4	28.0	13.4	(0.52)
Refrigerant–11	CCl_3F	137.0	7.7	(0.30)
Refrigerant–12	CCl_2F_2	121.0	8.3	(0.32)
Refrigerant–21	$CHCl_2F$	103.0	8.5	(0.33)
Refrigerant–22	$CHClF_2$	86.5	9.5	(0.37)
Refrigerant–112	$CCl_2F–CCl_2F$	204.0	6.5	(0.25)
Refrigerant–114	$CClF_2–CClF_2$	171.0	7.2	(0.28)
Refrigerant-134a	$C_2H_2F_4$	102.0	7.2	(0.28)
Helium	He	4.0	69.7	(2.70)
Hydrogen	H_2	2.0	67.1	(2.60)
Hydrogen sulfide	H_2S	24.1	13.7	(0.53)
Krypton	Kr	83.8	13.2	(0.51)
Methane	CH_4	16.0	18.6	(0.72)
Neon	Ne	20.2	28.4	(1.10)
Nitric oxide	NO	30.0	18.1	(0.70)
Nitrogen	N_2	28.0	17.5	(0.68)
Nitrous oxide	N_2O	44.0	13.4	(0.52)
Oxygen	O_2	32.0	17.5	(0.68)
Propane	C_3H_8	44.1	10.0	(0.39)
Sulfur dioxide	SO_2	64.1	10.8	(0.42)
Sulfur hexafluoride	SF_6	146.0	7.3	(0.28)
Water	H_2O	18.0	21.9	(0.85)
Xenon	Xe	131.0	10.8	(0.42)

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conditions of 100 kPa (1 atm) pressure and a temperature of 0 °C (32 °F). The diffusion coefficient values in Table 2 are calculated and converted from an empirical equation.[2]

Brownian Motion of Gases

One aspect of gaseous behavior that gives the strongest clue to the nature of gases is the phenomenon known as *Brownian motion*. This motion, first observed by the Scottish botanist Robert Brown in 1827, is the irregular motion of extremely minute particles suspended in a fluid. Brownian motion can be observed by focusing a microscope on a particle of illuminated cigarette smoke in a glass tube. The particle does not settle to the bottom of the container but continues to move randomly in all directions. The smaller the suspended particle under observation, the higher the temperature of the fluid, the more vigorous is the particle's movement. The existence of Brownian motion suggests that the molecules of gaseous matter are constantly moving. A visible small particle seems to be jostled by its neighboring invisible particles. The motion of the visible smoke particle thus indirectly reflects the motions of the smaller invisible particles of matter. This provides powerful support for the idea that gaseous or fluid matter consists of extremely small particles or molecules in constant motion. The theory of moving molecules of gases is the *kinetic molecular theory of matter*. Its basic postulates are these.

1. The molecules of gaseous matter are in motion.
2. Heat causes this molecular motion.

The kinetic theory of gases can be used to explain many of the properties and characteristics of tracer gases used in leak testing.

Assumptions Underlying the Kinetic Theory of Ideal Gases

The kinetic theory of gases applies only to ideal or perfect gases that behave in accordance with the following assumptions.

1. Gases consist of tiny molecules so small and so far apart that the actual volume of the gas molecules is negligible compared to the empty space between them.
2. There are no attractive forces between gaseous molecules.
3. The molecules of gases travel in random straight-line motion and collide elastically with each other and with the walls of their container.
4. In any collection of gas molecules, individual molecules have different speeds. However, their average speed (including many molecules over a significant period of time) is dependent on the absolute temperature (kelvin or rankine degrees). The higher the gas temperature, the higher the average molecular speed.

Kinetic Theory Explanations of Gaseous Pressure, Volume and Temperature

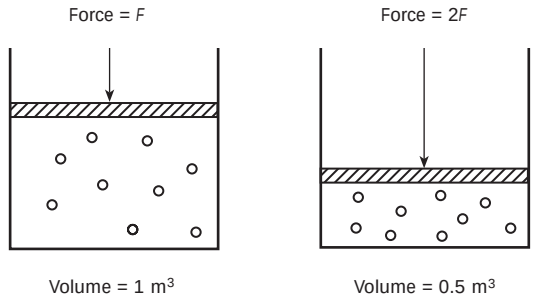
The kinetic theory of gases postulates that a gas consists mostly of empty space in which billions of tiny points representing molecules are moving randomly. The molecular particles collide with each other and with the walls of the container. The volume of a gas sample is the volume of its container. Pressure is exerted by gases because the molecules collide with the walls of the container. Each collision produces a tiny push or impulse as the molecule rebounds from the wall. The sum of all of these molecular pushes or force impulses of impact constitute the pressure of the gas on its containment walls. The temperature of a gas is a measure of the average speed or kinetic energy of the particles.

Kinetic Theory Explanations of the Gas Laws

The kinetic molecular theory of gases can be used to explain the observed behavior of gases as described by the gas laws.

Boyle's Law. The pressure exerted by a gas at a given temperature depends only on the number of impacts of gas molecules with the walls of the container. If the volume is reduced as sketched in Fig. 4,

FIGURE 4. Example of Boyle's law. Doubling of gas pressure concentrates gas molecules and doubles number of molecular impacts per unit area on chamber walls and piston in given time period.



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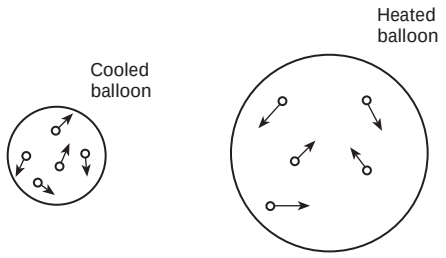
the molecules are more confined. This increases the frequency of molecular collisions with the walls. These more numerous impacts are observed as a greater pressure.

Charles' Law. If the temperature of a gas rises, the average molecular energy and so the average speed of the gas molecules rises. As the molecules move more energetically, they collide with the walls of the container more frequently and with greater momenta, thus producing greater pressure. (Force is equal to the time rate of change of momentum and pressure is the force per unit area.) As shown in Fig. 5, if the temperature is raised, the balloon responds to the increased pressure by stretching and expanding its diameter.

Dalton's Law. According to the kinetic theory of ideal gases, there are no attractive forces between the molecules of gases. On the average, the molecules of each constituent of a gaseous mixture will strike the walls of their container the same number of times per second and strike with the same impact forces as they would if there were no other gases constituents present (see Fig. 6.) Therefore, the partial pressure of a gaseous constituent in a gas mixture is not changed by the presence of other gases in the container. The total pressure exerted on the walls of the container (or on the diaphragm of a pressure measuring gage) is equal to the sum of the partial pressures exerted by the individual constituents of the gaseous mixture.

FIGURE 5. Example of Charles' law. Raising gas temperature increases molecular velocities and increases gas pressure on container wall. Under constant atmospheric pressure, impacts by higher velocity molecules cause increase in gas volume within elastic balloon.



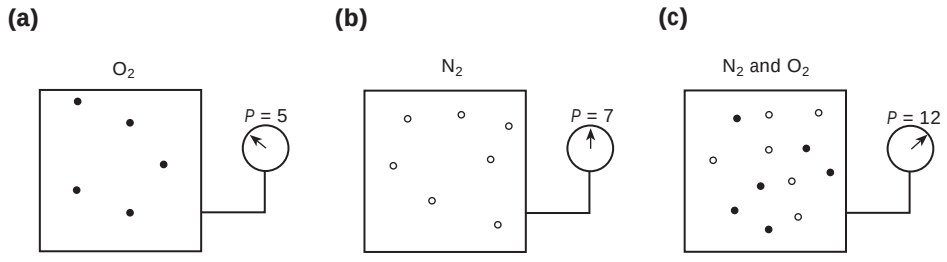
Determining Concentration of Tracer Gas in Gas Mixtures from Partial Pressures

In many leakage measurements, it is desirable or necessary to dilute the tracer gas being used for leak testing. Use of diluted tracer gas might be dictated by practical considerations such as:

1. high expense of pure tracer gas filling large volumes or attaining high pressures;
2. attainment of a more nearly linear or more stable instrument response at a lower concentration of tracer gas;
3. pure tracer gas providing a much higher leakage sensitivity than needed;
4. danger of fire or explosion with a flammable tracer gas (in some cases, a dilute gas mixture lowers the danger of explosions); and
5. inability to completely evacuate the test object or test system before filling with tracer gas. As a result, residual gas dilutes the tracer gas added during pressurizing.

Concentration of the tracer gas in a test system containing mixed gases depends on the partial pressure of the tracer gas. Dalton's law (Eq. 4) shows the contributions of each gaseous constituent to the total gas pressure. The fractional concentration of the tracer gas T is given by the term N_T in Eq. 13:

FIGURE 6. Example of Dalton's law: (a) oxygen; (b) nitrogen; (c) nitrogen and oxygen combined. Partial pressure of each gaseous constituent is not changed by presence of other gases in the same container. Pressure is exerted on container walls by impacts of individual molecules of all gas species.



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$$(13)\quad N_T = \frac{P_T}{P_{\text{total}}} = \frac{P_T}{P_1 + P_2 + P_3 + \ldots + P_n}$$

This fractional concentration is given by Eq. 13 in terms of the number of molecules of tracer gas as a fractional part of the total number of molecules in a gaseous mixture. The percentage concentration by mass would depend also on the molecular masses of each gaseous constituent. The partial pressure P_T of tracer gas required to provide a specific percentage of tracer gas molecules, percent T, is given in terms of total system pressure P_{total} by Eq. 14:

$$(14)\quad P_T = \frac{\%T \times P_{\text{total}}}{100}$$

Partial Pressures of Gaseous Constituents of Earth's Atmosphere

The composition of atmospheric air is 78 percent nitrogen, 21 percent oxygen, 0.9 percent argon and about 0.1 percent of other gases and vapors (including water vapor, whose concentration varies with the temperature and relative humidity of the atmosphere). The partial pressures of atmospheric constituents at sea level, where the total pressure of 100 kPa (1 atm) is equal to that of 760 torr, are given in Table 3.

The partial pressure in kilopascal is about the same as the percentage of each constituent gas, at standard atmospheric pressure. The partial pressures of atmospheric constituents at an altitude of 3600 m (12 000 ft), where the total pressure is equal to 64.4 kPa (9.3 $lb_f \cdot in.^{-2}$ absolute), are given in Table 2.

The percentage composition of atmospheric air changes very little until very high altitudes are reached. When test systems are pressurized with air pumped from the atmosphere, the percentage composition is also not changed from that in the atmosphere at sea level. Helium is present in the earth's atmosphere in the proportion of 5 $\mu L \cdot L^{-1}$. With mass spectrometer types of helium leak detectors, even this small proportion of helium can be readily sensed.

Mean Free Paths of Gas Molecules

The mean free path is the average distance a gas molecule travels between successive collisions with other molecules in the gas or vapor state. The mean free path is important in leak testing because it determines the type of gas flow that will occur through leaks or other passageways traversed by tracer or pressurizing gases. The mean free path can be calculated from the pressure, temperature and molecular properties by means of Eq. 15:

$$(15)\quad \lambda = 116.4 \frac{n}{P} \sqrt{\frac{T}{M}}$$

where λ is mean free path (meter) under static pressure; n is gas viscosity (pascal-second); P is absolute pressure of gas (pascal); T is absolute gas temperature, (kelvin); and M is molecular mass of gas, ($g\text{-}mol^{-1}$).

Table 4 lists the mean free paths of common gases at 20 °C (68 °F) and 1.0 mPa (7.6 µtorr).

Simple Approximation Formula for Mean Free Path of Common Gases

An easily remembered relation for approximating the mean free paths of common gaseous molecules is presented in Eq. 16:

$$(16)\quad \lambda = \frac{NF}{P}$$

where λ is mean free path length (meter), P is gas pressure in pascal and NF is a numerical factor (meter-pascal) given in Table 5. An NF value of 6.8×10^{-3} permits

TABLE 3. Partial pressures of atmospheric constituents at sea level, 100 kPa (1 atm).

Gas	Percent	kPa at sea level[a]	(torr at sea level)[a]	kPa at 3.6 km	(torr at 12 000 ft)[b]
Oxygen (O_2)	21.0	21.28	(159.6)	13.52	(101.4)
Nitrogen (N_2)	78.0	79.03	(592.8)	50.22	(376.65)
Argon (Ar)	0.9	0.91	(6.84)	0.58	(4.35)
Other	0.1	0.10	(0.76)	0.06	(0.45)
Total air	100.0	101.325	(760.00)	64.4	(483.0)

a. Atmospheric pressure at sea level = 101.325 kPa (1 atm).
b. Percentage × atmospheric pressure of 64.4 kPa.

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TABLE 4. Mean free path lengths of various atmospheric gases at 20 °C (68 °F) and at absolute pressure of 1.0 mPa (7.6 µtorr).

Gas	Mean Free Path m	(in.)
Air	6.8	(268)
Argon (Ar)	7.2	(284)
Carbon dioxide (CO_2)	4.5	(177)
Hydrogen (H_2)	12.5	(492)
Water (H_2O)	4.2	(165)
Helium (He)	19.6	(771)
Nitrogen (N_2)	6.7	(264)
Neon (Ne)	14.0	(551)
Oxygen (O_2)	7.2	(284)

use of Eq. 16 for estimating the mean free path lengths for air, argon, nitrogen and oxygen molecules. Table 5 lists the numerical factors used in the numerator of Eq. 16 for several other common gases. However, it is seldom necessary to know mean free path lengths to precisions better than one order of magnitude. For example, the molecular mean free path at 20 °C (68 °F) at atmospheric pressure is of the order of 30 to 300 nm. At a pressure of 1 Pa (1.5×10^{-4} $lb_f \cdot in.^{-2}$), the mean free path is in the range from 3 to 30 mm (0.12 to 1.2 in.).

Relation of Mean Free Path Lengths to Viscosity and Molecular Mass of Gas

The ratio of mean free path lengths for two different gases at the same temperature and pressure are given by Eq. 17:

$$\frac{\lambda_1}{\lambda_2} = \frac{n_1}{n_2}\sqrt{\frac{M_2}{M_1}} \quad (17)$$

In Eq. 17, n indicates the gas viscosity and M indicates its molecular mass. The subscript 1 indicates the first gas and subscript 2 indicates the second gas. (This relationship is derived from Eq. 15 when T and P are held constant.) For a leak or an orifice across which there is a sizable pressure differential, the mean free path length within the orifice is typically estimated from the average pressure in the orifice (the mean value of inlet and outlet pressures).

Effective Viscosity of Mixtures of Gases

In a mixture of various species of gases, the effective viscosity $n_{mixture}$ is assumed to be proportional to the sum of the products of viscosity and concentrations for each individual gaseous constituent, as indicated by Eq. 18:

TABLE 5. Physical properties of common gases used in leak testing.

Gas	Formula	Molecular Mass ($g \cdot mol^{-1}$)	Density[a] at 100 kPa ($g \cdot L^{-1}$)	Numerical Factor[b] for Mean Free Path ($m \cdot Pa$)	Dynamic Viscosity[c] at 20 °C (68 °F) ($\mu Pa \cdot s$)	Diffusivity[d] in Air at 0 °C (32 °F) and 101 kPa ($m^2 \cdot s^{-1}$)	Thermal Conductivity[e] at 20 °C (68 °F) ($W \cdot m^{-1} \cdot K^{-1}$)
Air[f]	Mixture	29.0	1.21	6.8×10^{-3}	18		26.2
Argon	Ar	40	1.79	7.2×10^{-3}	22	13.9×10^{-6}	17.9
Carbon dioxide	CO_2	44	1.97	4.5×10^{-3}	15	15.8×10^{-6}	16.0
Refrigerant-12	CCl_2F_2	121	5.25		13		9.8
Helium	He	4.0	0.179	19.6×10^{-3}	19		149.0
Hydrogen	H_2	2.0	0.090	12.5×10^{-3}	9	63.4×10^{-6}	183.0
Krypton	Kr	84	3.74	5.36×10^{-6}	25		9.4
Neon	Ne	20	0.90	14.0×10^{-3}	31		48.0
Nitrogen	N_2	28	1.25	6.7×10^{-3}	18		25.6
Oxygen	O_2	32	1.43	7.2×10^{-3}	20	17.8×10^{-6}	26.2
Sulfur hexafluoride	SF_2	146	6.60	2.5×10^{-3}	15		13.0
Water Vapor[g]	H_2O	18	0.83	4.2×10^{-3}	9	23.9×10^{-6}	18.7
Xenon	Xe	131	5.89	3.8×10^{-3}	22		5.5

a. Density in $oz \cdot ft^{-3} = g \cdot L^{-1} = mg \cdot cm^{-3}$ at 20 °C (68 °F) and 100 kPa (1 atm).
b. Numerical factor for calculating mean free path using Eq. 16. Mean free path in meters at 20 °C (68 °F).
c. Independent of pressure under conditions for viscous flow.
d. Diffusivity in $m^2 \cdot s^{-1}$ in air at 0 °C (32 °F) and 101 kPa (1 atm).
e. Thermal conductivity in $W \cdot m^{-1} \cdot K^{-1}$ at 20 °C (68 °F). Thermal conductivity is independent of pressure under conditions for viscous flow.
f. N_2, 78 percent; O_2, 21 percent; argon, 0.9 percent; others, 0.1 percent.
g. Vapor pressure of H_2O at 20 °C (68 °F) is 2.3 kPa (17.5 torr).

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$$(18)\quad n_{\text{mixture}} = N_1 n_1 + N_2 n_2 + \ldots + N_k n_k$$

where n_1 is viscosity of the first gaseous constituent and N_1 is fractional concentration of the first gaseous constituent, as defined by Eq. 14.

Molecular Masses of Gases and Vapors

Any combination of atoms in a chemical compound is called a molecule. The molecular mass equals the total number of nucleons in the atoms forming the molecule. Most elements in the gaseous state form diatomic molecules that consist of two atoms of that element loosely bound by electronic forces. The molecular mass of diatomic gases is twice the atomic mass. For example, the element oxygen O has an atomic mass of 16; gaseous oxygen O_2 has a molecular mass of 32. Exceptions to this diatomic arrangement in gases include most metallic vapors and the noble gases. The noble gases (argon, helium, neon, krypton, radon and xenon) have extremely stable electronic structures and typically do not combine with any other atom species. The molecular masses of the monatomic gases are identical to their atomic masses.

In chemical compounds containing different elements (for example, carbon dioxide) the molecular mass is the sum of the atomic masses of the constituent atoms. One carbon atom (atomic mass 12) combines with two oxygen atoms (atomic mass 16 each) to form CO_2 with a molecular mass of 44. The molecular masses of common gases and some vapors are tabulated in Table 6.

Vapors resulting from evaporation of liquid hydrocarbon compounds have molecules containing relatively large numbers of atoms. Molecular masses of such organic chemical compound vapors increase as the macromolecules increase in complexity and contain more atoms.

TABLE 6. Viscosity and molecular masses of typical gases and vapors used in leak testing.

Gas	Viscosity at 15 °C (60 °F) (μPa·s)[a]	Relative Molecular Mass (u)[b]
Hydrogen	8.7	2.02
Helium	19.4	4.00
Methane	10.8	16.0
Ammonia vapor	9.7	17.0
Water vapor	9.3	18.0
Neon	31.0	20.2
Nitrogen	17.3	28.0
Air	18.0	28.7
Oxygen	20.0	32.0
Hydrogen chloride vapor	14.0	36.5
Argon	21.9	39.9
Carbon dioxide	14.5	44.0

a. One μPa·s = 10 micropoise.
b. One unified atomic mass unit (u) ≅ 1.6605×10^{-27} kg.

Stratification of Constituents in Mixtures of Gases

If a tracer gas is added to air already within a vessel or system under test, a uniform mixture of gases is often difficult to achieve. The tracer gas will settle toward the top or toward the bottom of large containers, depending on the density of the tracer gas relative to the density of the air or other pressurizing gases within the system. This stratification of mixed gases is more pronounced with high molecular mass gases and with gases with low diffusion coefficients. Precautions should be taken to avoid or correct stratification effects during leak testing by (1) premixing of tracer gas with diluent gases before injection, during pressurization of the test system or enclosing hood or chambers and (2) providing some means for circulating and mixing the gases within large volume chambers or test systems.

Usually, there should be no problems with pooling or stratification inside test systems, if precautions are taken to mix the tracer gas thoroughly with the diluent gas in pressurization of the test system. However, if the test pressure is to be about atmospheric pressure in the test system, the system should first be evacuated to remove air at atmospheric pressure and to replace it by the thoroughly mixed combination of tracer gas with diluent gas.

Equilibrium Distribution Law for Gas Concentration Ratios with Gravity Effect

The preferred technique is that in which both the tracer and diluent gases used in pressurization of test systems are premixed or added simultaneously through a screened aperture or rake so as to be mixed rather uniformly from the start. There should then be no problem of pooling of denser constituents inside the system under test, provided that precautions are taken to mix the tracer thoroughly with diluent gas in the pressurization of the system. The equilibrium distribution law of Eq. 19

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gives the ratio C_h of tracer as concentration at the top of a tank relative to the concentration C_o of the same gaseous constituent at the bottom of the tank:

$$C_h = C_o\, e^{-\frac{Mgh}{RT}} \quad \textbf{(19)}$$

where M is molecular mass of gaseous constituent, h is height of interior volume of tank, R is universal gas constant, T is absolute temperature and g is local value of acceleration due to earth's gravity.

From Eq. 19, it is evident that with a specific tracer gas in equilibrium distribution, the concentration of tracer gas diminishes exponentially as height within the chamber increases. The greatest concentration of the gaseous constituent is at the bottom of the tank and the lowest concentration exists at the top of the tank. However, this effect takes no account of the relative densities of the tracer gas and diluent gas. If the tracer gas were lower in density than the diluent gas (as with helium tracer gas in air), stratification effects could have a predominant effect, with helium collecting at the top of the tank after a period of time. If the tracer gas were higher in density than the diluent gas (as with refrigerant-12 gas in air), stratification effects could also predominate and the denser tracer gas would tend to collect at the bottom of the tank after a period of time. In large test chambers or enclosing hoods, it would be desirable to provide constant internal circulation and mixing of the internal contents of tracer gas and diluent gas, as with a fan.

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PART 2. Mechanisms of Gaseous Flow through Leaks

Modes of Gas Flow through Leaks of Restrictions

To clarify the problem of leakage, it is necessary to consider gas flow through small restrictions. It is extremely important to know something about the basic modes of flow: viscous, transitional and molecular. Viscous flow may be further divided into laminar flow or turbulent flow. Other special modes of leakage or flow are permeation and choked flow. The factors that influence gaseous flow through leaks are (1) the molecular mass of the gas, (2) the viscosity of the gas, (3) the pressure difference causing the flow, (4) the absolute pressure in the system and (5) the length and cross section of the leak path.

An understanding of leakage mechanisms and controlling factors is vital to the proper interpretation of leak tests. A simple description of gaseous flow through leaks is presented here for leak testing operators and supervisory personnel, followed by a theoretical approach to leakage.

Permeation of Gases through Solids

Permeation is the passage of fluid into, through and out of a solid barrier having no holes large enough to permit more than a small fraction of molecules to pass through any one hole. The process also involves diffusion through a solid and may involve many phenomena such as adsorption, dissociation, migration and desorption.

The first implication of permeation is that if the system is to be relatively leaktight, the materials of construction have to exclude leakage by permeability. As an example, the permeation rate at room temperature of a natural rubber gasket (2.5 mm thick, with a 2.5 mm wide rim and a 125 mm diameter) with a 100 kPa (1 atm) hydrogen pressure differential is 1.6×10^{-6} Pa·m^3·s^{-1} (1.6×10^{-5} std cm^3·s^{-1}). In some uses, this permeation might represent an unacceptable leakage rate.

Another similar example of this type of permeation involves a rubber O-ring. Depending of the material and the type of gas, a rubber O-ring usually represents a permeability of about 5×10^{-7} Pa·m^3·s^{-1} (5×10^{-6} std cm^3·s^{-1}) for every 100 kPa (760 torr) of pressure differential per linear centimeter of exposed O-ring surface. This permeability does not have to be taken into consideration during routine leak testing if the leakage measurement occurs in a time too short to permit the saturation and mass transfer of gas through the O-ring.

Mean Free Path of Gaseous Molecules

Molecular flow occurs when the mean free path of a tracer gas is greater than the cross section dimension of the leak. The mean free path is the average distance a molecule travels between successive collisions with other molecules in vapor state. The mean free path is of some importance in leak testing because it establishes the type of gas flow that will occur. The mean free paths of several gases are given in Table 4.

In flow systems encountered in leak testing, knowing the mean free path allows one to know, or at least estimate, the type of flow occurring. Table 4 shows, in general, the relationship of mean free path to pressure and the information may be used as a guide to determine the nature of the flow.

Characteristics of Molecular Flow of Gases

It should especially be noted that in molecular flow the leakage rate is proportional to the difference of the pressures. Molecular flow occurs quite often in vacuum testing applications. In molecular flow, molecules travel independently of each other. It is possible for random molecules to travel from a part of a system at low pressure to another part of the system at a higher pressure. When an ultrahigh vacuum system is being tested by a mass spectrometer leak detector, the mass spectrometer leak detector operates at a pressure of about 10 µPa (0.1 µtorr) whereas the ultrahigh vacuum system might be operating at a pressure of

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0.1 μPa (1 ntorr). When a tracer gas enters the ultrahigh vacuum system through a leak, it will eventually travel from the 0.1 μPa (1 ntorr) vacuum system to the mass spectrometer operating at 10 μPa (0.1 μtorr) by the process of molecular flow. This does not imply that the total flow is from a system at low pressure to one at high pressure. The mass spectrometer operating at 10 μPa (0.1 μtorr) sends some gas molecules into the system at the lower pressure. However, when summing flows, total net flow is from the high-pressure to the low pressure region. The high pressure system is contributing gas molecules to the ultrahigh vacuum system. The tracer gas flow in the direction opposing the major flow of molecules is possible because of the random mode of molecular flow. The gas molecules, when traveling from one system to the other, do not come in contact with molecules traveling in the other direction.

Characteristics of Transitional Flow of Gases

Transitional flow occurs when the mean free path of the gas is about equal to the cross section dimension of the physical leak. It occurs under conditions intermediate between laminar and molecular flow. The transition from laminar flow to molecular flow is gradual. The mathematical treatment of this region is extremely difficult; however, a treatment of this region is necessary because leakage from an enclosed volume to a vacuum necessarily involves a transition from laminar to molecular flow.

Characteristics of Laminar Flow of Gases

The laminar flow of a fluid in a tube is defined as a condition in which there is a parabolic distribution of the fluid velocity in the cross section of the tube. The two most important characteristics of laminar leaks are (1) the flow is proportional to the square of the pressure difference across the leak and (2) the leakage is inversely proportional to the leaking gas viscosity. Table 1 shows that the viscosity of most gases varies by less than one order of magnitude. Changing the tracer gas will not markedly increase the sensitivity of the leak test unless this change of gas implies a change of instrument sensitivity. However, increasing the pressure difference across the leak by a factor of a little over three will increase the flow rate through this leak by a factor of ten. Obviously then, when the leaks to be measured are in the laminar flow range, the simplest means of increasing test sensitivity is by an increase of pressure across the leak.

Viscous Flow of Gases through Leaks

Laminar flow is one of the two classes of viscous flow; the other class is turbulent flow. Because turbulent flow is rarely encountered in leaks, the term *viscous flow* is sometimes incorrectly used to describe laminar flow in leak testing. *Viscous flow* implies that the flow occurs when the mean free path of the gas is smaller than the cross section dimension of the leak. It should especially be noted that the viscous flow leakage rate is proportional to the difference of the squares of the pressures. Viscous flow leakage occurs in high pressure systems, such as are encountered in detector probe leak tests. It is often related with the Reynolds number. The dimensionless Reynolds number is the ratio of the inertial to the viscous forces acting on the medium. In the case of tubes (or leak paths), the Reynolds number N_{Re} is expressed by Eq. 20:

$$N_{Re} = \frac{v d}{\eta} \quad (20)$$

where v is velocity ($m \cdot s^{-1}$), d is diameter of opening (meter) and η is kinematic viscosity ($m^2 \cdot s^{-1}$). However, any set of consistent units may be used in this equation. Above a critical value of the Reynolds number (about 2100 in the case of circular tube flow), flow becomes unstable. This results in innumerable eddies or vortexes in the flow. The partial path in turbulent flow leaks is very erratic. In laminar flow, the particles flow nearly straight line paths.

Characteristics of Choked (or Sonic) Flow of Gases

Choked flow, or *sonic flow* as it is sometimes called, occurs under certain conditions of leak geometry and pressure. Assume there exists a passage in the form of an orifice or a venturi, and assume that the pressure upstream is kept constant. If the pressure downstream is gradually lowered, the velocity through the throat or orifice will increase until it reaches the speed of sound through the fluid. The downstream pressure at the time the orifice velocity reaches the speed of sound is called the critical pressure. If the downstream pressure is lowered below this critical pressure, no further increase in orifice velocity can occur, with the

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TABLE 7. Composition and partial pressures of dry air at sea level (101.325 kPa or 1.00 atm). Note the similarity of partial pressures with the percentages. When less precision can be tolerated, use percentages × 10^3.

Constituent	Content		Partial Pressure	
	Percent	$\mu g \cdot g^{-1}$	Pa	(torr)
Nitrogen	78.084	——	7.9119×10^4	(4.9343×10^2)
Oxygen	20.946	——	2.1224×10^4	(1.5919×10^2)
Argon	0.934	——	9.460×10^4	(7.10)
Carbon dioxide	0.033	——	3.34×10^1	(2.50×10^{-1})
Neon	1.8×10^{-3}	18.18	1.84	(1.38×10^{-2})
Helium	5×10^{-4}	5.24	5.3×10^{-1}	(3.98×10^{-3})
Krypton	1×10^{-4}	1.14	1.16×10^{-1}	(8.66×10^{-4})
Xenon	——	0.087	8.8×10^{-3}	(6.61×10^{-5})
Hydrogen	——	0.5	5×10^{-2}	(3.80×10^{-4})
Methane	——	2.0	2×10^{-1}	(1.52×10^{-3})
Nitrous oxide (N_2O)	——	0.5	5×10^{-2}	(3.80×10^{-4})

consequence that the maximum mass flow rate has been reached. This condition is known as choked or sonic flow.

Leaks Dependent on a Critical Gas Temperature or Pressure

Both pressure dependent leaks and temperature dependent leaks have been observed, but in extremely limited number. *Pressure dependent* or *temperature dependent leaks* denote a condition where no leakage exists until a critical pressure or temperature is reached. At this point, the leakage appears suddenly and may be appreciable. Further changes in pressure or temperature cause the leakage to vary in the prescribed manner. When the pressure or temperature is reversed, the leakage follows the prescribed course to the critical point at which leakage drops to zero. No adequate explanation for this phenomenon is advanced, but in view of the very few times this occurs, such leaks can generally be ignored. Temperature and pressure are not normally applied in the course of leak testing for the purpose of locating such leaks. Instead, they are used to force existing discontinuities to open, so as to start or increase the leakage rate to a point of detection.

Physical Properties of Tracer Gases Used in Leak Testing

When performing any leak test it is important to have some knowledge of the residual gases present in the test area because this will have a bearing on the choice of tracer gas and test technique to be used. Table 7 lists the standard composition of dry air at sea level.

The physical properties of gases and vapors are also important, including the molecular mass, the molecular diameter and the viscosity. The gas streaming through a narrow bore tube experiences a resistance to flow so that the velocity of gas flow decreases uniformly from the center outwards until it reaches zero at the walls. Each layer of gas parallel to the direction of flow exerts a tangential force on the adjacent layer, tending to decrease the velocity of the faster moving layers and to increase that of the slower moving layers. The property of a gas or liquid by virtue of which it exhibits this phenomenon is known as internal viscosity.

The internal viscosity is directly proportional to the velocity gradient in the gas. Furthermore, the viscosity must depend on the nature of the fluid. In a more viscous fluid the tangential force between adjacent layers for constant velocity gradient will be greater than in a less viscous fluid. For any gas at constant temperature, the gas viscosity is independent of the pressure. However, gas viscosity increases as gas temperature rises. Conversely, the viscosity of all ordinary liquids decreases with increased temperature.

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PART 3. Practical Measurement of Leakage Rates with Tracer Gases

Principles of Leakage Measurement

All leak detection with tracer gases involved their flow from the high pressure side of a pressure boundary through a presumed leak to the lower pressure side of the pressure boundary. When tracer gases are used in leak testing, instruments sensitive to tracer gas presence or concentration are used to detect outflow from the low pressure side of the leak in the pressure boundary.

Where leak tests involve measurements of change in pressure or change in volume of gas within a pressurized enclosure, the loss of internal gas pressure or volume indicates that leakage has occurred through the pressure boundary. When evacuated or low pressure test systems or components are surrounded by higher pressure media such as the earth's atmosphere, or a hood or test chamber containing gases at higher pressures, leakage can be detected by loss of pressure in the external chamber or by rise in pressure within the lower pressure system under test.

Modes of Gas Flow through Leaks

For each type of leak test, it is essential that the test operator have a basic understanding of the types of flow that might occur in a leak. Different basic laws relate leakage rate to pressure difference across the leak, the range of absolute pressure involved and the nature of the gaseous fluid escaping through the leak. There are three basic types of gas flow through leaks.

1. *Viscous flow* typically occurs in probing applications with gases leaking at atmospheric or higher pressures.
2. *Molecular flow* usually occurs in leaks under vacuum testing conditions.
3. *Transitional flow* occurs under test conditions intermediate between vacuum and pressures higher than atmospheric pressure.

Figure 7 shows the range of conditions of gas pressure and leak radius under which each of these types of flow is typically encountered, for leakage flow of air.

Criteria to Determine Type of Gas Flow through Leaks

The type of flow that occurs through leaks depends on the factors listed earlier. In flow systems encountered in leak testing with gases, the length of the mean free path of the gaseous molecules can be used to estimate the type of flow occurring through leakage paths. (The mean free path lengths for various gas molecules can be calculated by means of Eq. 15 or 16. Tables 4 and 5 give data on mean free path lengths for several gases and pressure ranges.) When determining the nature of flow of gases through leaks, use is made of two parameters: (1) the mean free path length λ is determined by using the average pressure in the leak flow system.

The criteria that determine the mode of gas flow through leaks, given in terms of the mean free path length λ and the leak dimensional constant d, are as follows.

1. When the ratio $\lambda \cdot d^{-1}$ is less than 0.01, the gas flow is viscous.
2. When the ratio $\lambda \cdot d^{-1}$ has values between 0.01 and 1.00, the gas flow is transitional.
3. When the ratio $\lambda \cdot d^{-1}$ is greater than 1.00, the gas flow is molecular.

In molecular flow, the mean free path length is greater than the largest linear dimension of the cross section of the leak.

Relation of Viscous Leakage Flow to Pressure Differential across Leaks

Viscous flow occurs when the mean free path length of the gas is significantly smaller than the cross section of a leak. This condition is implied by the first criterion above, where λ is at least 100 times smaller than the leak's cross sectional diameter d. Viscous flow occurs in high pressure systems such as in probing applications where tracer gases leak into air at atmospheric pressures. With viscous flow through leaks, the flow rate or leakage Q is proportional to the difference in the squares of the pressures acting across the leak. This relationship is shown by Poiseuille's law for viscous flow through a cylindrical tube, in Eqs. 21 and 22 for the leakage rate Q:

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$$\textbf{(21)}\quad Q = \frac{\pi r^4}{8\,n\,l} P_a \left(P_1 - P_2\right)$$

or

$$\textbf{(22)}\quad Q = \frac{\pi r^4}{16\,n\,l} \left(P_1^2 - P_2^2\right)$$

where Q is gas flow rate ($Pa \cdot m^3 \cdot s^{-1}$), r is radius of leakage tube (meter), l is length of leakage tube (meter), n is viscosity of leaking gas (Pa·s), P_1 is upstream gas pressure (pascal), P_2 is downstream pressure (pascal) and P_a is average pressure within leak path, $(P_1 + P_2)/2$ (pascal).

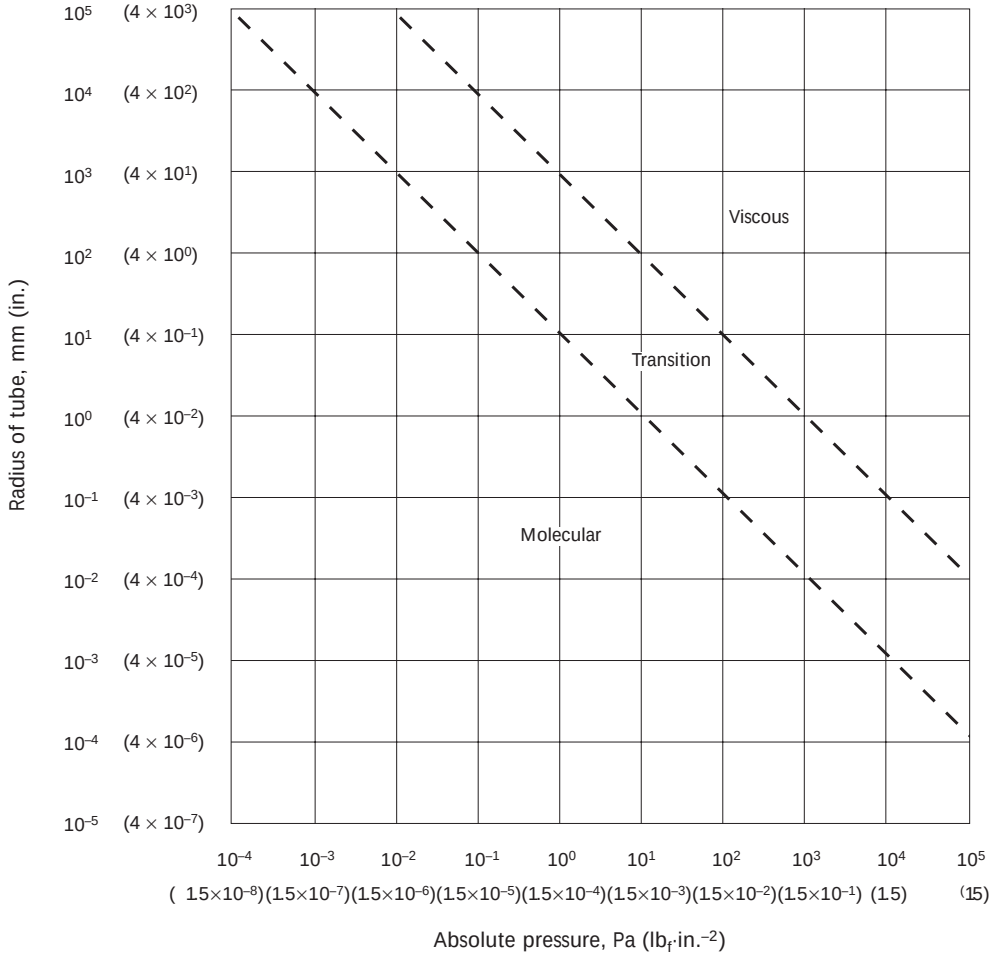
Relation of Molecular Leakage Flow to Pressure Differential across Leaks

Molecular flow occurs when the mean free path length of the gas molecules is greater than the largest cross sectional dimension of a physical leak. This condition is implied by the third criterion in the discussion above. Molecular flow usually occurs through leaks in vacuum systems or systems that have vacuum applied to the lower pressure side of the pressure boundary for purposes of leak testing. With molecular flow through leaks, the leakage rate Q is proportional to the difference in pressures applied across the leak. This relationship is shown by Knudsen's law for molecular flow through a cylindrical tube, neglecting the end effect, as shown in Eq. 23 for the leakage rate, Q through a tubular leak with molecular flow:

$$\textbf{(23)}\quad Q = 3.342 \frac{r^3}{l} \sqrt{\frac{RT}{M}} \left(P_1 - P_2\right)$$

where Q is leakage rate ($Pa \cdot m^3 \cdot s^{-1}$), r is radius of leakage tube (meter), l is length of leakage tube (meter), M is molecular weight of gas (kilogram per mole), P_1 is upstream pressure (pascal), P_2 is downstream pressure (pascal), T is absolute temperature (kelvin); and gas constant $R = 8.315\ J \cdot mol^{-1} \cdot K^{-1}$.

FIGURE 7. Types of flow characteristics of tracer gases though leaks as function of leak channel radius and gas pressure. Graph illustrates air at 25 °C (77 °F).



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If this value is substituted for R in Eq. 23, the leakage rate in SI units is given by Eq. 24:

$$\textbf{(24)} \quad Q = 9.637 \frac{r^3}{l} \sqrt{\frac{T}{M}} \left(P_1 - P_2\right)$$

If the molecular mass M is given in units of grams per mole. All other quantities are in SI units as listed above. The leakage rate in SI units of $Pa \cdot m^3 \cdot s^{-1}$ is given by Eq. 25:

$$\textbf{(25)} \quad Q = 304.8 \frac{r^3}{l} \sqrt{\frac{T}{M}} \left(P_1 - P_2\right)$$

In cgs (centimeter-gram-second) units, with tube radius and length given in centimeter and molecular weight in gram per mole, the rate of leakage ($L \cdot s^{-1}$) is given by Eq. 26:

$$\textbf{(26)} \quad Q = 30.48 \frac{r^3}{l} \sqrt{\frac{T}{M}} \left(P_1 - P_2\right)$$

This flow rate is related to the flow rate F_o for zero thickness orifice:

$$\textbf{(27)} \quad F = \frac{8}{3} \frac{r}{l} F_o$$

where both F and F_o are flow rates ($L \cdot s^{-1}$).

Relation of Transitional Leakage Flow to Mean Free Path Length of Gas and to Pressure Differential Applied across Leak Path

Transition flow occurs when the mean free path length of the gas molecules is about equal to the cross-sectional dimension of the leak. Transitional flow occurs under leakage conditions intermediate between those for viscous flow and those for molecular flow. For transitional flow, Knudsen's law (see Eq**s**. 23 to 27) for molecular leakage is modified by an additional term that depends on the ratio R equal r/λ or leakage tube radius r to the mean free path length λ that applies for the average pressure $(P_1 + P_2)/2$, existing within the leakage path. This correction term for transitional flow in leakage paths is given as the factor F_T, defined by Eq. 28 where $R_t = r/\lambda$:

$$\textbf{(28)} \quad F_T = 0.1472\, R_t + \frac{1 + 2.507\, R_t}{1 + 3.095\, R_t}$$

The leakage rate Q in SI units of $Pa \cdot m^3 \cdot s^{-1}$ is given by Eq. 29:

$$\textbf{(29)} \quad Q = 3.342 \frac{r^3}{l} \sqrt{\frac{RT}{M}} \left(P_1 - P_2\right) F_T$$

For Eq. 28 and 29, the symbols are explained below Eq. 23, with the exception of the mean free path length λ, which is determined at the average between upstream and downstream pressures acting across the leak, namely $(P_1 + P_2)/2$, from Eq. 15 or 16 and Tables 4 and 5.

Analogy between Electrical Conductance and Gaseous Conductance

Conductance is a term describing the property of a gas flow system that permits gas to flow. It is defined analogously to electrical conductance G, the reciprocal of electrical resistance R. Ohm's law for direct current flow i through a conductance or resistance is stated in Eq. 30:

$$\textbf{(30)} \quad i = \frac{V}{R} = VG$$

In Eq. 30, the quantity V equals the voltage drop across the resistance R or conductance G. Electrical conductance could be described as the property of an electric circuit that permits current to flow. In steady state direct current circuits, the conductance G is the ratio of the current i flowing in the resistive element to the drop in electrical potential (or electrical pressure) across the resistive element, as in Eq. 31 for the electrical conductance G:

$$\textbf{(31)} \quad G = \frac{1}{R} = \frac{i}{V}$$

With gas flow through the conductance of a leak path, for example, the flow rate Q is analogous to the electric current i. The pressure drop $(P_1 - P_2)$ is analogous to the voltage drop V. Leak conductance C is analogous to the electrical conductance G. The electrical current could be considered as the leakage of electrical charge through a resistive element such as a length of wire of given diameter and specific conductivity. The gaseous conductance of a tubular passageway permits the leakage of a gaseous constituent when a pressure drop exists between the ends of the tubular hole. The gaseous conductance is the reciprocal of the resistance of the leak passageway, as indicated by Eq. 32 for the gaseous conductance C:

$$\textbf{(32)} \quad C = \frac{1}{R_{gas}} = \frac{Q}{P_1 - P_2}$$

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The equivalent of Ohm's law for a gas conductance would be the linear relationship of Eq. 33 for the rate Q of leakage or of gas flow:

$$\textbf{(33)} \quad Q = \frac{P_1 - P_2}{R_{\text{gas}}} = (P_1 - P_2)C$$

However, Eq. 33 is only true for the case of molecular flow, as shown in Eqs. 23 to 27.

It is very important to keep in mind that, by definition, the relationships of gaseous conductance calculations always include a term describing a property of the flowing gas. This property usually is the gas viscosity that influences viscous flow through leaks or is the gas molecular mass that influences molecular flow through leaks.

Equations for Calculating Conductances of Leaks with Various Modes of Flow

The following equations give basic relationships required to calculate leak conductances under various conditions of leak geometry and of modes of gas flow and to estimate variations of leakage rate with different gas pressures.

The conductance of a leak exhibiting viscous flow of gas can be calculated by Eq. 34, assuming that the physical leak channel approximates a straight, cylindrical tube: The viscous conductance C of a tube is expressed as follows:

$$\textbf{(34)} \quad C = \frac{\pi r^4}{8\, n\, l} P_a$$

In Eqs. 34 through 38 for calculating the conductance of leaks, C is gas conductance ($m^3 \cdot s^{-1}$), r is radius of bore of tube (meter), l is length of tubular leak passageway (meter), n is viscosity of leaking gas in Pa·s, P_1 is upstream pressure (pascal), P_2 is downstream (pascal), P_a is average gas pressure within leak channel (pascal), $P_a = (P_1 + P_2)/2$, M is molecular mass of gas $g \cdot mol^{-1}$ and T is absolute temperature (kelvin).

If viscous leakage occurs through an ideal orifice where the ratio P_2/P_1 of downstream to upstream pressures is smaller than or equal to 0.52, the approximate conductance for viscous flow through an ideal orifice can be calculated by the empirical Eq. 35:

$$\textbf{(35)} \quad C = \frac{6.4\, r^2}{1 - \frac{P_2}{P_1}} \sqrt{\frac{T}{M}}$$

The conductance for molecular flow of gases through a long cylindrical tubular leak channel can be calculated from Eq. 36:

$$\textbf{(36)} \quad C = 3.342 \frac{r^3}{l} \sqrt{\frac{RT}{M}}$$

The conductance of a leak that can be approximated by an ideal orifice subject to molecular flow at low pressure is calculated by Eq. 37:

$$\textbf{(37)} \quad C = 3.613\, r^2 \sqrt{\frac{T}{M}}$$

The conductance of a long tubular leak with transitional gas flow can be calculated from Eq. 38:

$$\textbf{(38)} \quad C = 3.342 \frac{r^3}{P} \sqrt{\frac{T}{M}}\, F_T$$

In Eq. 38, the factor F_T is the correction term for transitional flow defined earlier by Eq. 28.

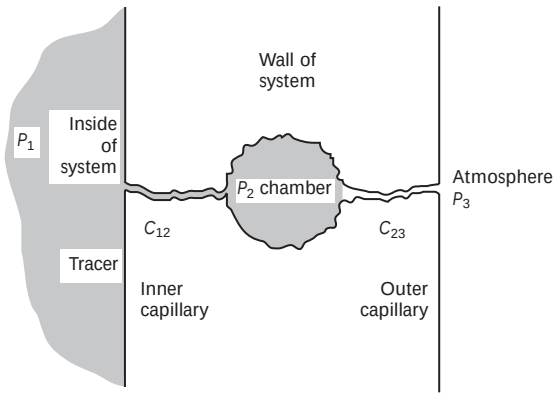
Gas Conductance with Two Leaks in Series

If two different diameter leaks with different conductance values are connected in series as in Fig. 8, the total conductance of the connection between extreme ends decreases (resistance increases). From Eq. 33, the conductance of the leak between the outer ends of sections 1 and 3 may be expressed as in Eq. 39:

$$\textbf{(39)} \quad C_{1-3} = \frac{Q}{P_1 - P_3}$$

The total pressure drop across the two leaks in series is given by Eq. 40:

FIGURE 8. Diagram of typical leak paths connected in series.



Legend
C = channel connecting points
P = point where fluid is present

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(40) $P_1 - P_3 = \left(P_1 - P_2\right) + \left(P_2 - P_3\right)$

The pressure drop across each individual leak is shown in Eq. 41:

(41) $P_1 - P_2 = \frac{Q}{C_{1-2}}$,

$P_2 - P_3 = \frac{Q}{C_{2-3}}$

Now, by combining Eqs. 39 to 41, the conductance C_{13} for the two leaks in series is given by Eq. 42:

(42) $C_{13} = \frac{Q}{\frac{Q}{C_{1-2}} + \frac{Q}{C_{2-3}}}$

or, in its reciprocal form,

(43) $\frac{1}{C_{13}} = \frac{\frac{Q}{C_{1-2}} + \frac{Q}{C_{2-3}}}{Q}$

$= \frac{1}{C_{1-2}} + \frac{1}{C_{2-3}}$

In its general form, Eq. 43 may be written as Eq. 44:

(44) $\frac{1}{C_T} = \frac{1}{C_1} + \frac{1}{C_2} + \ldots + \frac{1}{C_n}$

where the subscript T denotes the total conductance of a number of conductances C_1, C_2, C_3 ... C_n connected in series.

In the case of only two conductances connected in series, Eq. 44 should be written in the form of Eq. 45:

(45) $C_T = \frac{C_1 \times C_2}{C_1 + C_2}$

This case applies for two successive leak conductances connected in series. This is analogous to the case of two electrical resistors connected in parallel or of two electrical conductances connected in series.

Leak Conductance for Two Leaks Connected in Parallel

Figure 9 shows the case of two leaks connected in parallel. With this situation, the total leakage through two parallel leaks divides between the two leakage paths from the high pressure side to the low pressure side of the pressure boundary. The division of flows depends on the conductance of the individual leaks as indicated in Eqs. 46 and 47:

(46) $Q_a = C_a \left(P_1 - P_2\right) = C_a \Delta P$

(47) $Q_b = C_b \left(P_1 - P_2\right) = C_b \Delta P$

The total conductance through the pressure boundary between Points 1 and 2 is given by Eq. 48:

(48) $C_{1-2} = \frac{C_a \Delta P + C_b \Delta P}{\Delta P}$

Simplifying Eq. 48 gives Eq. 49:

(49) $C_{1-2} = C_a + C_b$

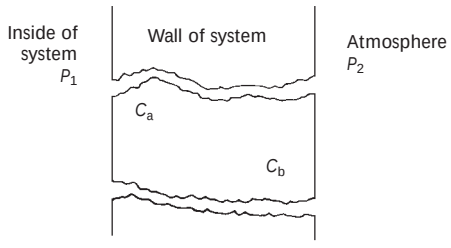
In its general form, the total conductance for n individual leaks connected in parallel is given by the sum of the individual conductances as in Eq. 50:

(50) $C_T = C_1 + C_2 + C_3 + \ldots + C_n$

Graphical Determination of Conductance for Molecular Flow through Tubes and Orifices

The preceding equations give the relationships required to calculate conductance under various conditions. In practice, calculation of the exact conductance often is not required in leak testing. Also, it has been found that most needed conductance values are for molecular flow through cylindrical tubing and orifices. Figures 10 and 11 have been provided to allow quick determinations. Note that the curves are plotted for air at 20 °C (68 °F) and values must be corrected if another gas or temperature is used.

FIGURE 9. Diagram of typical leak paths connected in parallel.



Legend
C = channel connecting points
P = point where fluid is present

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Improvement of Viscous Flow Leak Test Sensitivity by Increasing Pressure Differential

The change in leakage rate obtained by increasing the pressure applied to a leak to atmosphere is used to great advantage in leak testing under conditions where the leakage flow is viscous in nature, as illustrated in Fig. 12. For example, suppose that the leakage rate is 1×10^{-8} Pa·m^3·s^{-1} (1×10^{-7} std cm^3·s^{-1}) from a system with an internal gage pressure of 1 atm (absolute internal pressure of 200 kPa or 2 atm), as indicated by point P_1 in Fig. 12. It is desired to determine the new absolute internal pressure P_2 needed to make the leakage rate 50 times higher, or 5×10^{-7} Pa·m^3·s^{-1} (5×10^{-6} std cm^3·s^{-1}). From Fig. 12, the new flow rate at Point P_2 is seen to be obtained with an absolute internal pressure of 1.23 MPa (12.3 atm), as shown on the horizontal scales.

Effect of Variations in Tracer Gas Concentration

Under viscous flow conditions, which are usually encountered when leak testing pressurized systems, the flow rate increase resulting from higher pressure differentials may also be used to conserve tracer gas. In a mixture of two gases such as helium and nitrogen, each gas will flow through a leak at the same rate regardless of their concentrations in the mixture. Thus, if a 10 percent tracer gas in 90 percent carrier gas mixture is used, the test sensitivity will be 10 percent of what it would be if 100 percent tracer gas were used at the same working pressure. To bring the test sensitivity back to a leakage rate increase ratio of 1, the pressure would have to be raised enough to increase the flow by a factor of 10. Suppose that a tank must be brought to an absolute pressure of 10 MPa (1.5×10^3 lb$_f$·in.$^{-2}$) and leak tested with helium. To save money, it is desired (1) to use the smallest amount of helium that will give adequate sensitivity and (2) to

FIGURE 10. Conductance of cylindrical tubes of different lengths and inside diameters for air at 20 °C (68 °F). 1 L = 1 dm^3 = 0.028 ft^3.

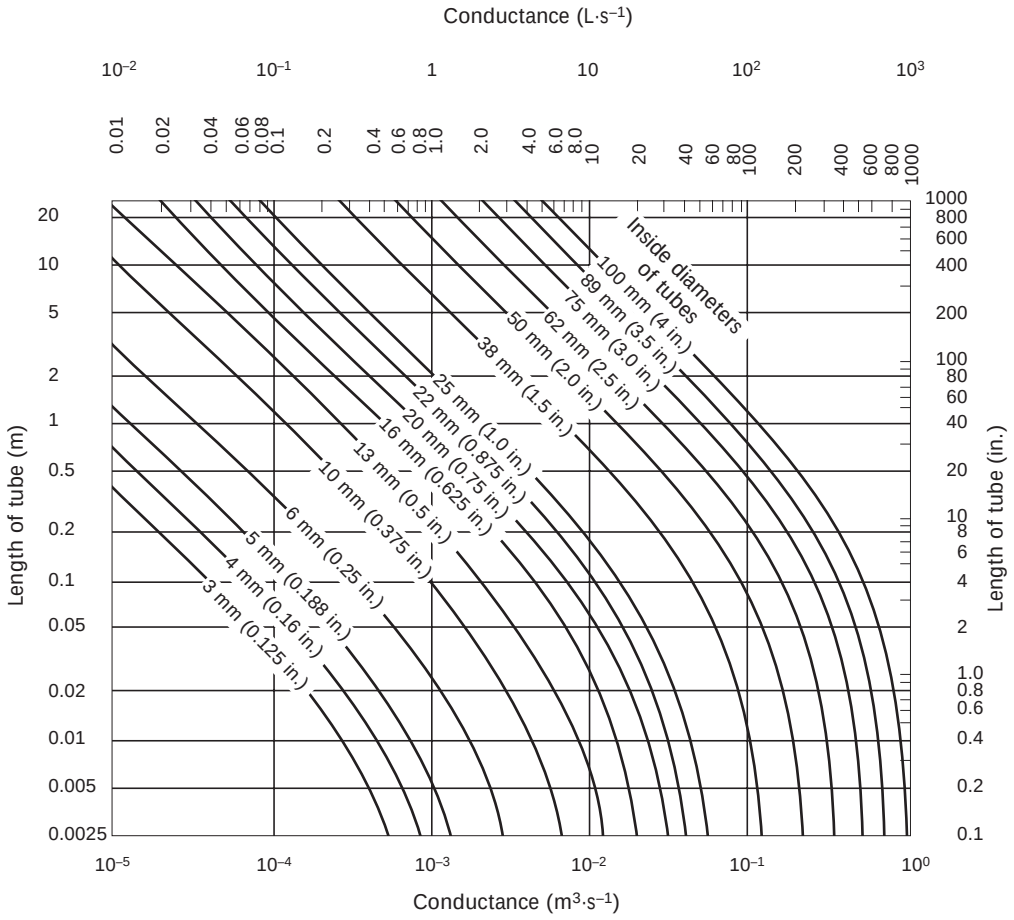

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FIGURE 11. Conductance of orifices for air at 20 °C (68 °F), molecular flow. For curve A read left vertical scale; for curve B read right vertical scale (1 m^3 = 35 ft^3).

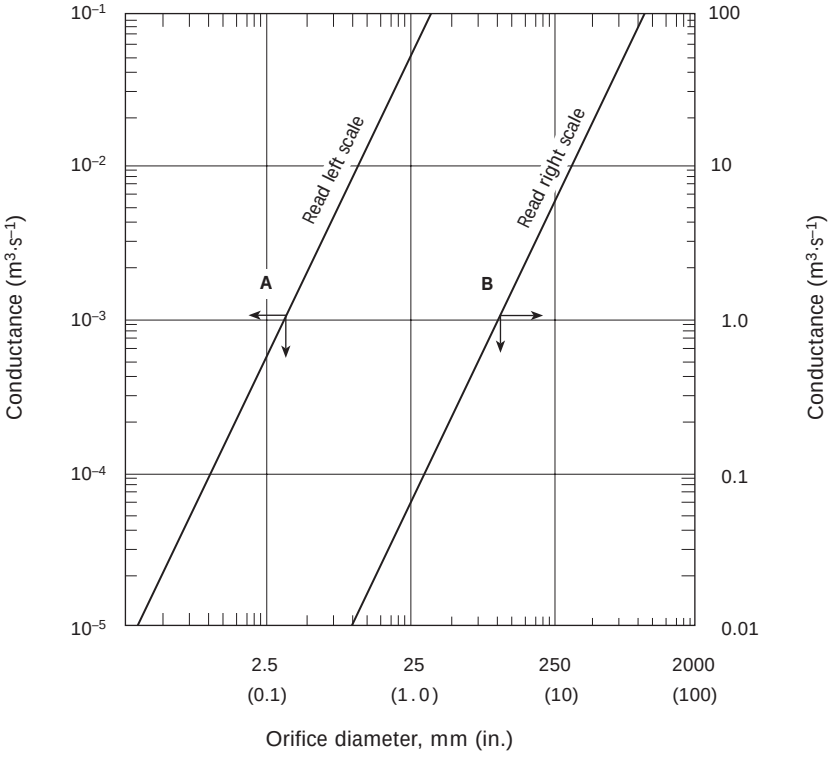
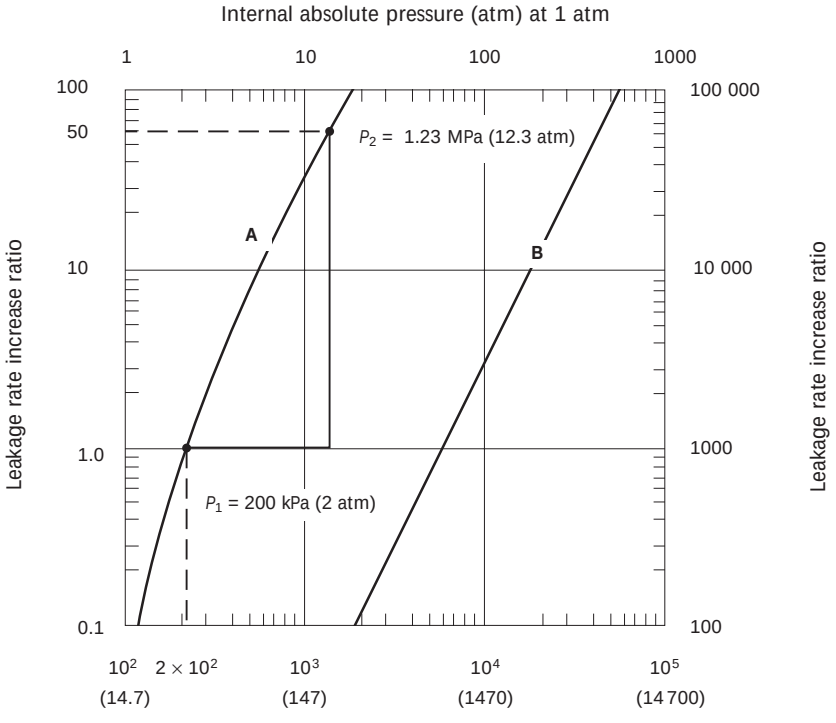


FIGURE 12. Viscous leakage rate as function of internal pressure of system leaking to atmosphere when pressurizing with 100 percent gas. For curve A read left vertical scale, for curve B read right vertical scale.



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pressurize the rest of the way with nitrogen. The minimum detectable leakage should be at least 1×10^{-8} Pa·m^3·s^{-1} (1×10^{-7} std cm^3·s^{-1}) at 100 kPa (1.0 atm) pressure differential. It is desired to calculate the percentage of helium that should be used after reaching an absolute pressure of up to 10 MPa (1.5×10^3 lb$_f$·in.$^{-2}$).

The specified minimum detectable leakage rate of 1×10^{-8} Pa·m^3·s^{-1} (1×10^{-7} std cm^3·s^{-1}) requires that the leak test sensitivity be standard or the same as it would be if 100 percent tracer was used at 100 kPa (1 atm) pressure difference. From Fig. 12 it is seen that an absolute pressure of 10 MPa (100 atm) results in a leakage rate increase factor of 3300. Thus, the helium concentration after pressuring up should be 1/3300 or 0.03 percent. Figure 13 is very similar to Fig. 12 and is used in the same manner. The difference is that Fig. 13 is plotted for conditions where high vacuum is on the low pressure side of the pressure boundary. Figure 13 still assumes viscous flow conditions.

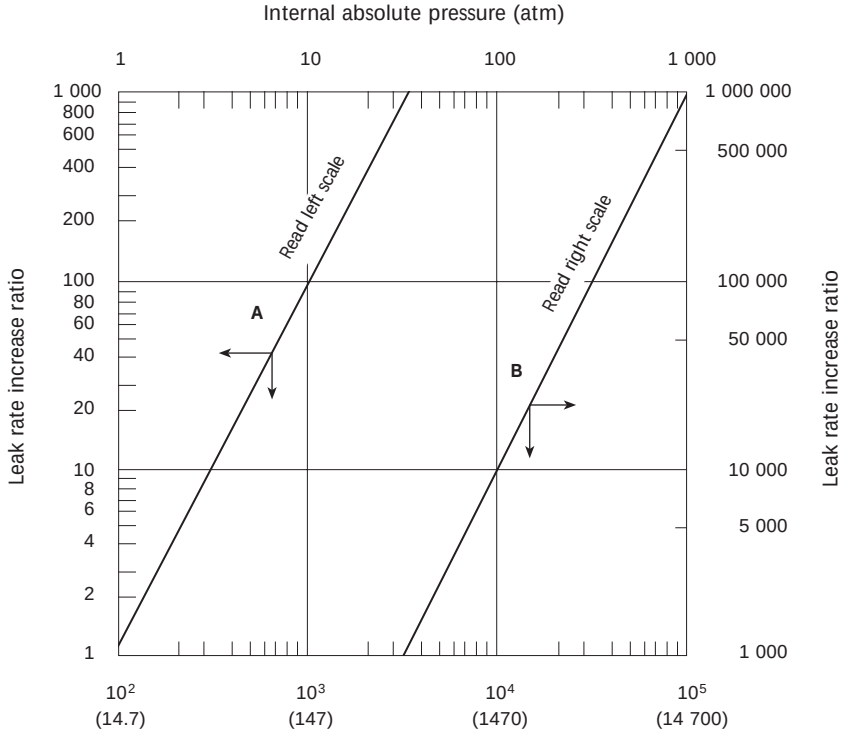
Effect of Increasing Pressure Differential across Molecular Leak Flow

Figure 14 shows the effect of changing the pressure differential across a leak when the flow conditions are molecular. As would be predicted by Eq. 23, the increase of gas flow is a linear function of pressure. Under conditions of molecular flow, the amount of tracer gas flowing through a leak is not a function of the total pressure. It depends only on the partial pressure of the tracer gas. Therefore, there would be no advantage in raising the total pressure difference without raising the tracer gas pressure.

Conversions between Leakage Rates with Different Tracer Gases

Many occasions will arise where it will be necessary to express a leakage rate (flow rate) or conductance in terms of a particular tracer gas when it has been measured using a different tracer gas. For

FIGURE 13. Viscous leakage rate as function of pressure differential during vacuum testing, pressurizing with 100 percent tracer gas. Read left vertical scale for curve A and right vertical scale for curve B.



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example, a specification may state that a certain part cannot leak more than a given amount for air, but helium tracer gas is used in testing. To be able to convert a measured helium leakage rate to an equivalent air leakage rate, the type of flow must first be identified. After the flow type is determined, the conversion may be made. Also, many times the conductance for a piece of tubing or other item must be determined. Conductance is the part of the flow equations that contains the term describing either molecular mass or viscosity of the gas that is flowing. The conductance of a given system will be quite different for two gases having different properties. In leak testing work, this situation is encountered where the pumpdown time of a system for air must be determined and then response and cleanup times for helium must be determined.

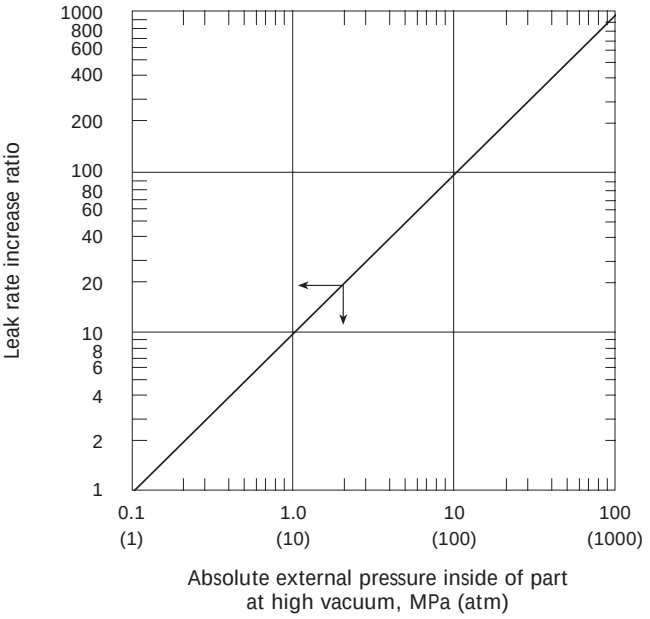
Conversion of Viscous Flow Rates between Different Gases

If a flow rate has been identified as viscous for one gas, the viscous flow for any other gas may be determined using the expression given in Eq. 51:

$$\textbf{(51)} \quad Q_2 = \frac{n_1}{n_2} Q_1$$

where Q_1 is flow rate (any units) for gas 1, Q_2 is flow rate (same units as gas 1) for gas 2, n_1 is viscosity (any units) for gas 1 and n_2 is viscosity (same units as gas 1) for gas 2.

FIGURE 14. Molecular leakage rate as function of pressure differential in vacuum leak testing, pressurizing with 100 percent tracer gas.



Conversion of Viscous Conductance between Different Gases

Dividing both sides of Eq. 51 by the pressure drop will give conductance C rather than flow Q. Any two conductances C_1 and C_2 will then have a relationship given in Eq. 52:

$$\textbf{(52)} \quad C_2 = \frac{n_1}{n_2} C_1$$

where C_1 is conductance (any units) for gas 1, C_2 is conductance (same units as gas 1) for gas 2, n_1 is viscosity (any units) for gas 1 and n_2 is viscosity (same units as gas 1) for gas 2.

A few comparisons that may be used for converting higher conductance or flow from helium flow rates to flow rates for other gases are shown in Table 8.

TABLE 8. Comparison of viscous flow rates of other gases with helium flow rates.

To Convert to	Multiply Helium Flow by
Q of argon	0.883
Q of neon	0.626
Q of hydrogen	2.23
Q of nitrogen	1.12
Q of air	1.08
Q of water vapor	2.09

Conversion of Molecular Flow Rates between Different Gases

If molecular flow occurs, the flow rate for one gas may be compared to the flow rate for any other gas by Eq. 53:

$$\textbf{(53)} \quad Q_2 = \sqrt{\frac{M_1}{M_2}} Q_1$$

where Q_1 is flow (any units) for gas 1, Q_2 is flow (same units as gas 1) for gas 2, M_1 is molecular mass for gas 1 and M_2 is molecular mass for gas 2.

Conversion of Flow Rates for Molecular Conductance

The conductance under conditions of molecular flow for one gas may be compared to the conductance for another by using the expression of Eq. 54:

$$\textbf{(54)} \quad C_2 = \sqrt{\frac{M_1}{M_2}} C_1$$

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where C_1 is conductance (any units) for gas 1, C_2 is conductance (same units as gas 1) for gas 2, M_1 is molecular mass for gas 1 and M_2 is molecular mass for gas 2.

A few comparisons that may be used for converting either conductance or flow are given in Table 9.

TABLE 9. Comparison of molecular flow rates of other gases with helium flow rates.

To Convert to	Multiply Helium Flow by
Q of argon	0.316
Q of neon	0.447
Q of hydrogen	1.410
Q of nitrogen	0.374
Q of air	0.374
Q of water vapor	0.469

Effect of Temperature on Gas Conductance with Molecular Flow

The effect of temperature on conductance when the flow is molecular should not be overlooked. As can be seen in Eq. 35 and 36, the conductance changes in direct proportion with the square root of gas temperature. The expression of Eq. 55 is for a variation in gas conductance resulting from a change in temperature only, with pressure and dimensions remaining constant:

$$C_2 = \sqrt{\frac{T_2}{T_1}}\, C_1 \quad (55)$$

where C_1 is conductance at temperature T_1; C_2 is conductance at temperature T_2; T_1 is starting temperature, kelvin; and T_2 is new temperature in kelvin. T_1 and T_2 must be absolute temperatures.

Relative Sensitivities of Leak Testing Techniques

When choosing a test technique it is advantageous to have an insight into the relative sensitivities of the various techniques. Obviously, the test sensitivity does not equal the published ultimate sensitivities of the various detecting devices because of many variables. Table 10, showing relative sensitivities, may be used to assist in choosing potentially satisfactory leak testing techniques.

Test Variables Limiting Leak Testing Sensitivities

Some factors that prevent leak testing devices from attaining their ultimate sensitivities include geometry, sampling efficiency, tracer economy and noise (or contamination). Geometry enters the picture because any instrument should and must respond only to local conditions at its sampling inlet. Two things are of interest in the leak evaluation process: the space coordinates of the leaking orifice and the mass rate of leakage. The effects of leak location and leakage rate on the concentration of tracer at the instrument depend on convection and diffusion of the tracer gas.

Sampling efficiency may be thought of both as a measure of how nearly all of the quantity to be measured is used in making the measurement and as a measure of how well extraneous responses can be excluded. Many leak detectors must operate with their active parts in a partial vacuum. This limits the rate at which samples of the surrounding air can be ingested for analysis. Other leak testing instruments may take in the sample so violently that extra turbulences are created near the sampling point. The sampling problem is somewhat interrelated with the noise and contamination problem.

The ultimate sensitivity of most leak testing instruments is quoted on the basis of 100 percent tracer concentration in the system or, equivalently, on the amount of tracer leaking. In a practical situation this concentration is necessarily kept down for reasons of safety or economy and sometimes because of corrosiveness of the tracer. With reduced tracer concentration, the leakage sensitivity is reduced proportionately. With 1 percent tracer concentration the sensitivity figure is correspondingly reduced by a factor of 100.

Control of Ambient Concentrations of Tracer Gases

Changes in tracer gas concentration due to leaks are self obscuring in the presence of random variations in the ambient tracer gas concentration. Background levels of tracer gas in the atmosphere disturb the predicted gas concentration pattern. The problem of distinguishing leaks from increasing and randomly varying background contamination may reduce instrument sensitivities by orders of magnitude or even destroy test sensitivity altogether.

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TABLE 10. Relative ultimate leakage sensitivities of various leak testing methods under ideal conditions with very high concentrations of tracer gases. (These numbers are not intended to be used as guides in practical leak testing.)

Minimum Detectable Leakage Test Technique	Leakage Rate	
	$Pa \cdot m^3 \cdot s^{-1}$	(std $cm^3 \cdot s^{-1}$)
Pressure drop using liquids	Depends on volume tested and gage range	
Pressure drop using gases	Depends on volume tested	
Pressure rise	Depends on volume tested	
Ultrasonic leak detector	10^{-2}	(10–1)
Volumetric displacement (gas flow meter)	10^{-3}	(10^{-2})
Gas discharge	10^{-3}	(10^{-2})
Ammonia and phenolphthalein	10^{-3} to 10^{-4}	(10^{-2} to 10^{-3})
Ammonia and bromocresol purple	10^{-3} to 10^{-4}	(10^{-2} to 10^{-3})
Ammonia and hydrochloric acid	10^{-3} to 10^{-4}	(10^{-2} to 10^{-3})
Ammonia and sulfur dioxide	10^{-3} to 10^{-4}	(10^{-2} to 10^{-3})
Halide torch	10^{-4}	(10^{-3})
Air bubble in water	10^{-4} to 10^{-5}	(10^{-3} to 10^{-4})
Air and soap or detergent	10^{-4} to 10^{-5}	(10^{-3} to 10^{-4})
Thermal conductivity	10–5	(10^{-4})
Infrared	6×10^{-4} to 6×10^{-5}	(6×10^{-3} to 6×10^{-4})
Hydrogen Pirani technique	10^{-7}	(10^{-6})
Hot filament ionization gage	10^{-7} to 10^{-8}	(10^{-6} to 10^{-7})
Mass spectrometer detector probe	10^{-6} to 10^{-8}	(10^{-5} to 10^{-7})
Halogen diode detector	10^{-7} to 10–9	(10^{-6} to 10^{-8})
Hydrogen bubbles in alcohol	5×10^{-7}	(5×10^{-6})
Palladium barrier detector	10^{-8} to 10^{-9}	(10^{-7} to 10^{-6})
Mass spectrometer envelope	10^{-10}	(10^{-9})
Radioactive isotopes	10^{-9} to 10^{-13}	(10^{-8} to 10^{-12})

Any gas tracer system, no matter how sensitive, that responds to the simple absolute level of concentration will soon become incapable of detecting leakage when the ambient tracer concentration rises to the level capable of giving spurious signals. This is the major failing of the simple halogen leak detector.

Two solutions to the background problem immediately present themselves: (1) keep the ambient concentration low and (2) use a gradient sensor (differential detector). One such instrument actually has two separate detection cells (Chambers where the temperature compensator detects are mounted). Each cell has an individual intake port. The dual detectors continually compare the thermal conductivity of the sample gas (from potential leakage sources) with that of the ambient atmosphere. When the sample cell intake is not near a leak, the two detection cells are sampling the same gas concentration and their combined output is zero, giving no output reading. Only when the leak area is encountered by the leakage sample intake does the instrument respond.

The differential detector prevents interference from gases in the atmosphere and working area. It eliminates the need for selectivity to any particular gas. Leak testing can be performed in areas of high gas concentration that are caused by accumulated leakage or by venting tracer gases. The need for controlled environment and ventilating systems is minimized. The reference intake of the differential detector is prevented from sampling in the immediate area of the leak to avoid fast transients and confusing indications. However, the differential detector leak sensor is less sensitive than either the heated anode halogen leak detector or the helium mass spectrometer leak detector.

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PART 4. Mathematical Theory of Gas Flow through Leaks

Mechanisms of Mass Transfer in Gas Flow

Mass transfer attributed to leakage can occur in two modes: pneumatic flow and permeation. Pneumatic flow occurs when leakage is by passage of fluid through finite holes. Permeation is passage of a fluid into, through and out of a solid barrier having no holes large enough to permit more than a small fraction of the molecules to pass through any one hole.

Leakage Rates for Different Modes of Pneumatic Flow of Gas in Leaks

Pneumatic gas flow in leaks may be placed in five categories: turbulent, laminar, molecular, transition and choked leakage flows. The approximate ranges of flow rates for various pneumatic modes of gas flow follow.

1. Turbulent flow occurs with leakage rate above 10^{-3} Pa·m^3·s^{-1} (10^{-2} std cm^3·s^{-1}).
2. Laminar flow occurs with leakage rates in the range from 10^{-2} to 10^{-7} Pa·m^3·s^{-1} (10^{-1} to 10^{-6} std cm^3·s^{-1}).
3. Molecular flow is most probable with leakage rates below 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}).
4. Transition flow occurs in the gradual transition from laminar to molecular flow.
5. Choked flow occurs when the flow velocity approximates the speed of sound in the gas.

Laminar and molecular flows are the predominant modes of leakage flow in the range of leakage rates of interest in most leak testing.

Characteristics of Laminar (or Viscous) Flow

The laminar flow of a fluid in a tube is defined as a condition where the velocity distribution of the fluid in the cross section of the tube is parabolic. Laminar flow is one of the two classes of viscous flow, the other class being turbulent flow. Because turbulent flow is rarely encountered in leaks, the term viscous flow is sometimes incorrectly used to describe laminar flow in leak testing work.

The most familiar laminar flow equation was developed by Poiseuille. Poiseuille's equation for laminar flow through a straight tube of circular cross section is given in Eqs. 21 and 22. Poiseuille's equation has been substantially verified experimentally and is applicable where the length and diameter of the flow passage are known. This is not the case for most leaks. Equation 21 can be rewritten in the form of Eq. 56:

$$\textbf{(56)}\quad Q = KP_a \frac{P_1 - P_2}{n}$$

K represents the constants of the two geometry factors of length l and diameter d of the tubular leak passage, as shown in Eq. 57:

$$\textbf{(57)}\quad K = \frac{\pi r^4}{8l}$$

Laminar flow takes place when the Reynolds' number of flow is lower than the defined critical value. The Reynolds' number is a unitless quantity that defines the flow conditions and is given by Eq. 58:

$$\textbf{(58)}\quad N_{\mathrm{Re}} = \frac{d\rho F}{n}$$

where N_{Re} is Reynolds' number, p is fluid density, n is gas viscosity, F is average flow velocity across a plane in the tube and d is diameter of the leak (compare with Eq. 20).

Reynolds' Number for Ideal Gas

By substituting the ideal gas equation (see Eq. 9) into Eq. 58, the expression for the Reynolds' number for an ideal gas becomes Eq. 59:

$$\textbf{(59)}\quad N_{\mathrm{Re}} = \frac{Q}{d}\frac{4M}{\pi nRT}$$

where M is molecular mass, R is molar gas constant, T is absolute temperature, Q is leakage rate, d is leak diameter and n is gas viscosity.

The critical value of Reynolds' number has been shown to depend on the

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entrance conditions, roughness of the walls of a tube and shape of the flow path. In general, for smooth tubes with well rounded entrances, the critical value is about 1200.

Equation for the Viscosity of a Gas

The kinetic theory of gases states that the viscosity of a gas is given by the relationship of Eq. 60:

$$\textbf{(60)} \quad n = \frac{mF_a}{3\sqrt{2}\,\pi\sigma^2}$$

where F_a is a average velocity of the individual molecules, m is molecular mass, σ is molecular diameter and n is viscosity of gas. The average velocity of a gas molecule is given by Eq. 61:

$$\textbf{(61)} \quad \bar{F} = \sqrt{\frac{8RT}{\pi M}}$$

The mass m of the individual molecules is given in terms of the molecular mass M of a specific gas:

$$\textbf{(62)} \quad m = \frac{M}{N}$$

In Eq. 62, N is Avogadro's number, i.e., number of molecules per mole. Substituting Eq. 61 and 62 into Eq. 60 results in Eq. 63 for the viscosity of a gas:

$$\textbf{(63)} \quad n = \frac{2\sqrt{MRT}}{3\sqrt{\pi^3}\,N\sigma^2}$$

Equation 63 shows that the viscosity of a gas is independent of pressure and is proportional to the square root of absolute temperature.

Characteristics of Laminar Gas Leaks

The two most important characteristics of laminar leaks shown by Eq. 21 and 22 are (1) the flow is proportional to the difference between the squares of the pressures upstream and downstream of the leak and (2) the leakage is inversely proportional to the leaking gas viscosity. Table 11 shows that the viscosity of most gases is similar. Therefore, a change of leaking gas will not markedly increase the sensitivity of the leak testing technique unless this change of gas implies a change of instrument sensitivity.

However, as shown in Fig. 15, increasing the pressure difference across the leak by a factor of a little over three will increase the flow rate through this leak by a factor of ten. Obviously then, when the leaks to be measured are in the laminar flow range, the simplest way to increase leakage sensitivity is by an increase of pressure across the leak.

Equations for the Mean Free Path of Gaseous Molecules

The mean free path length is the average distance that a molecule travels between successive collisions with the other molecules of an ensemble. The mean free path λ of gas molecules is given by Eq. 64:

TABLE 11. Mean free paths at 25 °C (77 °F), molecular diameters, and viscosities for gases and vapors used in leak testing.

Gas	Mean Free Path (mm·Pa)	Molecular Diameter (pm)	Viscosity (μPa·s)
Acetylene			9.2
Air			16.9
Ammonia			9.4
Argon	7.23	358	20.8
Benzene	1.53	765	6.9
Carbon dioxide	4.49	465	13.5
Carbon disulfide			8.9
Carbon monoxide			17.1
Dichloromethane			
Ethane	3.21	537	8.5
Ethyl alcohol			8.2
Ethylene			9.3
Refrigerant–11			10.3
Refrigerant–12			11.8
Refrigerant–21			10.8
Refrigerant–22			12.0
Refrigerant–113			9.8
Refrigerant–114			
Refrigerant–134a			
Helium	19.5	218	17.8
Hydrogen	12.2	275	8.3
Hydrogen sulfide			11.8
Methane	5.27	419	10.0
n–Butane	1.86	706	10.0
n–Pentane	1.51	782	
n–Hexane	1.31	842	
Neon	13.70	260	
Nitric oxide			17.8
Nitrogen			16.8
Nitrous oxide			13.3
Oxygen	6.99	364	19.1
Propane	2.32	632	7.7
Sulfur dioxide			11.6
Sulfur hexafluoride			
Water	4.23	468	8.8
Xenon			21.0

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$$\textbf{(64)} \quad \lambda = \frac{1}{\sqrt{2}\,\pi\, n_1\, \sigma^2}$$

where n_1 is number of molecules in 1 cm^3 volume and σ is molecular diameter. The molecular density n_1 of gaseous molecules per unit volume is given by Eq. 65:

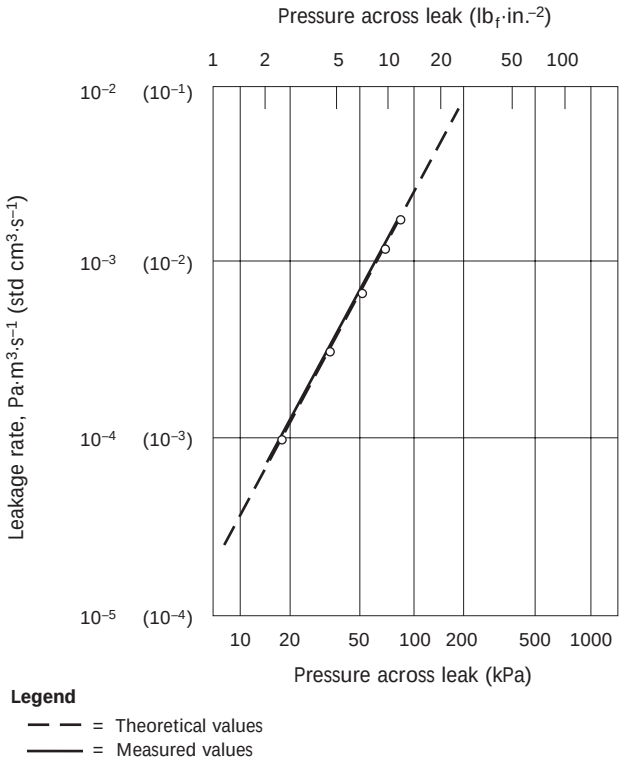
$$\textbf{(65)} \quad n_1 = \frac{m}{M}\,\frac{N}{V}$$

where m is mass of gas, M is molecular mass of gas, N is Avogadro's number, i.e., 6.023×10^{23} molecules per mole; n_1 is number of gaseous molecules per unit volume; and V is volume containing the gas. Replacing the volume V in Eq. 65 by its value mRT/P from the ideal gas law of Eq. 9 and substituting it in Eq. 64 results in the equation for the mean free path length λ of Eq. 66:

$$\textbf{(66)} \quad \lambda = \frac{MRT}{\sqrt{2}\,\pi P N \sigma^2}$$

which shows that at constant pressure the mean free path is proportional to absolute temperature. However, if the amount of gas in a volume is kept constant, the mean free path is independent of temperature, as indicated in Eq. 64. In Eq. 66, R is the specific gas constant and MR equals the molar gas constant.

FIGURE 15. Relation of leakage to pressure differential with laminar flow of helium gas in typical hardware leak.



The molecular diameters and mean free paths of typical leak testing gases and vapors are listed in Table 11. As a convenient calculation guide, the mean free path, in meters, of air at room temperature is given by Eq. 67:

$$\textbf{(67)} \quad \lambda_{air} = \frac{6.8 \times 10^{-3}}{P}$$

The pressure P is expressed in pascal in Eq. 67 (compare with earlier Eq. 16).

Equation for Molecular Flow of Gases

Molecular flow is flow through a duct under conditions where the mean free path is greater than the largest dimension of a transverse section of the duct. In such a flow, each atom moves independently by random movement. Net flow is from a volume of high concentration to one of low concentration. The original mathematical derivations of molecular flow are attributed to Knudsen (see Eq**s**. 23 to 27). The rate of gas flow in a long tube is given by Eq. 68:

$$\textbf{(68)} \quad Q = \sqrt{\frac{2\pi RT}{M}}\,\frac{d^3}{6l}\left(P_1 - P_2\right)$$

where d is diameter of the tube, l is length of the tube and P_2 and P_1 are pressures at the two ends. For the formula of Eq. 68 to apply, the tube must be of a circular cross section. For tubes and ducts of a noncircular cross section, the conductance is less than for tubes of circular cross section and equal area. Equation 68 applies only if the tube is much longer than its diameter. Any difficulty experienced by a molecule in entering the tube must be negligibly small compared to the difficulty in transversing its length.

Equation for Free Molecular Entry of Gases into a Small Aperture

If gas molecules experience difficulties in entering a small leak opening, the kinetic theory shows that the rate of free molecular escape of gas from the container into a small aperture of area A is given by Eq. 69:

$$\textbf{(69)} \quad Q = \sqrt{\frac{RT}{2\pi M}}\,A\left(P_2 - P_1\right)$$

In the case of an aperture, the leak opening does not have to be circular for this equation to apply.

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Flow Characteristics of Molecular Leaks

The conductance of lines and apertures in molecular flow is independent of pressure. Calculations may be made of the effect of turns, apertures and change in tube diameter to calculate the overall flow in a leak.

Equations 23 to 27, 68 and 69 demonstrate the general form of relations for molecular flow through leaks. They are not applicable in most leakage situations because the leak length and diameter are not known. The molecular flow of each individual species in a gas mixture is inversely proportional to the square root of the individual masses. Therefore, a certain amount of separation of gaseous species takes place during flow through a leak. In molecular flow, the gas molecules travel independently of each other. Thus, it is possible for random molecules to travel from a part of a system at low pressure to another part of the system at a higher pressure.

Knudsen Equation for Transition Flow

The transition from laminar flow to molecular flow is gradual. The mathematical treatment of this region is extremely difficult, but is necessary because a leak from a volume to a vacuum necessarily involves a transition from laminar to molecular flow. Equation 68 shows that the conductivity of a passage in molecular flow is proportional to the cube of the passage diameter and independent of pressure. Conversely, Eq. 21 and 22 show that the conductivity of the same passage in laminar flow is proportional to the pressure.

Knudsen derived a semiempirical formula for the conductance of gas flowing through long tubes in the transition flow region:

$$\textbf{(70)}\quad C = C_{\text{viscous}} + ZC_{\text{molecular}} = \frac{\pi}{8}\left(\frac{d}{2}\right)^4 \frac{P_a}{n\,l} + \left(\frac{1}{6}\sqrt{2\,\frac{RT}{M}}\;\frac{d^3}{l} \times \frac{1 + \sqrt{\frac{M}{RT}}\;P_a\;\frac{d}{n}}{1 + 1.24\,\sqrt{\frac{M}{RT}}\;P_a\;\frac{d}{n}}\right)$$

In this case, the gas flow rate $Q = C(P_1 - P_2)$, where C is defined by Eq. 70. Equation 70 is valid providing that:

1. the flow is not turbulent in any part of the pipe and
2. the pressure difference between the ends is not so great that the mechanism of the flow, i.e., laminar or molecular, changes along the pipe.

Although the first of these conditions is usually satisfied in the leak, the second generally is not: that is, the transition from laminar to molecular flow does take place within a leak. Equation 70 at low pressures becomes an equation of molecular flow, whereas at high pressure this equation reduces to one of strictly laminar flow.

Knudsen used Eq. 70 to represent his experimental data. This equation has the effect of molecular flow added to the effect of laminar flow; consequently, it is not an actual representation of the flow mechanism taking place in the leak. The phenomenon is better visualized by realizing that both are occurring at the same time.

Burrows Equation for Transitional Flow

Burrows combined Eq. 23 to 27 for laminar flow with that of Eq. 68 for molecular flow to obtain the general relation for transitional flow given in Eq. 71:

$$\textbf{(71)}\quad Q = \frac{\pi}{8}\left(\frac{d}{2}\right)^4 \frac{P_a}{nl}\left(P_1 - P_2\right) + \sqrt{\frac{2\pi RT}{M}}\;\frac{d^3}{6l}\left(P_1 - P_2\right)$$

In a way, Eq. 71 accurately represents the events occurring in the leak. Both laminar and molecular flow always occur in a leak. However, laminar flow is insignificant at low pressures. The molecular flow mode contributes little to total flow at high pressures.

Equation 71 is not completely accurate because of a slipping of molecules in transition flow. In laminar flow, the velocity of the molecular layers is proportional to their distance from the wall, the first layer being stationary. In the transition region, slipping of the gas over the walls of the tube occurs; that is, the flow velocity at the walls is not zero. At pressures below the viscous limit, the slip correction becomes an appreciable contribution to the total conductance.

With further reduction in pressure, the dependence of flow conductance on pressure becomes more complex. The flow characteristics begin a progressive change from those of viscous slip flow to those of molecular flow, where the conductance becomes independent of the pressure. The complete transition from viscous to molecular flow takes place over roughly

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two orders of magnitude change in pressure. This effect of slip can change the predicted flow rate by at least 20 percent. Because of this effect, Eq. 70 better represents flow in the transition region but cannot handle the total transition region.

Other authors have attempted to derive equations to represent this phenomenon of transition from one type of flow to another. One simple way is to calculate laminar flow through one section of the tube, calculate molecular flow through another and approximate the region between them.

Characteristics of Turbulent Flow of Gases

In viscous flow above a critical value of the Reynolds' number (about 2100 in the case of circular pipe flow), flow becomes unstable, resulting in innumerable eddies or vortexes in the flow. Any particle in turbulent flow follows a very erratic path, whereas in laminar flow the particle follows a smooth line. Turbulent flow occurs only in rather large leaks because it requires relatively high velocity.

The laws for turbulent flow are quite different from the laws for laminar flow. The equation relating mass flow rate Q in units of pressure × volume/time may be written as Eq. 72:

$$\textbf{(72)} \quad Q = \pi\sqrt{d^5}\sqrt{\frac{RT\left(P_1^2 - P_2^2\right)}{16fMl}}$$

The friction factor f depends on roughness of the channel walls and can be considered a constant in fully developed turbulent flow.

Theory of Choked (or Sonic) Flow of Gases through Leaks

The phenomenon of choked flow (also known as sonic flow) of gases is described above. Two conditions required for choked flow to occur are:

1. The flow passage must be in the form of an orifice or venturi in which only negligible fractional losses occur upstream of the orifice or throat of the venturi.
2. The ratio of downstream to upstream pressure must be below a certain critical value.

The critical ratio r_c of downstream pressure P_2 of upstream pressure P_1 required for choked flow is given by Eq. 73:

$$\textbf{(73)} \quad r_c = \frac{P_2}{P_1} = \left(\frac{2}{\gamma + 1}\right)^{\frac{\gamma}{\gamma - 1}}$$

The term γ is the ratio of specific heats defined by Eq. 75 below. The velocity of sound through a gas can be written as in Eq. 74:

$$\textbf{(74)} \quad F_c = \frac{2\gamma}{\gamma + 1}\sqrt{\frac{RT_1}{M}}$$

where F_c is velocity of sound and T_1 is absolute temperature upstream of the orifice where the velocity is low. The ratio of specific heat at constant pressure to that at constant volume is described by gamma (γ), the ratio of specific heats defined in Eq. 75:

$$\textbf{(75)} \quad \gamma = \frac{C_p}{C_v}$$

where C_p is heat capacity at constant pressure and C_v is heat capacity at constant volume.

The mass flow rate under a choked flow condition is given by Eq. 76:

$$\textbf{(76)} \quad Q = \frac{\frac{\pi d^2 P_1 C_o}{4M}}{\left(RT_1\,\gamma\,\frac{2}{\gamma+1}\right)^{\frac{\gamma+1}{\gamma-1}}}$$

where d is orifice diameter, P_1 is upstream pressure and C_o is orifice discharge coefficient.

The value of γ for an ideal monatomic gas is 1.67. For polyatomic molecules, the heat energy supplied is used for increasing not only the kinetic energy of translation but also the kinetic energy of rotation and vibration. Because the same amount of extra energy is required at both constant pressure and constant volume, γ decreases with molecular complexity. Characteristic values of γ are listed in Table 12.

TABLE 12. Specific heats of gases at constant pressure C_p, at constant volume C_v, and as the ratio γ of $C_p \cdot C_v^{-1}$, in joule per mole of gas at 25 °C (77 °F) and 100 kPa (1 atm) pressure.

Gas	C_p	C_v	$C_p \cdot C_v^{-1} = q$
Argon	20.8	12.5	1.67
Helium	20.8	12.5	1.67
Hydrogen	28.8	20.5	1.41
Oxygen	29.5	21.1	1.40
Nitrogen	29.0	20.7	1.40
Carbon dioxide	37.5	28.9	1.29
Ammonia	36.1	27.5	1.31
Ethane	53.1	44.5	1.19
Propane	73.6	65.2	1.13

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Because of the stringent requirements, choked flow is rarely encountered as the predominant flow mode except in very large leaks.

Criteria for Distinction between Modes of Gas Flow in Leaks

Equations have been presented for the various possible modes of flow that can be encountered in a leak. The following rules may be used to predict the mode most likely to occur. In distinguishing between laminar and molecular flow, the size of the passage and the mean free path are the two important parameters. The distinction may be specified by a dimensionless parameter called the Knudsen number. The Knudsen number is defined as the ratio of the mean free path of the molecule to a characteristic dimension of the channel through which the gas is flowing. The Knudsen number is defined by Eq. 77:

$$\textbf{(77)} \quad N_K = \frac{\lambda}{d}$$

where N_K is Knudsen number, λ is mean free path and d is channel diameter.

The type of flow encountered in the various Knudsen number ranges is described by Eqs. 78 to 80:

$$\textbf{(78)} \quad \frac{\lambda}{d} < 0.01$$

for laminar flow,

$$\textbf{(79)} \quad \frac{\lambda}{d} > 1.00$$

for molecular flow and

$$\textbf{(80)} \quad 0.01 > \frac{\lambda}{d} > 1.00$$

for transition flow.

Flow in the viscous region is determined by the Reynolds' number described earlier in Eq. 58 and 59 and repeated in Eq. 81:

$$\textbf{(81)} \quad N_{\mathrm{Re}} = \frac{d\rho F}{n} = \frac{Q}{d}\,\frac{4M}{\pi n R T}$$

where d is channel diameter, ρ is fluid density, F is flow velocity, n is gas viscosity, Q is leakage rate, M is molecular mass, R is gas constant and T is absolute temperature.

The distinction between laminar and turbulent flow is shown by the numerical criteria of Eqs. 82 to 84:

$$\textbf{(82)} \quad N_{\mathrm{Re}} > 2100$$

for turbulent flow,

$$\textbf{(83)} \quad N_{\mathrm{Re}} < 1200$$

for viscous flow and

$$\textbf{(84)} \quad 1200 < N_{\mathrm{Re}} < 2100$$

for either turbulent or viscous, depending on duct conditions.

Choked flow takes place when the pressure ratio between outlet and inlet reaches a certain minimum value. This, of course, depends on other characteristics, such as aperture dimension. The formula for the critical pressure ratio for choked flow depends on the ratio r defined in Eq. 73. The critical ratio below which choked flow takes place is given by Eq. 73. Choked flow cannot take place when P_1 is so low that molecular flow exists.

General Formula for Gaseous Permeation Flow Rate

Permeation is passage of a fluid into, through and out of a solid barrier having no holes large enough to permit more than a small fraction of the molecules to pass through any one hole. The process always involves diffusion through a solid and may involve other phenomena such as adsorption, solution, dissociation, migration and desorption. The general formula for permeation is given by Eq. 85:

$$\textbf{(85)} \quad q = K_p\, A\,\frac{\Delta P}{l} = (SD)\, A\,\frac{\Delta P}{l}$$

where $K_p = SD$; q is rate of mass flow ($\mathrm{Pa \cdot m^3 \cdot s^{-1} \cdot m^2}$); S is solubility coefficient; D is diffusion coefficient; K_p is permeation rate constant (per second); A is area normal to flow (square meter); ΔP is pressure drop along the flow path (pascal); and l is length of flow path (meter). The ΔP in Eq. 85 does not represent absolute pressures, but the difference in partial pressure of the leaking fluid between the two sides of the barrier.

Permeation of Helium through Rubber

Permeation presents a problem in leak testing equipment where the construction materials have a high permeability to the tracer gas. For example, if a component containing a rubber diaphragm 1 mm (0.04 in.) thick and 650 $\mathrm{mm^2}$ (1.0 $\mathrm{in.^2}$) in surface area is leak tested using helium

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gas, a leakage of about 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}) will be measured across the diaphragm.

This leakage is due to permeation of helium through the diaphragm and not to any actual holes. It represents the maximum sensitivity of helium leak testing that can be performed on this component. However, if the component is to be used with another fluid to which the membrane is impermeable, the apparent leakage due to permeation measured during the leak testing has little meaning under operating conditions.

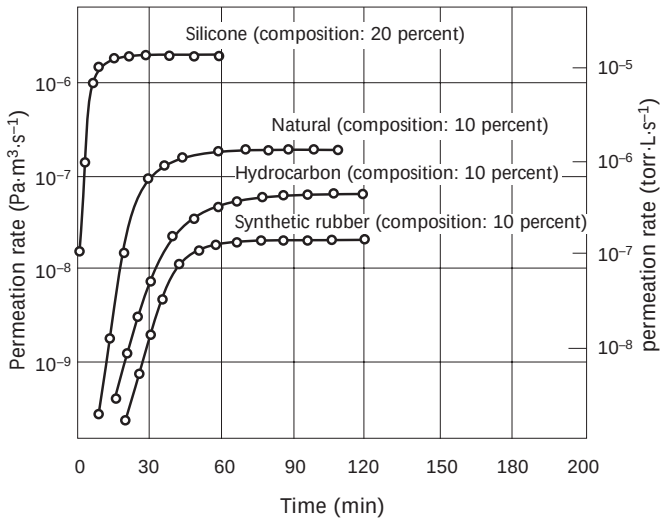
Another example of this type of false reading is a rubber O-ring. Depending on material, a rubber O-ring usually represents a permeability of about 5×10^{-7} Pa·m^3·s^{-1} per centimeter of O-ring surface exposed for every 100 kPa (14.5 lb$_f$·in.$^{-2}$) of differential pressure. Figure 16 is an example of the permeation rates of O-ring of various materials. This permeability does not have to be taken into consideration during routine leak testing if leakage measurement occurs in a time too short to permit the saturation and mass transfer of helium through the O-ring.

Procedures for Reducing Gas Permeability Effects during Leak Testing

To reduce permeability as a factor in leakage measurement, three procedures may be used:

1. The leakage measurement may be taken rapidly, not allowing the material to be saturated with gas. This is only possible if the material is relatively thick. For example, a rubber diaphragm will rapidly saturate and almost immediately show leakage. On the other hand, O-rings are relatively thick and will not saturate rapidly enough to give a reading within a reasonable period of time (5 min). If the diffusivity and solubility of the fluid in the material are known, it is possible to calculate the rate of increase of leakage. However, in many cases (where the leakage path is long), this calculation is not necessary. Rather than calculations, experimental results can determine very quickly if leakage through a thick gasket is inconsequential for short time periods.
2. The maximum permeability of all components and the resulting mass transfer produced by permeability during leak testing may be calculated (refer to Eq. 85, below). In this way, the permeability value will be known and only leakage above this value will be considered as leakage flow.
3. The last and most difficult way is to quantitatively measure the leakage at various pressure differentials. If gas leaks through a hole in the component so that the leak being measured is pneumatic and laminar, the flow is proportional to the square of the pressure differential across the leak. However, if the flow is strictly due to permeation, then the flow through the leak will be directly proportional to the difference in tracer gas concentration across the leak. In this way, the presence of holes in the component can be differentiated from permeation.

FIGURE 16. Permeation rate of helium at differential pressure of 100 kPa (1 atm) through O-rings of 4 × 4 mm (0.16 × 0.16 in.) cross section, per 25 mm (1 in.) of length at 25 °C (77 °F) in units of pascal cubic meter per second (left vertical scale) and torr liter per second (right vertical scale).



General Guide to Estimating Gas Flow Rates through Leaks

Table 10 lists the theoretical ultimate leakage sensitivities of various leak testing techniques under ideal conditions with very high concentrations of tracer gas. It is derived from the various flow equations presented in the text. As may be seen from Tables 8 to 10, the influence of varying the gas is not so great as that of varying the flow mode. Once the flow mode is determined, the conversion to another gas should be relatively easy to make, providing the relationships in Table 10 are in fact correct. The major difficulty is identifying the predominant flow mode.

The data necessary for the conversion of leakage rates between various gases are relatively easy to obtain. For example, the viscosity of many gases is published. Even if the viscosity is not known, approximation should not produce a large

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error. As shown in Table 11, the viscosity of gases at constant temperature varies by less than half an order of magnitude between the most viscous and the least viscous.

For molecular flow, data on the molecular mass of the gases is easily available and should cause no problem in the conversion (see Table 5). If choked flow does occur, the gamma of Eq. 75, necessary for conversion of choked flow leakage, is 1.67 for monatomic gases and rapidly approaches 1 as the complexity of the gas molecule increases.

Effect of Leak Size on Mode of Gas Leakage Flow

By working with a variety of leaks of different sizes and under different conditions, some of the flow modes may readily be eliminated. For example, if the leakage rate is small, it is relatively easy to assume that no turbulent flow will take place. If the leakage goes from high pressure to a slightly lower pressure, but not to a vacuum, it is likely that molecular flow is not the flow mechanism. In this case, the flow may be of a laminar nature and therefore conversion to a second flow pressure is relatively easy. Choked flow is rarely encountered in small leaks.

Another example is that of converting the leakage rate for gas flowing into a vacuum to an anticipated rate for a different pressure driving gas into the same vacuum. If the leak is of relatively small size, 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}) or less, molecular flow will play a major role in such a leak. However, should the leak be relatively large, 10^{-4} Pa·m^3·s^{-1} (10^{-3} std cm^3·s^{-1}) or greater, the leakage will be predominately laminar. If one can accurately predict the type of flow that will predominate in a leak, one could therefore make accurate conversions to a different set of conditions. Unfortunately, the state of the art is such that these predictions are usually not possible.

Estimating Mode of Gas Leakage Flow from Leakage Rate and Pressure

Many authors have predicted the following predomination flow modes in leaks of various sizes: turbulent flow, 10^{-3} Pa·m^3·s^{-1} (10^{-2} std cm^3·s^{-1}); laminar flow, 10^{-2} to 10^{-7} Pa·m^3·s^{-1} (10^{-1} to 10^{-6} std cm^3·s^{-1}); transition flow, 10^{-5} to 10^{-7} Pa·m^3·s^{-1} (10^{-4} to 10^{-6} std cm^3·s^{-1}); molecular flow, 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}) . When there is doubt about the correctness of flow identification, the following procedure is recommended.

1. If pressure is increased, correlate as laminar.
2. If pressure is decreased, correlate as molecular.
3. If gas is changed, correlate as molecular.

Correlation should be performed so that, if an error is made, actual leakage will be no greater than that predicted in the correlation. Correlation of leaks resulting from increased pressure across a leak is not recommended. An actual measurement should be made whenever possible to verify leakage rate.

Equation for Gas Leakage Flow Rate in Laminar Flow

Assuming the flow mode has been identified, the following are sample calculations for correlation of flow rates with the use of different gases and pressure. The first sample calculation is for laminar flow. The general equation for laminar flow of gases is given by Eq. 86:

$$\textbf{(86)} \quad Q = \frac{\pi}{8nl}\left(\frac{d}{2}\right)^4 P_a \left(P_1 - P_2\right)$$

where Q is leakage (mass flow in units of pressure × [volume/time]), d is average diameter of leak hole, P_2 is pressure on the entrance side of the leak, P_1 is pressure on the exit side of the leak, average leak inlet and leak outlet pressures $P_a = (P_1 + P_2)/2$, n is viscosity of the leaking fluid or fluid mixtures and l is leak length. Note that Eq. 86 is equivalent to Eq. 21 given earlier.

The leak dimension of d and l are usually not known. An apparent conductance C may be calculated by the formula, where this apparent conductance is the product of $\pi(d/2)^4/8l$ and any unit conversion factors. From this calculation, an apparent leak geometry factor can be calculated from Eq. 87:

$$\textbf{(87)} \quad C = \frac{\pi}{128}\frac{d^4}{l}$$

If C is calculated only for conversion from one flow to another, the constant does not have to be in compatible units, providing that the same units are used both in solving for C and using the C in correlation equations.

Using the apparent conductance C calculated above, the flow of any gas at operating pressure may be predicted by using Eq. 88:

$$\textbf{(88)} \quad Q = \frac{C}{n}\left(P_1^2 - P_2^2\right)$$

A similar apparent conductance may be calculated for other flow modes using the

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equations given earlier in this section. Such calculations are correct only if the flow mode has been correctly chosen.

Categories of Anomalous Leaks

Four types of leaks have been encountered that do not fit in the categories already discussed: (1) check valve leaks, (2) surface flow leaks, (3) geometry change leaks and (4) self-cleaning leaks.

The errors in leak measurement because of these types of leaks could be greater than any errors inherent in the preceding equations for flow conversions.

Effects of Check Valve Leaks

Examples of check valve and geometry change leaks have been found during studies of leakage phenomena. Figure 17 is a plot of the leakage-pressure differential obtained on a damaged needle valve. It was observed that although the typical laminar flow curve was obtained at a high pressure differential, below this pressure, the leakage abruptly stopped. On increasing the pressure, the leak reappeared. This phenomenon was repeatable. This type of leak would be particularly hard to detect because the leak cannot be seen below a critical pressure.

Effects of Geometry Change in Leaks

The shape of a leak may change with changes in system pressure. As pressure increases, the expansion of system parts resulting from stresses induced by the increased pressure can cause leakage rates of known leaks to increase beyond the predictions of laminar flow theory. Figure 18 illustrates this increase of leakage rate with geometry change.

Effects of Self-Cleaning Leaks

If gaskets under compression are subjected to a high helium pressure and the leakage rate is determined quantitatively, the slope of the pressure leakage line is found to be greater than two. No flow regime would produce such a slope. However, these curves consist of a series of lines with a slope corresponding to that for laminar flow.

Because the increase in leakage could result from a permanent deformation of the gasket, an experiment was run using an aluminum gasket too sturdy to be deformed. Figure 19 shows the data obtained during this experiment. During the original increase in pressure, the leakage increased at a rate greater than the

FIGURE 17. Check valve leakage effect in hardware leak.

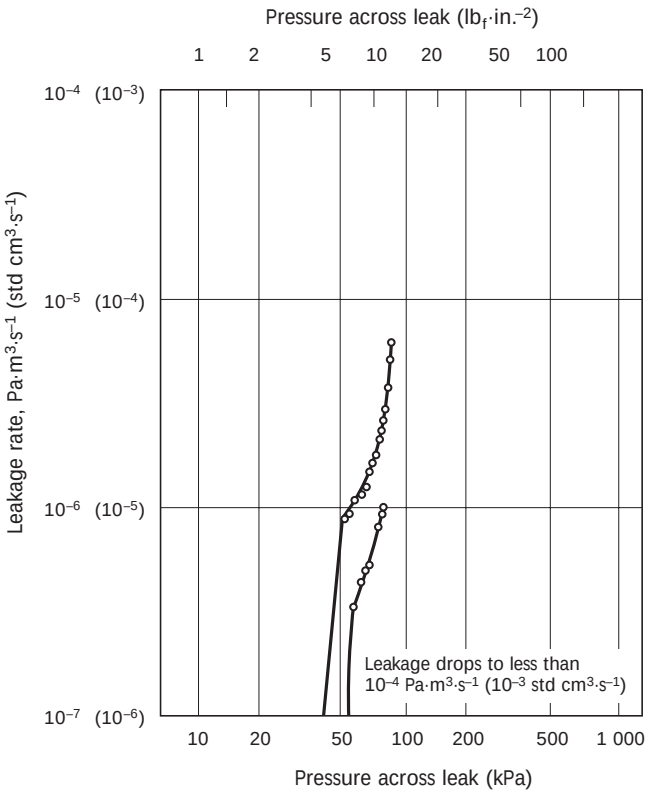
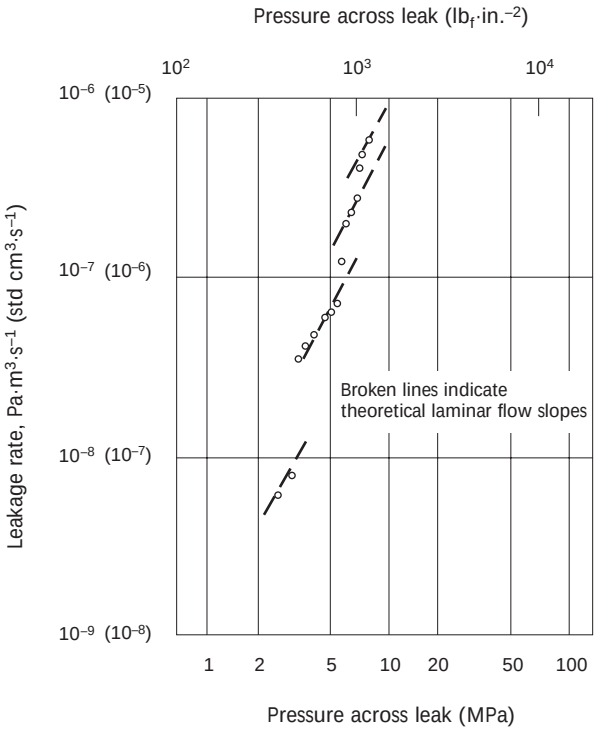


FIGURE 18. Effects on leakage of geometry changes in gasket.



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square of the pressure increase. However, on releasing the pressure, the leakage decrease was proportional to the square of the pressure decrease. A second increase in pressure produced an increase that retraced the leakage encountered during the pressure decrease. It is believed that the original pressure increase cleaned the leakage passages. Further pressure cycling did not affect the maximum leakage. This suggests that whenever possible, leak testing should be done at the proposed operating pressure, in order that potential leaks may be formed and observed.

Characteristics of Absorbed or Surface Flow Leaks

The flow of gases and noncondensing vapors through fine capillaries and micropores cannot be dealt with by means of simple techniques analogous to those applicable to molecular and laminar flow. The narrow passages and large surface areas involved cause surface adsorption and surface flow to become important factors.

The adsorption may be physical, where only relatively weak van der Waals attractions are involved. However, the adsorption may also be regarded as chemical. In this case, the surface of the solid provides binding sites for the gas atoms and the electronic structure of the solid permits the formation of a chemisorption bond. The nature of the binding sites, the bonds between the gas atoms and the surface all influence the degree of surface migration of the atoms.

The flow along a fine capillary or micropore is assumed to consist of two mechanisms working simultaneously: (1) molecular flow along the bore of the capillary, whereby molecules are supposed to collide with the wall, reevaporate and collide with the wall again without intermolecular collisions; and (2) surface flow along the wall of the capillary, whereby molecules are adsorbed and diffuse along the surface of the wall. Both these mechanisms promote gas flow from regions of higher gas concentrations to regions of lower gas concentrations.

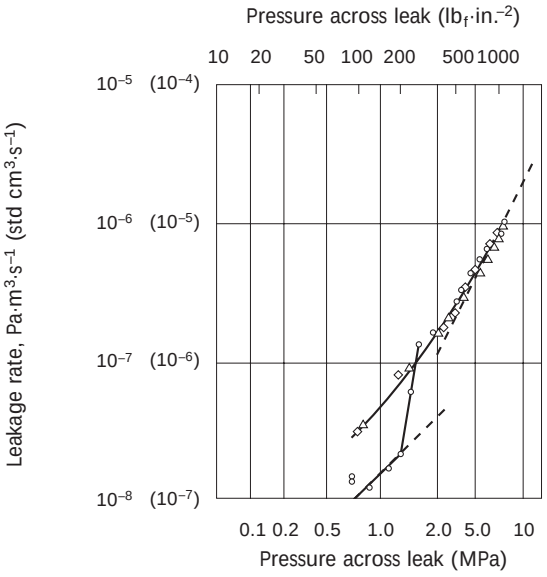
Factors Influencing Surface Flow of Gases

For a given set of conditions, the proportion of molecules that follow the mechanisms of adsorbed or surface flow leakage depends on a variety of factors, including (1) the sticking probability (the probability that a molecule sticking the surface will become adsorbed), (2) the length of time the molecule remains adsorbed (the mean surface lifetime of the gas molecules) and (3) the coefficient of surface diffusion of the gas molecules. These features are, in turn, influenced by other characteristics, such as the number of sites occupied by the adsorbed molecules or whether a complete monolayer is involved.

The nearer the properties of a gas approach those of a condensable vapor, the greater the proportion of surface flow. Therefore, a reduction of temperature or an increase of pressure may sometimes promote a total flow in excess of that predicted by the laminar molecule theory.

Although the final leakage rate achieved with a condensable gas may be higher than predicted from flow theory, there may be an initial delay of flow because of condensation of the tracer gas on the leak surfaces. This delay is important if a tracer probe technique is used for testing. For example, if butane, a readily condensable gas, is used in the tracer probe, some small leaks will be missed because of the delay caused by the adsorption. Two remedies can be suggested to counter this problem: use of a noncondensable gas and use of a detector probe with condensable gases. With use of a detector probe, the gas is continually in contact with the leak and equilibrium is established.

FIGURE 19. Leakage curves showing self-cleaning effects in leaks.



Legend

◇ = Pressure decrease
△ = Second pressure increase
○ = Initial pressure increase

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3

CHAPTER

Calibrated Reference Leaks[1]

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PART 1. Calibrated Reference Leaks

Terminology Applicable to Reference, Calibrated or Standard Leaks

Physical leaks suitable for checking leak detector performance and leak test sensitivity are a vital component of instrumentation for leak testing. The terms reference, calibrated and standard leaks have been used in the past to identify these physical leaks. To many people, the term calibration implies the existence of a universally accepted standard such as those at the National Institute of Standards and Technology.

The National Institute of Standards and Technology has performed calibration of helium leaks (capillary and permeation) over the range of 10^{-14} to 10^{-6} mol·s^{-1} (2.3×10^{-11} to 2.3×10^{-3} Pa·m^3·s^{-1}) on a routine basis. The uncertainties in leak rate vary from less than 1 percent at 10^{-6} mol·s^{-1} (2.3×10^{-3} Pa·m^3·s^{-1}) to as much as 5 percent at 10^{-14} mol·s^{-1} (2.3×10^{-11} Pa·m^3·s^{-1}). Additionally, the National Institute of Standards and Technology will calibrate leaks with other gases over this range on a special test basis. All of these calibrations are performed while the gas is exhausted into a vacuum. Leaks may also be calibrated by commercial companies that derive their measurement uncertainty from either of two techniques. The first is that they derive their measurements from leaks calibrated at the National Institute of Standards and Technology and perform calibrations using a comparison technique. The second technique uses secondary techniques that derive the leak rate through measurements of pressure, volume, temperature and time with instruments whose calibration can be traced to the National Institute of Standards and Technology. The appropriate type of calibration will depend on particular measurement requirements including the required accuracy, traceability or regulatory issues.

In some cases, accuracy in leakage measurement is not of prime importance. Rather, most practical situations require that some particular leakage value not be exceeded. It need only be established that no leakage in the tested system is greater than this allowable maximum leakage rate. This practical approach to leakage specification requires some arbitrary standard. However, if any doubt exists, one need only reduce the leakage of this arbitrary standard physical reference leak by a sufficient safety factor to ensure that test sensitivity meets the practical leakage requirement within some estimated confidence interval.

Classification of Common Types of Calibrated or Standard Physical Leaks

Calibrated physical leaks are designed to deliver gas at a known rate. The most common use of such leaks is in the measurement of sensitivity of leak detectors. However, calibrated leaks are also used to measure the speed of vacuum pumps and to calibrate pressure gages. A standard physical leak makes feasible the establishment of leakage rate requirements for specifications. It also provides a uniform reference standard for calibrating leak detectors at different locations where products are inspected. This ensures more uniform agreement of all tests.

Calibrated leaks may be divided into two distinct categories: (1) reservoir leaks that contain their own tracer gas supply and (2) nonreservoir leaks to which tracer gas is added during testing. Figure 1 shows a classification of physical leaks used for reference, calibration or standard leaks.

Accuracies of Reservoir Calibrated Leaks

The uncertainty in the leak rate of fixed reservoir leaks is due to a combination of calibration uncertainty, leak rate decay because of calibration, temperature effects and leak instability. Of these, uncertainty in the stability of the leak is hardest to quantify. Changes in the leak rate may occur in capillary leaks because of partial blockage of the capillary. Changes in the leak rate of glass permeation leaks may occur because of the development of microcracks in the glass. In general, these leaks are more stable than leaks without closed reservoirs, particularly for calibrated leaks with values less than 10^{-9} mol·s^{-1} (2.3×10^{-6} Pa·m^3·s^{-1}).

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Accuracies of Nonreservoir Calibrated Leaks

The nonreservoir type of leak provides only a hole or a series of holes and passages that permit gas to pass through at a known rate. The users of this type calibrated leak must provide gas at a known concentration, purity and pressure.

The uncertainty in the leak rate of nonreservoir leaks is due to a combination of calibration uncertainty, temperature effects, leak instability, pressurizing gas purity and uncertain measurements of gas pressures and temperatures. Most if not all nonreservoir leaks are physical leaks and are susceptible to plugging. Because of this it is very important that the input gas be free of particulates and hydrocarbons. In addition the output should be exposed to as little contamination as possible, especially when the leak is not pressurized. Nonreservoir type leaks are typically used for higher leak rates, greater than 10^{-9} mol·s^{-1} (2.3×10^{-6} Pa·m^3·s^{-1}), where the depletion rate of reservoir leaks becomes greater than 20 percent per year. The temperature coefficients of leaks can be measured to account for changes in the leak rate as a function of temperature.

Comparison of Standard Leaks with and without Tracer Gas Reservoirs

In proper leak testing practice, the sensitivity of leak detectors is checked frequently by calibrated leaks of reservoir types with internal gas supply. For system sensitivity checks, a calibrated leak without a reservoir is preferable because it closely imitates the behavior of an actual leak in the object or system under test. The calibrated leak without a reservoir is open to local atmospheric pressure; therefore, it requires no sensitivity correction for pressure, temperature and other environmental factors. In the tracer probe mode of leak detection, tracer gas is sprayed on the calibrated leak under the same conditions that exist when the leak detector is used to measure a leak in any system or enclosure under test.

In the case of a leak containing a reservoir, the measured sensitivity of the leak detector is independent of the test gas pressure and of the tracer gas contamination of ambient air surrounding the leak testing area. If the calibrated leak is to be used for the measurement of an absolute value, as in the case of the calibration of a pressure gage or measurement of the speed of a pump, a leak carrying its own gas supply is desirable.

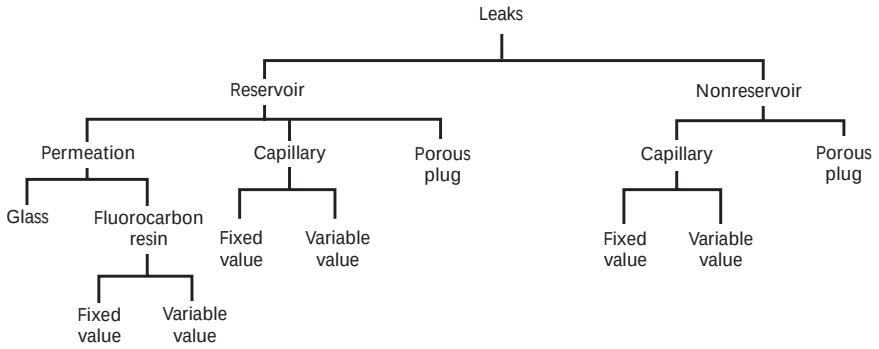
Basic Categories of Calibrated Gas Leaks

Generally, leaks may be grouped into either of two categories: (1) leaks that depend on the permeation of some materials by certain gases and (2) leaks in orifices that permit the flow of any gas when a pressure differential is exerted across the element.

Variation of the material composition, the membrane dimensions and the partial pressure differential of gas across the element permit the attainment of an almost infinite range of flow rates. The temperature coefficients of the permeation leak systems are appreciable. This provides an additional means of extending the flow range, particularly when the other parameters are fixed or limited.

Leaks that permeate through a fluorocarbon resin membrane are also available with properties similar to those of gas leaks. The second category of orifice leaks permits the attainment of a wide range of flow rates by modification of the

FIGURE 1. Categories of artificial physical leaks commonly spoken of as “reference,” “calibration” or “standard” leaks.



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element dimensions and the pressure differential across the element. Temperature is not as great a factor because the temperature coefficients are small with glass orifice standard leaks.

Properties Designed in Calibrated Gas Leaks

The range of possible flow rates of calibrated leaks is rather severely limited by practical considerations in the selection of parameters for the construction of leaks for quantitative standards. An ideal calibrated leak should have the following properties.

1. The leakage rate should be constant and should remain unaffected by ambient conditions.
2. The calibration should be accurate.
3. The physical size should be convenient.
4. The calibrated leak should not be too delicate or fragile.
5. The calibrated leak should have its own gas supply.

Temperature Coefficients of Calibrated Leaks

Unfortunately, those parameters useful in extending the possible range of flows are not conducive to constancy. The high temperature coefficients of the membrane leaks are particularly disturbing when the changes in ambient temperatures are frequent and there is no way of determining whether or not the equilibrium flow rate is reached at any one temperature. Even the relatively small temperature coefficients of orifice leaks are appreciable when the temperature varies over wide ranges.

The National Institute of Standards and Technology measures the temperature coefficients of leaks as a normal part of their calibration service over the range of 0 to 50 °C (32 to 122 °F). Some manufacturers of calibrated leaks may also be able to measure temperature coefficients. Normally manufacturers assume a linear temperature coefficient of 3 to 4 percent per 1 °C (2 °F) for glass helium permeation leaks. For the lowest uncertainties the temperature coefficients should be measured.

Size, Weight and Portability of Calibrated Gas Leaks

The convenience of the physical size is a property that would vary considerably, depending on the use to which the calibrated leaks is applied. In general, complete and convenient portability of standard leaks is desirable and is usually available in nonreservoir standard leaks. Portability is easily attainable with reservoir standard leaks with low leakage rates of the order of 2×10^{-7} Pa·m^3·s^{-1} (2×10^{-6} std cm^3·s^{-1}) or less.

During manufacture of calibrated leaks, additional effort and weight can extend the upper limit of flow by as much as a factor of 50 without allowing the depletion of the gas supply to cause a falloff in leakage rate greater than 10 percent per year. Greater increases of the upper limit call for nearly linear increases in volume of the leak gas reservoir and even greater increases in weight. These reduce the portability and ease of installation of standard leaks.

Limitations of Flow Rate Calibration of Standard Gas Leaks

The lower limit of flow rate that is practical for direction calibration is about 10^{-11} Pa·m^3·s^{-1} (10^{-10} std cm^3·s^{-1}). The degassing of the system becomes a problem as the size of the leak is decreased. In this range the changes of both true leakage and virtual leakage caused by pressure increase are nearly equal.

Two indirect techniques may be used, either separately or in combination, to calibrate with reasonable accuracy in the low ranges; both techniques have been experimentally justified. The calibration may be made by comparison with a standard of greater flow rate by means of a mass spectrometer. The actual rate is extrapolated (assuming linear response of the instrument). Alternatively, the leakage rate may be increased in a manner in which the response is predictable (i.e., the pressure response of membrane leaks is linear) and calibration made at the higher flow rate.

Limitations of Glass Membrane Standard Gas Leaks

Construction of all-glass membrane leaks that vary in flow range at ambient temperatures from 0 to 50 °C (32 to 122 °F) is rather simple. Larger flows require either higher pressures or modification of membrane parameters that tend to make them excessively fragile.

It is possible to combine a number of the large leak elements in parallel to obtain greater flow when necessary. Advantage has been taken of the relatively sturdy nature of glass tubing of very small cross section and correspondingly thin walls. Elements have been made using literally miles of such tubing in systems designed for use at relatively high temperatures to separate low concentrations of helium from natural gases. However, these elements do not seem suitable for use under high vacuum conditions.

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Limitations of All-Glass Orifice Standard Leaks

All-glass orifice leaks are more difficult to produce with flow rates smaller than 5×10^{-9} Pa·m^3·s^{-1} (5×10^{-8} std cm^3·s^{-1}) unless precautions are taken to maintain the pressure differential significantly positive in the downstream direction while the upstream pressure is made subatmospheric.

The upper limit in flow rate is determined mainly by the maximum acceptable physical size. Glass reservoirs become bulky when they are of adequate size to supply a leak of the order of 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}) without having the leakage rate fall off more than 10 percent per year.

Improving Calibrated Leakage Rate Stability by Increasing Envelope Pressure

Stability of leakage rate may be improved greatly without sacrificing compactness by enclosing the leak element in a metal envelope and filling the envelope to a significantly greater pressure. Membranes that leak 5×10^{-8} Pa·m^3·s^{-1} (5×10^{-7} std cm^3·s^{-1}) at a pressure differential of 100 kPa (1 atm) will raise their leakage 20× to a rate of 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}) when used with a partial pressure differential of 2 MPa (20 atm).

The leakage rate will fall off one twentieth as much as that of a membrane that will leak 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}) at atmospheric differential, with the same volume reservoir. Maximum envelope membrane leak pressures are limited by their nature to not more than 2.8 MPa (400 lb$_f$·in.$^{-2}$ gage). Orifice leaks have been used with maximum pressures in the envelope of 12 MPa (1700 lb$_f$·in.$^{-2}$).[1]

Basic Characteristics of Membrane Standard Leaks

Membrane standard leaks share several characteristics.

1. They are restricted to usable gases, even at elevated temperatures.
2. They have relatively high temperature coefficients.
3. They are relatively fragile when constructed with glass.
4. They normally have a response linear with respect to reservoir concentration.
5. They are almost impossible to plug.

Basic Characteristics of Orifice Standard Leaks

The orifice standard leaks share several characteristics.

1. They may be used with almost any gas under conditions sufficiently removed from liquidus conditions.
2. They have relatively low temperature coefficients.
3. They are relatively sturdy, being able to stand high pressure differentials, in excess of 10 MPa (100 atm).
4. They have pressure responses that vary from linear response for very small leaks, about 1×10^{-9} Pa·m^3·s^{-1} (1×10^{-8} std cm^3·s^{-1}), to direct proportion to the square of the pressure for very large leaks, about 5×10^{-4} Pa·m^3·s^{-1} (5×10^{-3} std cm^3·s^{-1}).
5. They are subject to plugging by solids or by condensation of vapors of materials close to liquidus conditions.

Precautions with Calibrated Gas Leaks

For maximum accuracy in the use of calibrated leaks, the following precautions should be taken.

1. Leakage rates should be defined as mass units per unit time. When volume units are used, they must be defined by specification of the temperature and pressure conditions under which they are to be measured.
2. The temperature at which the calibration is made and the temperature at which the calibrated leak is used should be specified. If they are not identical, the temperature coefficient should be used to correct the leakage rate. For best results the leak should be calibrated and used under constant temperature conditions.
3. A considerably higher than ambient temperature surrounding the element of the orifice leaks will tend to decrease the possibility of plugging by condensation of liquid.
4. If a leak is not equipped with an integral gas supply, care should be taken to use dry gas with orifices and to maintain a positive pressure differential across the element in the downstream direction if possible. Membrane leaks should be given adequate time to reach an equilibrium rate if the partial pressure differential of the tracer gas is changed.

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Design and Construction of Permeation Physical Leaks

Permeation leaks use the principle of gas diffusion through a thin wall. Tracer gas permeates from the high leak reservoir concentration through the wall to air or vacuum. Leakage is governed by the permeability of the thin membrane. The major advantage of permeation leaks is that they deliver extremely small quantities of gas. The commercially available helium leak standard range extends from 10^{-7} to 10^{-11} Pa·m^3·s^{-1} (10^{-6} to 10^{-10} std cm^3·s^{-1}). Because a long period of time is necessary to achieve permeation equilibrium, these leaks usually come with a self-contained gas supply. However, at small leakage rates, the leakage remains constant over a long period of time. The two disadvantages of calibrated permeation leaks are (1) that they can only be made for gases that permeate through membranes and (2) that their leakage temperature coefficient is large (three percent or more per degree kelvin).

Permeation Leak for Helium Tracer Gas

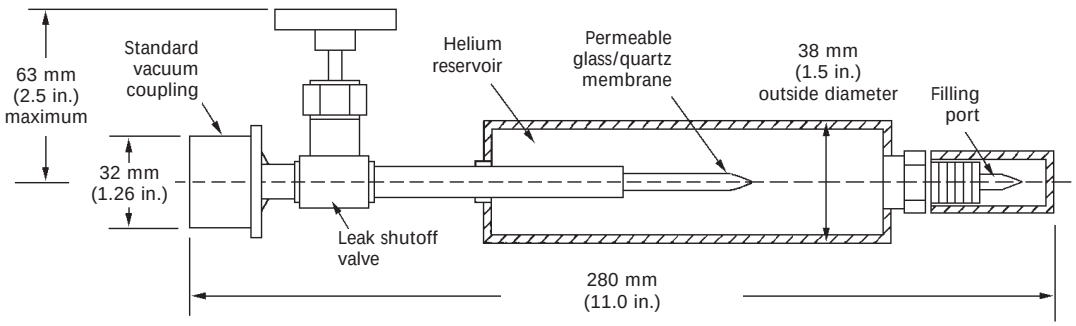
A common helium permeation leak is shown in Fig. 2. The helium permeation leak consists of a small helium filled metal or glass cylinder with an integral glass membrane at one end. Helium diffuses through this glass at a measurable rate. Each leak should be calibrated and labeled with the following information: (1) name of manufacturer, (2) model number, (3) type of leak (glass permeation, orifice etc.), (4) serial number, (5) composition of fill gas, (6) leak rate, (7) calibration temperature, (8) estimated uncertainty of leak rate, (9) date of calibration, (10) temperature coefficient and (11) reservoir pressure, date of fill and estimated depletion rate.[2]

The leak may contain two valves: a vacuum valve downstream of the leak element and a pressure (or reservoir valve). The reservoir valve is used for

FIGURE 2. Helium permeation leak with self-contained reservoir: (a) photograph of standard helium leak and cut away model; (b) schematic cross section.

(a)

(b)



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refilling of the leak reservoir. The vacuum valve is used for briefly shutting off the helium flow for purposes of zeroing a helium leak detector during the process of calibration. The vacuum valve should not be shut off for extended periods of time (greater than 10 min) or the stability of the leak may be affected severely.

Porous Plug Calibrated Leaks Providing Molecular Flow of Gas

Porous plug calibrated leaks are not commercially available but have frequently been cited in literature. They consist of a metal, ceramic or glass plug containing extremely fine pores. The major advantage of this type of calibrated leak is that molecular gas flow occurs through the plug. Therefore, the change of leakage flow resulting from a change of tracer gas can be calculated from the kinetic theory of gas flow. Porous plug leaks can be either reservoir or nonreservoir type, with the choice of materials cited above.

Design and Characteristics of Capillary Calibrated or Standard Physical Leaks

Another type of commercial calibrated leak is a single orifice in heat resistant glass or metal, encased in a stainless steel fixture. Tracer gas leaks through the orifice at the rated leakage, when the leak is placed under a specified gage pressure (relative to atmospheric pressure). Such capillary leaks are available in two types, fixed value leaks and variable value leaks, as next described.

Fixed Leakage Value Orifice Capillary Leaks

Capillary type calibrated leaks are made from constructed glass tubing or collapsed thin metal tubing. These orifice leaks can be produced from large sizes down to about 10^{-8} Pa·m^3·s^{-1} (10^{-7} std cm^3·s^{-1}). Although smaller leaks of this nature can be made, they become extremely difficult to handle because of leak clogging. Capillary leaks can be calibrated to deliver one or a variety of tracer gases. Some leaks are to be used with an independent tracer gas supply, i.e., they simply consist of a capillary leak attached to the system under test. In the tracer probe method of leak testing, tracer gas is simply sprayed over the capillary. Alternatively, a physical reference capillary leak can be made with a self-contained gas supply that can be permanently attached to the leak. Figure 3 shows a physical capillary orifice leak with its own tracer gas reservoir and a leak factor gage. The gage pressure may be changed to change the leakage rate at which tracer gas flows out of the physical reference leak.

Variable Value Orifice Physical Reference Halogen Leaks

The variable value physical halogen vapor leak shown in Fig. 4 is available for different ranges, such as 10^{-5}, 10^{-6}, 10^{-7} and 10^{-8} Pa·m^3·s^{-1} (10^{-4}, 10^{-5}, 10^{-6} and 10^{-7} std cm^3·s^{-1}). A schematic flow

FIGURE 3. Reservoir variable rate physical orifice leak standard (top) and fluorocarbon resin permeation leak standard (bottom) for calibration of detector probe instruments.

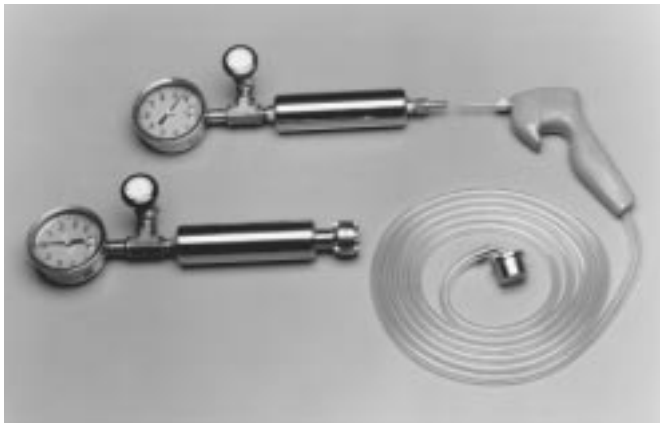

FIGURE 4. Variable leak rate halogen refrigerant leak standard with physical (capillary) leak element: (a) photograph of leak standard with internal reservoir of refrigerant gas and another of refrigerant liquid and (b) schematic flow diagram.

(a)

(b)

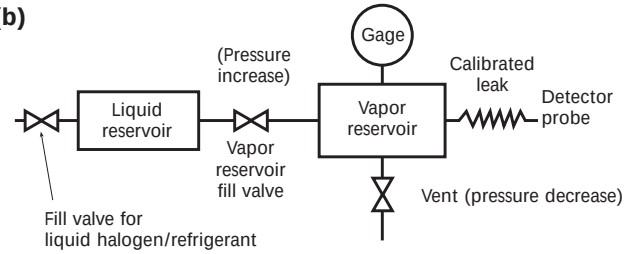

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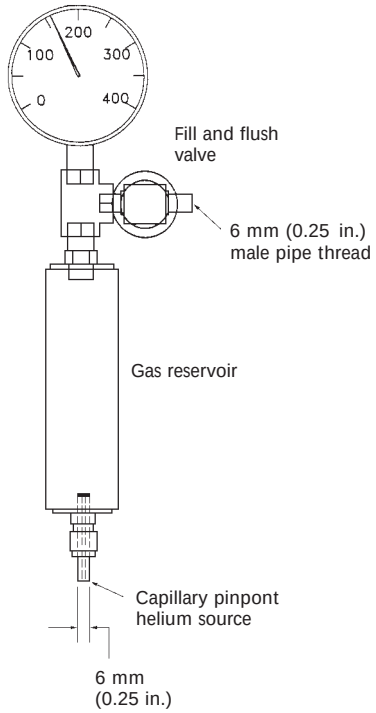
diagram of its system is shown in Fig. 4b. This leak contains a reservoir of liquid Refrigerant-134a halogenated hydrocarbon tracer to be valved into a ballast tank in gaseous form. Also connected to the ballast tank is a glass capillary tube and pressure gage. The rate of gas leakage through the calibrated leak depends on the pressure in the ballast tank. Laminar gas flow occurs through the leak. This permits the pressure gage to be marked in leakage units, where leakage is proportional to the ratio of the difference between the squares of the absolute pressures. The halogen leak standard is commonly used with heated anode halogen leak detectors. It is an excellent leak standard to use with probe instruments because the probe may be passed directly across the leak exit. The calibration then approximates detector probe operating conditions.

Variable Leak Rate Helium Reference Leaks

Variable reference leaks have been designed to leak helium for use with helium mass spectrometer leak detectors equipped with a detector probe. The leak arrangement is shown in Figs. 3 and 5. The leak standard in Fig. 5 uses either a capillary or fluorocarbon resin permeation membrane as the gas flow restriction (the time response for a glass membrane at room temperature would be much too slow). The leak is designed to yield a point source of helium to simulate a pin hole leak. The point source of leakage may be used to calibrate the detector probe of a helium mass spectrometer leak detector. The helium detector probe calibrator may also be used as a training tool to train operators on the distance and speed a probe must be from a certain size leak to detect the leak. The major disadvantage of the helium detector probe calibrator is that it requires an external tank of helium for refilling, unlike refrigerant calibrators that can store extra refrigerant in an on-board liquid tank (Fig. 4).

Other types of variable value reference leaks are controlled by elegant needle valve or crushed tubing whose conductance is changed by flexing. Although the conductance of these leaks can be made quite repeatable, they should not be considered calibrated leaks because of a complete lack of standardization of leakage rates in these artificial orifice types of physical leaks.

FIGURE 5. Variable rate helium leak standard (capillary style sniffer).



Sources of Inaccuracy of Leakage Measurements with Standard Leaks

The inaccuracy of leak detector measurements made with physical standard leaks can be caused by factors such as: (1) inaccuracy in calibrating the leak, (2) nonlinearity of the leak detection instrument, (3) variation in pressure differential applied across the leak, (4) impurity of gas applied to the leak and (5) variation in the amount of gas reaching the detector.

Accuracies of Calibrations of Commercially Available Physical Reference Leaks

Beginning in 1987, the National Institute of Standards and Technology established a leak calibration program that calibrated leaks over the range of 10^{-11} to 10^{-3} Pa·m^3·s^{-1} (10^{-10} to 10^{-2} std cm^3·s^{-1}).

In a 1980 study, tests of standard leaks from various manufacturers have shown that their accuracies could differ by more than ±50 percent of a mean value.[1] This is shown by the experimental plot of Fig. 6, which shows the calibrated leakage reading compared to the response of a linear mass spectrometer. The straight line drawn in the graph is the least mean square value of leakage as a function of spectrometer response. This line does not imply the correct value, but the general pattern around which the values of the leaks congregate.

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Even the leaks made by any single manufacturer vary by about 10 percent. This is usually the guarantee that is presented on purchase of the permeation calibrated leak. Leaks of a variable type, such as that shown in Fig. 4, are claimed to be accurate only to ±20 percent.

Beginning in 1987, many manufacturers of leaks began deriving their measurements directly from leaks calibrated by the National Institute of Standards and Technology. The existence of national standards in leak measurement should improve the relative agreement between the manufacturers of leaks and may also reduce the uncertainties that manufacturers provide for calibrated leaks.

Errors in Response of Commercial Electronic Leak Detectors

Most commercial leak detectors display the response to a detected leak as a current reading on a sensitive microammeter. It is usually assumed by the operator that current reading twice the magnitude of a previously observable one represents a leak of twice the size. This assumption of linearity in response is not necessarily correct; nonlinearity may result from the structure of the pumping system, the background usually associated with the leak testing practice, the electronic circuitry associated with the detection system and the mode of gas flow through the leak.

A typical leak detector response error curve is shown in Fig. 7. The instrument response is not linear with leakage. This error is added to the error that occurs because of the difference in leak calibration. Because of this lack of linearity, the farther apart the two leaks are in nominal value, the greater the error in the calibration. Because such deviations exist it is recommended that, when the leakage measurement is done to a specified high tolerance, a calibrated leak to the exact specified value be used as a standard.

Effect of Barometric Pressure on Leakage Measurements

Leakage depends on the pressure differential acting across the leak. When leak detection is done by a tracer probe, the pressure differential is usually 100 kPa (1 atm). The gas is sprayed over the suspected area without aid of additional pressure. Should leak detection be performed at high altitudes, the atmospheric pressure is less than 100 kPa (1 std atm). The magnitude of this reduction is as much as 20 percent in places such as Boulder, Colorado. If the leaks that are being located are of a laminar nature, the laminar flow through

FIGURE 6. Comparison of leakage values for leaks supplied by various vendors, measured by linear mass spectrometer. Resulting ion current depends on mass spectrometer configuration.

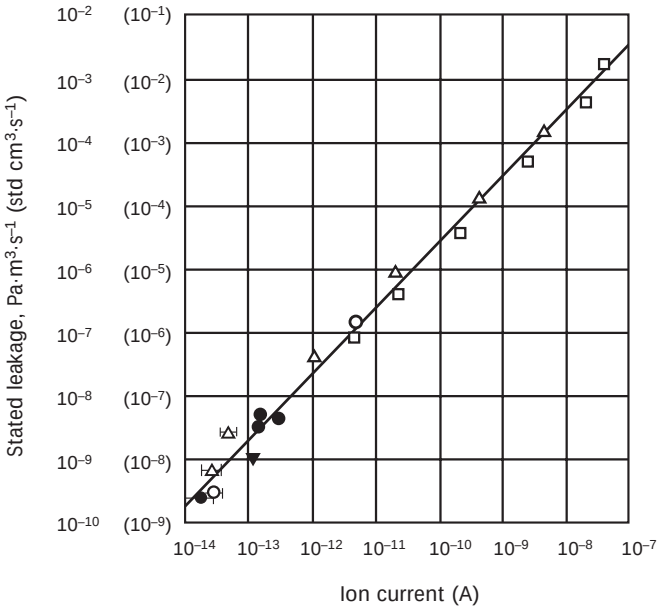
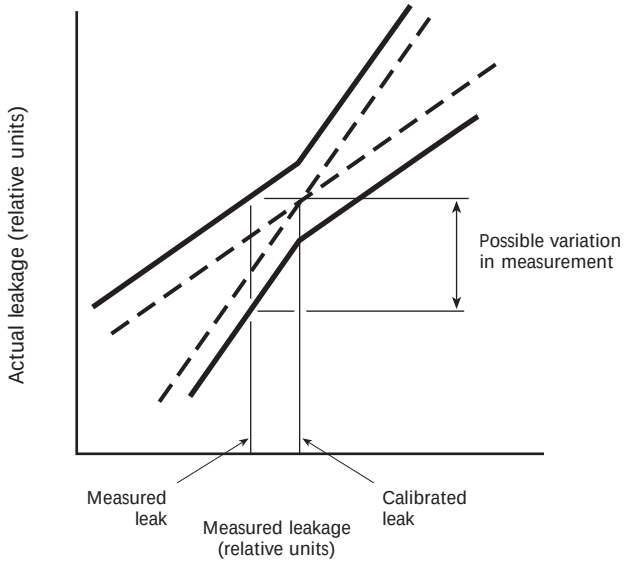


FIGURE 7. Typical range of error possible in actual leakage measurements with a leak detector. Variations with magnitude of leakage increase the difficulties of correlating measured leakage rates with standard reference leaks.



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the leak is proportional to the square of the pressure differential. The values obtained for leakage readings at the altitude of Boulder, Colorado, are 40 percent less than those obtained with a 100 kPa (1 std atm) pressure during use. Therefore, a leakage rate measured to atmosphere in Boulder, Colorado will be only 60 percent as large as with the same leak measured at Cape Kennedy on the seashore of Florida.

Certain calibrated leaks contain their own gas supply, whereas others have the tracer gas sprayed onto the entry orifice of the leak at 100 kPa (1 atm) pressure during use. Calibrated leaks with a self contained gas supply always deliver to the detector a fixed amount of gas that can be used to measure the sensitivity of the leak detector. On the other hand, leaks where gas is added during use produce the calibrated amount of leakage only when a 100 kPa (1 atm) pressure differential is supplied. These nonreservoir physical reference leaks therefore deliver less than the calibrated amount of leakage when used at high altitudes where the atmospheric pressure is lower. However, at these altitudes, the pressure of the tracer gas across the leak is lower. In such cases, a physical reference leak without its own gas supply describes more accurately the sensitivity of the leakage test. It is this test sensitivity that is important in practical leak testing.

Effect of Tracer Gas Purity on Accuracy of Leakage Measurements

Another source of inaccuracy is the impurity of the tracer gas used for leakage measurement. If a tracer probe technique of leak location is used, the gas is sprayed over the suspected area in the environmental atmosphere. In such a case, it is quite possible that the tracer gas is diluted with air as it approaches the leak. Therefore, the response of the leak detector operating on the internal vacuum of the test system will be reduced by the amount air impurity entering the detector with the tracer gas. In this case, a calibrated leak with a self-contained gas supply is undesirable because it would not reproduce the leakage measurement technique. In other words, the gas should be sprayed onto the calibrated leak in the same manner as onto the tested leak. The gas in a self-contained calibrated leak would be purer than the gas encountered by simple spraying from a tracer probe.

Effect of Position of Calibrated Leak on Test System

Tracer gas may be absorbed on test system surfaces as it travels to the detector. This would decrease the response of the leak detector. Therefore, calibrated leaks should be positioned on the system as near as possible to suspected leak sites to improve accuracy. Alternatively, they may be positioned as far away from the detector as possible to show minimum sensitivity. Both of these positions are conservative choices that ensure that leakage from test object discontinuities will not be underrated.

Specifying Maximum Allowable Leakage Rate

Because of the variations discussed here, the accuracy of any leakage measurement probably varies from half to twice the actual value. This implies that, if a leak is measured as 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}), the actual value of this leak is between 2×10^{-6} and 0.5×10^{-6} Pa·m^3·s^{-1} (2×10^{-5} and 0.5×10^{-5} std cm^3·s^{-1}). Therefore, if the maximum allowable leakage rate of a particular system is 2×10^{-6} Pa·m^3·s^{-1} (2×10^{-5} std cm^3·s^{-1}), the specification may be written with a leakage tolerance of 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}), knowing that the accuracy of the leakage measurement is a factor of two. There is reasonable assurance that if the measured leakage is not higher than that stated on the specification, 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}), the actual system leakage will be no greater than the allowable rate, 2×10^{-6} Pa·m^3·s^{-1} (2×10^{-5} std cm^3·s^{-1}). This technique of specifying leakage is much more sensible than specifying a slightly higher leakage value, such as 2×10^{-6} Pa·m^3·s^{-1} (2×10^{-5} std cm^3·s^{-1}), and thereby requiring an unreasonably high accuracy (such as ±10 percent) during leak testing.

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PART 2. Operation of Standard (Calibrated) Halogen Leaks

Functions of Known Leakage Standards

The halogen gas leak detector (known also as the alkali ion diode halogen leak detector) is a transfer agent or compactor. Leak testing with halogen tracer gas requires use of a known reference halogen leak to calibrate the leak testing operation properly. The halogen leak detector is adjusted to produce an alarm or meter indication of the panel indicator when exposed to a known leakage rate. The detector is then used to compare unknown leakage rates to the specific known leakage rate of a calibrated reference leak.

The maximum acceptable leakage rate, however, must first be determined, either by the user or from specifications that the user must meet. The type and range of leak standard then may be selected, but only after this has been accomplished. Three types of halogen leak standards are (1) the calibrated standard leak (no gas), (2) the leak capsule (single gas reservoir) and (3) the halogen leak standard (reserve gas supply).

FIGURE 8. Calibrator for halogen leak standards with small bore capillary tube orifices for leaks from 3×10^{-5} to 3×10^{-8} Pa·m^3·s^{-1} (3×10^{-4} to 3×10^{-7} std cm^3·s^{-1}) or with larger bore capillary tube orifices for leaks from 3×10^{-4} to 3×10^{-7} Pa·m^3·s^{-1} (3×10^{-3} to 3×10^{-6} std cm^3·s^{-1}). Movement of a colored liquid within the calibrated capillary tube over a specific period of time permits calculation of the rate of leakage from the standard leak, when the calibrator is attached to the standard through a vent valve.

Halogen Leak Calibrator without Reservoir

Figure 8 shows a leak calibrator that has no reservoir for halogen tracer gas. It contains a single orifice in heat resistant glass. When a reservoir of refrigerant-134a is attached, the pressure of the refrigerant-134a gas is 165 kPa gage (24 lb$_f$·in.$^{-2}$ gage) and that gas will leak through its orifice at a fixed rate.

Calibrated Halogen Leak with Gas Reservoir

The calibrated halogen leak of Fig. 3 has its own refrigerant-134a reservoir plus a leak factor gage. The gage reads in multiplying factors, used when the pressure is changed to vary the leakage rate. The gage is set at a factor of 1 at the factory (165 kPa or 24 lb$_f$·in.$^{-2}$ gage). These leak capsules, used when a precise leakage rate is required, are frequently mounted in the halogen leak detector control unit.

Adjustable Halogen Leak Standards with Ballast Tank

The halogen leak standard shown in Fig. 4a contains a reservoir of liquid refrigerant-134a, which is valved in gaseous form into a ballast tank. Connected to the ballast tank is a glass capillary tube and pressure gage. The amount of leakage is dependent on the amount of refrigerant-134a tracer gas pressure in the ballast tank. Pressure is indicated by a Bourdon gage and controlled by two valves (Fig. 4b).

Applications of Calibrated Halogen Leaks and Capsules

Predetermined standard halogen leaks are of great advantage to quality control engineers in refrigeration, air conditioning and space vehicle

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manufacturing, where critical checks of lines, valves and hydraulic systems are of the utmost importance. They afford great accuracy wherever halogen leak detectors are used and where a leak of one specific value is required.

The adjustable halogen leak standard in Fig. 4 provides the same advantages as the calibrated leaks and capsule but has the additional advantage of being adjustable to the full scale rating. Thus, they can be used more easily for quantitative measurements of actual leaks and of background contamination. Leak standards also enable the establishment of leakage rate specifications and provide uniform standards for calibrating leak detectors at each location of product inspection.

Halogen Leak Standards to Prolong Life of Alkali Ion Diode Sensing Element

All three leak standards can be used to extend the useful life of the alkali ion diode sensing elements in heated anode halogen leak detectors. Users frequently replace the detector's sensitive element long before the end of its useful life. A sensing element can be used until it no longer responds to the desired setting of the leak standard. Additionally, any leak standard permits use of the lowest possible anode heater current to provide adequate leak detector sensitivity. This practice increases element life and results in reduced maintenance and lower replacement costs.

Accuracy of Adjustable Halogen Leak Standards

Typical accuracy of the adjustable halogen leak standard of Fig. 4 is about ±20 percent of scale setting on the upper two thirds of the scale and ±30 percent of scale setting on the lower one third of the scale.

Description of Adjustable Halogen Leak Standard

The leak standard of Fig. 4 is a simple, accurate instrument that expels a halogen compound gas, refrigerant-134a through a glass capillary marked probe to the atmosphere at a known rate. This known rate is adjustable when using certain halogen leak standards. The leak standard is intended primarily for use with halogen sensitive leak detectors. The leakage rate for each unit is marked on the scale plate. Leakage rates are customarily labeled in units of standard cubic centimeter per second (std $cm^3 \cdot s^{-1}$) and also in ounce per year ($oz \cdot yr^{-1}$) by the manufacturer of these standard leaks. The SI units are mole per second ($mol \cdot s^{-1}$) and pascal cubic meter per second ($Pa \cdot m^3 \cdot s^{-1}$).

Components of Adjustable Halogen Leak Standard

The adjustable halogen leak standard is a compact instrument consisting of the following seven functional components (see Fig. 4):

1. direct reading leakage rate indicator (calibrated in ounces of refrigerant-134a per year);
2. probe fitting in the center of which is a glass leak capillary (a different capillary for each leakage rate);
3. leakage increase valve and control knob;
4. leakage decrease valve and control knob;
5. vent (with protective cap) for exhausting refrigerant-134a gas;
6. tank for holding liquid refrigerant-134a (the tank contains some refrigerant-134a when shipped from the factory); and
7. a reservoir for holding refrigerant-134a gas at a pressure corresponding to the desired leakage rate.

Principles of Operation of Adjustable Halogen Leak Standard

The adjustable halogen leak standard (Fig. 5) operates as discussed below. The filler tank provides a supply of refrigerant-134a liquid under its own partial pressure. The increase valve controls the amount of refrigerant gas fed from the filler tank to the ballast tank, the leakage rate meter and the leak capillary. The pressure in the system is maintained by the ballast tank. With the increase and decrease valves closed, the system is practically in a static state, except for the minute amount of refrigerant gas that escapes through the leak capillary. The decrease valve provides a means of decreasing the pressure built up in the system. With the decrease valve opened, refrigerant gas is allowed to escape through the vent opening on the front of the leak standard. The rate of refrigerant gas escaping through the leak capillary is a function of the pressure in the system and is indicated on the leakage rate meter.

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Preparation for Operation of Adjustable Halogen Leak Standard

The following procedure is used with adjustable halogen leak standards.

1. Remove the protective caps from the leak capillary in the probe fitting and from the vent.
2. To increase the leakage rate, turn increase valve knob counterclockwise slowly until the instrument pointer starts to move upscale. As the pointer approaches the desired leakage rate, gradually close the increase valve so the pointer will stop at the desired leakage rate. If the instrument pointer continues to go upscale, this indicates that the increase valve is not firmly closed. Always make sure the increase valve is closed firmly. (Avoid running the instrument pointer off scale. This can subject the instrument to as much as 500 percent over pressure. Although the unit can withstand the overload, repeated abuse may damage it.)
3. To decrease the leakage rate, turn the decrease valve knob counterclockwise slowly until the instrument pointer starts to move downscale. As the pointer approaches the desired leakage rate, gradually close the decrease valve so that the pointer will stop at the desired leakage rate. If the instrument pointer continues to go downscale, indication is that the decrease valve is not firmly closed. Make sure the decrease valve is closed firmly.
4. After increasing or decreasing the leakage rate, be sure both valves are closed by turning knobs clockwise.
5. After increasing or decreasing the leakage rate and noting that the valves are firmly closed, wait about 60 s for the leakage rate to stabilize before calibrating the leak detector. When the leakage rate is being decreased, refrigerant-134a gas is allowed to escape through the vent to atmosphere. During this operation it is best to remove the leak standard from the test area to avoid building up a background of halogen vapor at the test site. If this is not possible, attach the vent tubing to the vent and discharge the gas from the test area through a window or other vent.
6. The leak standard is now ready for use.

Applications of Adjustable Halogen Leak Standard

The adjustable halogen leak standard of Fig. 4 may be used in several ways.

1. To check the operation and sensitivity of the halogen leak detector. The probe of the detector to be checked is moved past the probe fitting of the leak standard, which is set at the maximum leakage rate allowable for any single leak on the item being leak tested. If an adequate signal is obtained, the leak detector has sufficient sensitivity (or more) to detect this rate of leakage.
2. To determine size of leaks. If the leak standard is set so that the leak detector gives the same signal for the leak standard as for the leak, the leak standard then indicates the size of the leak, only if 100 percent pure refrigerant-134a is in the test system.
3. To extend the useful life of the sensing element of the halogen leak detector. Users frequently replace the detector's sensing element long before the end of its useful life. A sensing element can be used until it no longer responds to the desired standard leak setting. Additionally, the leak standard permits use of the lowest possible heater current to provide adequate leak detector sensitivity. These practices increase element life and result in reduced maintenance and lower replacement costs.
4. To simplify establishment of leakage rate specifications. The leak standard makes feasible the establishment of leakage rate specifications and provides a uniform standard for calibrating leak detectors at each location of product inspection.
5. To improve product quality. By calibrating leak detectors with the leak standard, it becomes possible to locate and repair all significant leaks. This ensures that products are manufactured in accordance with leakage specifications.

Precautions for Adjustable Halogen Leak Standard

The following precautions should be applied when using the adjustable halogen leak of Fig. 4. Never allow any grease or liquid to enter the leak capillary, as it may plug the leak or alter its leakage rate. When the leak standard is not in use, it is recommended that the instrument pointer be set up scale and that the protective caps be placed over the vent and leak capillary. This must be done to prevent plugging of the capillary.

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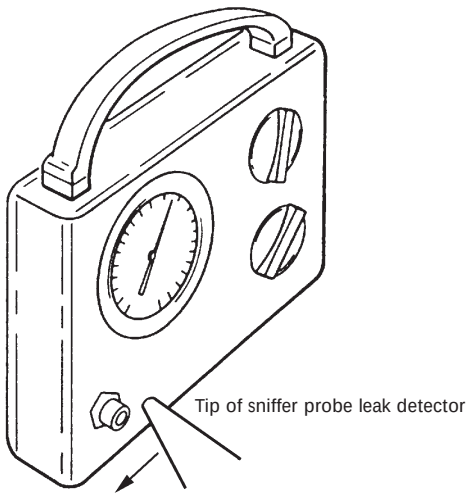
Operational Procedure When Pressurized System Contains 100 Percent Refrigerant

With the leak standard prepared for use, proceed as follows.

1. Turn on the leak detector and let it warm up for the time prescribed in the applicable leak detector instruction book. Set the leak detector in the same mode of operation as that to be used during leakage testing.
2. Place the probe squarely against the probe fitting on the leak standard (see Fig. 4) and observe the indicator reading. Remove the leak detector probe tip from the leak standard probe fitting. When the leak detector reading has settled to a stationary indication, pass the tip of the leak detector probe past the probe fitting on the leak standard at a rate of about 25 mm·s^{-1} (1 in.·s^{-1}). The tip of the probe should just graze the front circular edge of the probe fitting and pass across the center of the probe fitting as shown in Figs. 4 and 9.
3. Repeat the procedure of step 2 above, reducing or increasing the sensitivity setting of the leak detector each time, until the leak detector signal is adequate for the specified leakage rate. The results of this test will indicate the allowed probing speed and the safety factor required and provide the operator with a feeling for the difference in indications between a 100 percent tracer gas probe intake and the signal obtained during normal probing procedure.

FIGURE 9. Technique for checking leak testing sensitivity with sniffer probe tip moving past the orifice of an adjustable leak standard.



Interpretation of Unknown Leakage Rate from Comparable Standard Leak

A leak that gives the same leak signal as the standard is the same size as that indicated by the leak standard. A larger or smaller signal indicates a larger or smaller leak, respectively. If it is desired to determine the size of any leak that is located, adjust the leak standard in small leakage rate increments (waiting about 60 s after each change) until the signal caused by the leak standard is the same as that caused by the leak. The leak standard then indicates the size of the leak in question, in terms of its leakage rate.

Operational Procedure When Pressurized System Contains Less than 100 Percent Refrigerant

Halogen leak standards can also be used to calibrate a leak detector when the system being checked contains less than 100 percent refrigerant-134a. For applications using mixed gases in pressurized components, the leak standard may be used to calibrate a leak detector. However, a leak from the vessel (such as a tank, pipe or steam condenser) that produces the same leak signal as does the leak standard will have a total leakage rate that is approximately inversely proportional to the percentage of refrigerant-134a tracer gas in the enclosure. For example, suppose that, with 10 percent refrigerant-134a in the vessel, the leak standard indication is 30 g per annum (1 oz·yr^{-1}). The total leakage rate is then $100/10 \times 30 = 300$ g·yr^{-1} (10 oz·yr^{-1}).

Halogen leak standards are also used to calibrate a leak detector when test systems contain a halogen tracer gas other than refrigerant-134a, such as refrigerant-22, refrigerant-114 or refrigerant-11. The leakage rates for these other tracer gases may be read directly in standard cubic centimeter per second. Leakage rates in ounce per year can be obtained by multiplying the readings in standard cubic centimeter per second by 5.5×10^4. Readings in pascal cubic meter per second can be obtained by dividing the reading in standard cubic centimeter per second by a factor of 10.

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Measuring Atmospheric Contamination with Adjustable Halogen Leak Standard

To measure the amount of atmospheric contamination with a heated anode halogen vapor leak detector, the equipment required includes an adjustable halogen leak standard, a halogen leak detector and a pure air supply. The procedure recommended by the leak detector manufacturer is as follows.

1. In contaminated test areas, with the leak detector operating at an air flow of 4 $cm^3 \cdot s^{-1}$ (0.5 $ft^3 \cdot h^{-1}$), allow the leak detector to breathe pure air for about 1 min, then allow the leak detector to breathe air from the contaminated area. If the leak detector gives a signal, the area is contaminated. Note the magnitude of the leak detector signal. Do not adjust the sensitivity setting of the leak detector between this measurement and that which follows.
2. Move the leak detector and leak standard to an area where there is no atmospheric contamination. Adjust the leak standard so that when the leak detector sniffs the reference leak, the leak signal is the same as when the leak detector sniffed air in the contaminated area. Note the leakage rate shown on the dial of the leak standard. This is a measure of the level of atmospheric contamination with halogen vapors in the original contaminated area measured in step 1.
3. If it is desired to determine (approximately) the degree of contamination of the contaminated area in parts per million ($\mu L \cdot L^{-1}$) of halogen gas, the reading of the leak standard from Step 2 in ounces per year can be multiplied by 16. For example, if the indication on the reference leak is 1.5 $oz \cdot yr^{-1}$ the contamination level is $1.5 \times 16 = 24\ \mu L \cdot L^{-1}$. (For a leakage in grams per year, divide the number by 1.8 to arrive at the number of parts per million.)
4. When using a leak detector that has its own integral pure air supply, an indication of the degree of atmospheric contamination with halogens can be obtained by holding a finger over the probe tip for 30 s and then switching the leak detector to manual zero with the other hand. If the leak detector reading is then greater than the indication received with leakage of the rejection level, the halogen contamination of the air in the test area is excessive.

Use of Calibrator for Halogen Leak Standard

The calibrator is an accessory designed to check the accuracy of calibrated leaks, leak capsules and halogen leak standards. Three models of the calibrator differ in the bore size of the calibrated glass capillary tube, which is the major component of the calibrator. A small bore capillary tube is used for leaks from 3×10^{-5} to 3×10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (3×10^{-4} to 3×10^{-7} std $cm^3 \cdot s^{-1}$). A larger bore capillary tube is used for leaks from 3×10^{-4} to 3×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (3×10^{-3} to 3×10^{-6} std $cm^3 \cdot s^{-1}$). Another model is supplied with both capillary tubes.

The major component of the calibrator is a calibrated glass capillary tube. Accessories are provided allowing the capillary tube to be connected to and supported by the halogen standard leak under test. To perform a calibration, the calibrator is attached to the standard leak through a vent valve. A colored indicating liquid is inserted into the open end of the capillary tube and the vent valve is opened. The indicating liquid is drawn into the capillary tube by applying a slight suction to the plastic suction tube connected to the vent valve. The vent valve is then closed, retaining all the escaping halogen vapor in the capillary tube of the calibrator. By noting the amount of movement of the indicating liquid in the capillary tube for a specific period of time, the magnitude of the leak from the standard can be calculated and compared with the reading of the leakage rate gage on the standard.

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PART 3. Operation of Standard (Calibrated) Helium Leaks

Functions of Calibrated Helium Reference Standard Leaks

Calibrated helium standard leaks are essential when helium is used as the tracer gas in leak testing for quantitative leakage rate measurements. Calibration serves to determine the user's ability to detect leakage and to perform quantitative measurement of leakage rates. It is imperative that the entire leak testing system be calibrated. It is not sufficient merely to calibrate the leak testing instrument. In the case of a detector probe test, for example, the detector probe must be included in the leak testing system during the calibration operations and used in the normal manner as during testing.

The difficulty of repeating exact detector probe techniques virtually precludes the detector probe method as a way of measuring leakage rates quantitatively, although detector probe tests are good qualitative tools. In vacuum pumped systems, the system leak and the artificial reference leak must be located very close to each other for the quantitative measurement to be valid.

Rating of Calibrated Helium Leaks

Calibrated helium leaks are usually measured in units of pascal cubic meter per second ($Pa \cdot m^3 \cdot s^{-1}$) or standard cubic centimeter per second (std $cm^3 \cdot s^{-1}$). It is expected that standards in the future will be calibrated in mass flow units of mole per second. However, when discussing the flow of a compressible fluid, it is necessary to state not only the volumetric flow rate (V/t) but also pressure P and temperature T. Note that the units of leakage are identical to the units of throughput (the product PS of pressure P and pumping speed S). Both leakage and throughput describe the mass flow rate or, actually, the number of gas molecules escaping per unit time if the temperature is given.

Characteristics of Gaseous Flow Involved in Leakage Calibrations

At least three additional variables must be considered when using standard calibrated leaks: (1) the nature of flow (viscous, transitional or molecular) of gas passing through the leak, (2) the specific tracer gas or gas mixture flowing through the leak and (3) the pressure differential acting across the leak.

In the viscous flow range, the mass flow rate is inversely proportional to the gas viscosity and directly proportional to the difference in the squares of the upstream and downstream pressures. In the molecular flow range, the mass flow rate is inversely proportional to the square root of the mass of the gas molecule and directly proportional to the difference in partial pressure. Leakage at rates of 1×10^{-5} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-4} std $cm^3 \cdot s^{-1}$) or greater will be most likely to be viscous flow. Leakage at rates between 10^{-5} and 10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (between 10^{-4} and 10^{-7} std $cm^3 \cdot s^{-1}$) will usually be transitional in nature, exhibiting characteristics of both molecular and viscous flow. Leakage at rates in the range of 10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-7} std $cm^3 \cdot s^{-1}$) or smaller will probably be molecular.

Membrane or Diffusion Calibrated Reference Helium Leaks

Two types of helium standard leaks or calibrators are in general use, namely the membrane type and the capillary type. The design of a membrane or diffusion type standard leak is shown in Fig. 2. This standard leak has a reservoir filled with helium surrounding a sealed glass tube through which helium diffuses at a very low rate, usually from 10^{-8} to 10^{-10} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-7} to 10^{-9} std $cm^3 \cdot s^{-1}$). The standard calibrated helium leak shown in Fig. 2 is fitted with a shutoff valve that uses a metallic seal rather than an elastomeric seal. This avoids spurious changes in leakage rate due to helium hangup. The reservoir is filled with 100 percent helium at 100 kPa (1 atm or 14.7 $lb_f \cdot in.^{-2}$ absolute) of pressure. During calibration of this leak, the pressure differential feeding helium tracer gas into an evacuated test system is therefore from 100 kPa to 0 kPa (14.7 to 0 $lb_f \cdot in.^{-2}$). Because the partial pressure of helium in air is only about 0.5 Pa (4 mtorr), the glass membrane calibrator of Fig. 2 continues to leak helium even when it is not under vacuum.

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Capillary Standard Calibrated Helium Leaks

The capillary helium leak consists merely of flattened tubing or glass capillary enclosed in a protective metallic sheath. They are generally calibrated with one end at vacuum and the other end at atmospheric pressure. Capillary leak standards are available with fixed leakage rates varying from 10^{-3} to 10^{-6} Pa·m^3·s^{-1} (10^{-2} to 10^{-5} std cm^3·s^{-1}). These capillary leaks may have self-contained helium reservoirs. They are more susceptible to drastic changes in leakage rate caused by clogging or by foreign agents such as dust or condensation than are membrane leaks.

Computation of Molecular Flow Leakage Rates for Other Gases from Helium Leakage Rates

Although helium is commonly used as the tracer gas for mass spectrometer leak testing, it is usually necessary to determine the rate at which air would leak through a similar discontinuity. In the molecular flow range (and only in the molecular flow range), the air leakage rate will be about 35 percent of the helium leakage rate through the same pressure differential. When molecular flow occurs, the flow rate for one gas can be compared to the flow rate of any other gas by use of Eq. 1:

$$(1) \quad Q_2 = \sqrt{\frac{M_1}{M_2}}\, Q_1$$

where Q_1 is flow rate for gas 1 (any units of leakage rate), Q_2 is flow rate for gas 2 (same units of leakage rate) and M_1 is molecular mass for gas 1 (relative atomic mass).

Computation of Viscous Flow Leakage Rates for Other Gases from Helium Leakage Rates

If the flow rate has been identified as corresponding to viscous flow for one gas, the viscous flow rate for any other gas can be determined by use of Eq. 2:

$$(2) \quad Q_2 = \frac{n_1}{n_2}\, Q_1$$

where Q_1 is flow rate for gas 1 (any units of leakage rate), Q_2 is flow rate for gas 2 (same units of leakage rate), n_1 is viscosity of gas 1 (pascal second) and n_2 is viscosity of gas 2 (pascal second).

Table 1 lists the viscosities and molecular masses of helium, argon and neon inert tracer gases, air, nitrogen, ammonia and other gases and vapors commonly encountered in leak testing. The values of the viscosities and molecular masses from this table can be used in Eqs. 1 and 2 to compute leakage

TABLE 1. Physical properties of certain gases and vapors.

Gas	Chemical Symbols	Relative Molecular Mass (M_r)	Gas Constant (J·kg^{-1}·K^{-1})	Viscosity at 15 °C (59 °F)	
				µPa·s	(millipoise)
Air		29.00	287	174	(1.74)
Ammonia	NH_3	17.03	488.22	97	(0.97)
Argon	Ar	40	207.86	220	(2.20)
Carbon dioxide	CO_2	44.01	188.89	145	(1.45)
Dichlorodifluoromethane	CCl_2F_2	120.93	68.75	127	(1.27)
Dichloromethane	CH_2Cl_2	84.83	98		
Helium	He	4.00	2078.60	192	(1.92)
Hydrochloric acid	HCl	36.50	227.79	140	(1.40)
Hydrogen	H_2	2.02	4116.04	86	(0.86)
Krypton	Kr	83.80	99.22	246	(2.46)
Methane	CH_4	16.04	518.35	107	(1.08)
Neon	Ne	20.18	412.01	309	(3.09)
Nitrogen	N_2	28.01	296.84	173	(1.73)
Nitrous oxide	N_2O	44.00	188.96	143	(1.43)
Oxygen	O_2	31.99	259.91	199	(1.99)
Refrigerant R-134a	$C_2H_2F_4$	102.03	81		
Sulfur dioxide	SO_2	64.00	129.91	123	(1.23)
Sulfur hexafluoride	SF_6	146	57	152	(1.52)
Water vapor	H_2O	18.02	461.40	93	(0.93)

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rates for other gases from helium leakage rates determined by helium leak tests.

Computation of Transitional Flow Rates

Transitional flow, flow between the viscous and molecular regimes, is not fully understood. A number of models have been proposed to estimate the flow in this regime. The simplest and most used is the slip flow model. The flow is given by the following equation,

$$(3) \quad Q_2 = -\frac{\pi r^4}{8 n l} P_a \times \left(P_1 - P_2\right)\left(1 + 4\frac{\lambda}{r}\right)$$

where r is tube radius (meter), l is tube length (meter), P_1 is pressure upstream (pascal), P_2 is pressure downstream (pascal), λ is mean free path and $Pa = (P_1 + P_2)/2$. If the tube diameter or effective diameter is known, the flow for any gas can be calculated with the equation from the known pressures.

Also, the flow for gas can be estimated from the known flow for another gas under the same pressure conditions. Alternately, a crude estimate could be made using the formula for molecular flow. In general, estimation of flow based on the known flow of another gas in the transition range is not recommended.

Effect of Absolute Gas Temperature on Molecular Flow Leakage Rates

The effect of absolute gas temperature on conductance when the leakage flow is molecular should not be overlooked when estimating leakage rates by use of standard leaks. The conductance of both orifices and of tubes changes directly with the absolute gas temperature. Equation 4 shows how the new flow rate Q_2 at the new absolute temperature T_2 (K) compares with the original flow rate Q_1 at absolute temperature 1, through a leak for which both the leak path dimensions and the pressure difference across the leak remain constant, for the specific case of the molecular flow rate at new absolute temperature:

$$(4) \quad Q_2 = \sqrt{\frac{T_2}{T_1}}\, Q_1$$

Varying the Pressure Differential across a Leak

Because the rate of flow of a gas through a leak will be a function of either the molecular mass or the viscosity of the gas flowing, it is sometimes very important to know which type of leakage flow is occurring. This is especially true if a leakage rate must be expressed in terms of one gas such as air, when leakage must be measured by detecting helium flow. Often, the capillary leak is used under conditions that vary greatly from the conditions under which the leak was calibrated. The test gas, test pressure or both may be different from those used in the calibration of the leak. If it is not possible to obtain a true calibration figure under the new test conditions, it becomes necessary to attempt an estimation. The principles used in such estimations are presented next.

Varying System Pressure to Identify Types of Flow in Leaks

If leakage, or flow, can be measured by using a leak detector or a residual gas analyzer, the type of flow can often be identified by changing the pressure causing the flow of gas. All techniques of leak testing using a mass spectrometer leak detector involve the passage of a tracer gas through a presumed leak in a pressure barrier. This involves application of tracer gas to the high pressure side of the barrier and the subsequent detection of the tracer gas on the lower pressure side. In general, there are three types of gas flow: viscous, transition and molecular. The variables that control the type of gas flow that occurs in leaks are (1) viscosity of the flowing gas or gas mixture (Pa·s), (2) relative molecular mass M_r of the gas, (3) pressure difference causing the flow (Pa), (4) absolute pressure in the system (Pa absolute) and (5) absolute temperature of the flowing gas or gas mixture (K). Figure 10 shows the general relationship of flow type to gas pressure and radius of tubular conductance.

Conditions for Identification of Viscous Flow through Leaks

When the pressure across a leak is changed and the flow changes in proportion to the differences of the squares of the absolute pressures, the leakage can be identified as viscous flow. Viscous flow occurs in high pressure systems, such as systems pressurized with helium tracer gas and checked by the

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helium detector probe method. Figure 11 shows graphically how the viscous leakage rate changes as internal pressure is varied in test objects and systems leaking to the atmosphere. Figure 12 shows similar graphical relationships for externally pressurized test objects with leakage to an internal volume that is highly evacuated.

Conditions for Identification of Molecular Flow through Leaks

If the flow of gas through a leak changes in proportion with the difference between the pressures acting across the leak, the flow of gas is molecular. Molecular flow usually occurs in vacuum testing applications with helium spray application of tracer gas and mass spectrometer leak detectors attached to the internal volume of evacuated test objects. Figure 13 shows graphically how the molecular leakage rate varies linearly with the pressure differential as external pressure is varied on test objects and systems that are internally evacuated.

Conditions for Identification of Transitional Flow through Leaks

If the flow changes in response to changes of pressure by some relation between proportionality to differences in squares of pressures and proportionality to difference in pressures, the leak involves transitional flow. Figure 10 illustrates the regimes for each of these three types of flow of gases through leaks as a function of absolute gas pressures and diameter of leak passageways. In many cases, it is not always practical to vary pressures on parts under test to determine the types of leaks being detected. In instances where the leakage of a gas other than the tracer gas is of concern, it is best to assume the worst possible condition, which may be either viscous or molecular flow.

Calculating Effect of Pressure Changes with Viscous Flow through Leaks

Viscous flow occurs when the mean free path of molecules of the gas is much smaller than the cross sectional dimension of the physical leak. In this case, the leakage rate Q is proportional to the differences in the squares of the pressures on the opposite sides of the pressure barrier through which the leak penetrates. If the viscous flow rate Q_1 has been determined for a difference between pressure P_1 and pressure P_2 and then the pressures are changed to new values P'_1 and P'_2, the new flow rate Q_2 can be calculated by means of Eq. 5, for viscous flow rate at a new pressure,

$$\textbf{(5)} \quad Q_2 = \frac{P'^2_1 - P'^2_2}{P_1^2 - P_2^2} Q_1$$

FIGURE 10. Graphical presentation of conditions for viscous, molecular and transitional flow of gases through leaks, in terms of absolute gas pressure at 25 °C (77 °F) and radius of tubular conductance. Note that 1 Pa = 1.5×10^{-4} $lb_f \cdot in.^{-2}$.

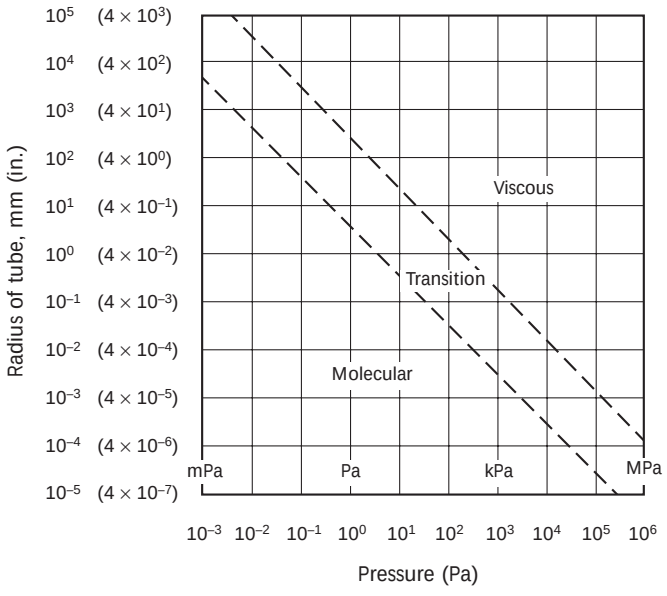

FIGURE 11. Graphical presentation of increase in viscous flow leakage ratio when pressurizing with 100 percent tracer gas, as a function of internal system pressure when leaking to atmospheric air.

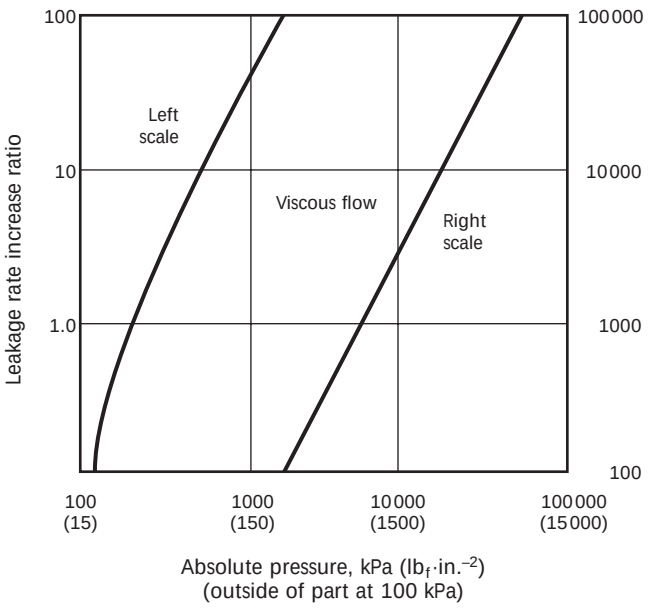

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FIGURE 12. Graphical presentation of increase in viscous flow leakage rate ratio when pressurizing a chamber with 100 percent tracer gas, as a function of external system pressure, when leaking into internally evacuated test objects in the chamber.

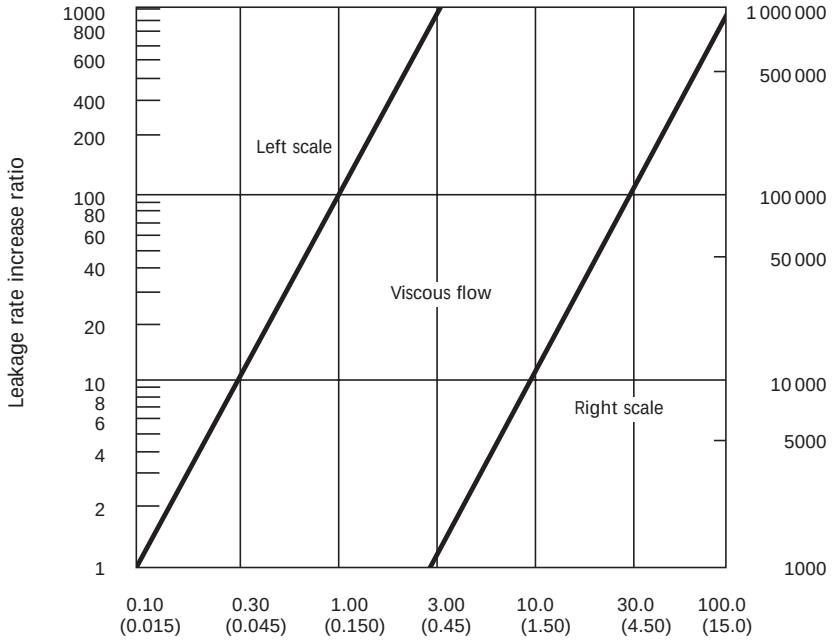
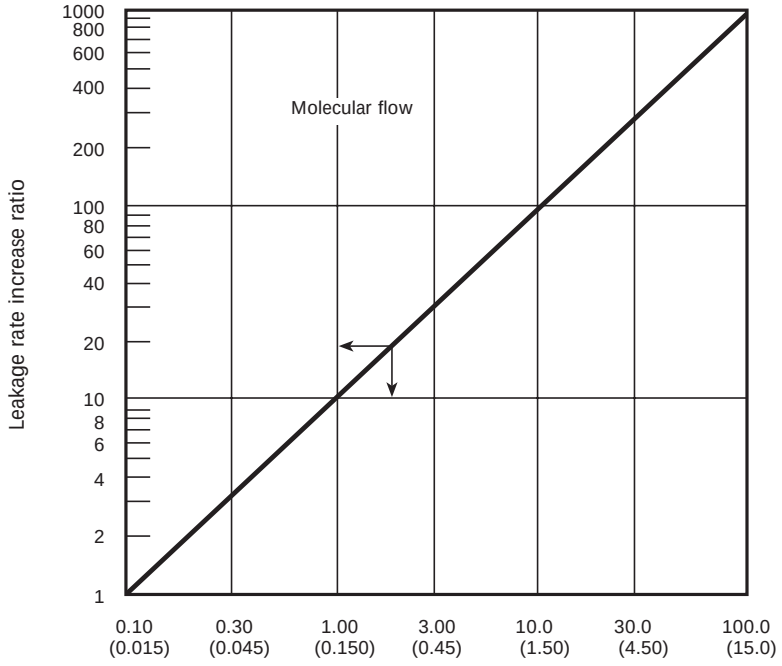


FIGURE 13. Graphical presentation of increase in molecular flow leakage rate ratio with molecular flow, as a function of external pressure of 100 percent tracer gas, when leaking into internally evacuated test objects of systems (inside of test system or parts at high vacuum).



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In Eq. 5, the pressures are all absolute pressures in pascal or pound per square inch ($lb_f \cdot in.^{-2}$). The old and new flow rates must be in the same units of leakage. Equation 5 for viscous flow through leaks would apply for leak testing of systems at higher than atmospheric pressure. It applies to a helium detector probe test on an internally pressurized test system leaking to the atmosphere.

Example Calculation of Capillary Leakage Rate at Different Pressures

Assume that a capillary standard leak (flattened tube) has been calibrated for a nitrogen flow rate of 2×10^{-5} Pa·m^3·s^{-1} (2×10^{-4} std cm^3·s^{-1}) with atmospheric pressure on the high side and zero pressure (vacuum) on the low side. It is desirable to predetermine the leakage rate if this same capillary leak is to be used with twice atmospheric pressure on the high side and atmospheric pressure on the low side. (Note that the pressure differential between high and low sides of the leak is atmospheric pressure of 100 kPa (1 atm) in both old and new cases.) Because the leakage rate is so high, it will be assumed that leakage occurs as viscous flow. By Eq. 5, the new flow rate Q_2 is calculated in Pa·m^3·s^{-1}:

$$Q_2 = \frac{2^2 - 1^2}{1^2 - 0^2} \; 2 \times 10^{-5}$$
$$= 6 \times 10^{-5}$$

This new flow rate represents a threefold increase when compared to the original flow rate obtained with internal atmospheric pressure leaking to vacuum.

Example Calculation of Leakage Rate after Pressuring Up Helium with Nitrogen

Another situation often encountered in mass spectrometer leak testing involves a standard capillary leak used with a mixture of helium and nitrogen at high pressure. This case occurs most commonly when the user attempts to increase helium leak testing sensitivity by the technique of pressuring up. This technique is used, for example, when a large volume test object or system is tested with helium tracer gas and leaks are detected with a detector probe. The vessel under test is originally filled with air at atmospheric pressure. The calibrated capillary leak is attached to the vessel and absolute pressure is raised to a total of 200 kPa (2 atm) by injection of helium tracer gas. Then compressed air or nitrogen is forced into the vessel, raising its absolute pressure even higher, for example, to 400 kPa (4 atm). (For large test volumes, 100 percent helium at high pressure may not be economical.)

In this example of pressuring up, the new total viscous flow rate Q_2 can be estimated:

$$Q_2 = \frac{4^2 - 1^2}{1^2 - 0^2} \; 2 \times 10^{-5}$$
$$= 30 \times 10^{-5}$$

The actual helium leakage rate, because the final pressurized mixture is only 25 percent helium, is only about 7×10^{-5} Pa·m^3·s^{-1} (7×10^{-4} std cm^3·s^{-1}) of helium. The result of pressuring up with air or nitrogen is an approximately linear increase in the helium flow rate through the leak. This example calculation would be valid only for viscous leakage. (Note that 1 Pa·m^3·s^{-1} = 10 std cm^3·s^{-1}.)

Limitations of Increasing Pressure with Molecular or Transitional Flow Leaks

For molecular flow leaks, increasing pressure with air would not result in an increase in the helium flow rate. In the transitional flow range, particularly when dealing with gas mixture, the situation (degree of enhancement of leak signals) is extremely difficult to predict. In these cases of unknown effects, it would be useful to make a graphical plot of leak signal amplitude as a function of total pressure within the leaking vessel to aid in determining the nature of a leak.

Correction for Aging of Helium Membrane Calibrated Leaks

As time passes, the internal helium pressure of glass permeation leaks is depleted (see Fig. 14). This depletion results from the gas leaking from the reservoir through the glass element and through any discontinuities in the reservoir into the atmosphere. If there is no appreciable leakage except through the glass permeation membrane, then the depletion rate of the leak can be estimated from the original number of moles of helium in the reservoir and the original leakage rate as follows. The leak rate $N(t)$ at a time t after the original calibration $N(t_o)$ can be determined from

$$\textbf{(6)} \quad N(t) = N(t_o)\, e^{-\frac{N(t_o)}{C(t_o)} \cdot t}$$

where the leakage rates are in mole per second, $C(t_o)$ is the original number of moles of gas in the reservoir and t is the elapsed time since the original calibration.

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The amount that the leakage rate changes as a function of time depends on the design of the calibrated leak and on usage conditions and can vary from less than 1 percent per year to more than 20 percent per year.

Correction for Temperature of Helium Membrane Standard Leaks

The permeation rate of helium through glass is described by an exponential expression:

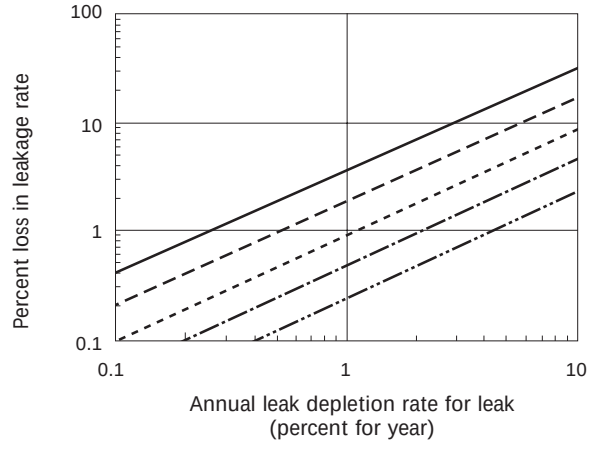
$$\textbf{(7)} \quad Q = AT\, e^{-\frac{b}{T}}$$

where A ($Pa \cdot m^3 \cdot s^{-1} \cdot K^{-1}$) and b (K) are constants and T is the absolute temperature (K). Table 2 gives typical values of the temperature coefficient b.[3]

TABLE 2. Temperature coefficients (measured by the National Institute of Standards and Technology) and corresponding glass types for helium permeation leaks.

Temperature Coefficient (K)	Probable Glass Type
≤ 2500	borosilicate
2700	fused silica
3000	Pyrex® 7740
3600	Corning® 7052

FIGURE 14. Decline in leakage rate as a function of depletion rate.



Frequently a linear approximation is used:

$$\textbf{(8)} \quad Q = Q_c \left[1 + a\left(T - T_c\right)\right]$$

where Q_c is the leak rate at the calibration temperature, T_c is the calibration temperature, T is the temperature at test conditions and a is a linear temperature coefficient, about 0.03 $°C^{-1}$ (0.05 $°F^{-1}$). Using the linear temperature expression will generally give adequate representation over small temperature variations, less than 5 °C (9 °F). For temperatures differences of 30 °C (54 °F), errors as large as 75 percent can be made using this simplifying assumption. For the lowest uncertainties the leak should be calibrated close to the temperature at which it will be used. A rough approximation to the linear correction is given in Fig. 15.

Glass Capillary Leaks for Tracer Gases Other than Helium

For gases other than helium, such as argon, neon or hydrogen, permeability rates in glass become small. The most common calibrating leak for these gases is a glass capillary leak (glass being chosen for its ease of fabrication of small capillary tubes and orifices). There are two areas in which these glass capillary leaks differ in characteristics from glass membrane leaks.

1. Depletion of internal pressure due to aging of capillary glass leaks is a function of the length of time the standard leak is in use, because the rate of gas flow in a capillary leak is a function of the total pressure drop across the reference leak (not the helium partial pressure difference that controls the flow rates of helium through glass membrane leaks). In a capillary leak, there is no helium flow unless the leak is being pumped on a vacuum pump or leak detector system.
2. Glass capillary standard leaks exhibit a negative temperature coefficient. This means that the capillary tube or orifice must decrease in diameter as temperature rises. This diameter reduction reduces the gas flow at a faster rate than the internal pressure rise increases the flow rate as temperature rises.

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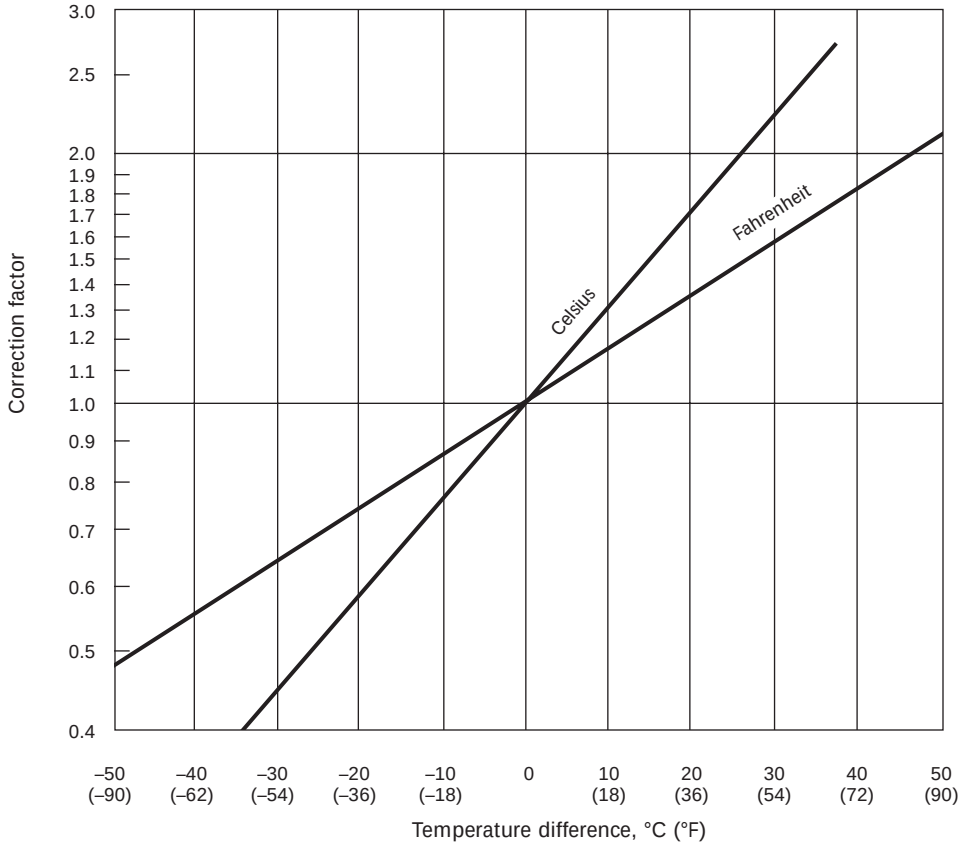
Figure 15. Temperature correction factor for a silica membrane standard helium leak used at operating temperatures that differ from temperature during initial calibration. To correct calibrated leakage rate for temperature, multiply by correction factor.



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PART 4. Calibration of Standard Reference Leaks

Commercial Sources for Calibrated Leaks

Commercially available permeation leaks have been limited to helium in the past because glass elements were predominantly used. It is now possible to obtain polymer permeation elements that function with other gases including argon, sulfur hexafluoride and many refrigerants. In addition many calibrated physical leaks are also commercially available. The choice of gases in these physical leaks, predominantly capillary type, are large and include most noncorrosive, nontoxic industrial gases.

Calibration Techniques for Artificial Physical Reference Leaks

Calibrated leaks are available with eight decades of leakage values. Because of this large range of leakage, calibration is difficult. The five techniques of measuring leakage rates are (1) isobaric volume change, (2) pressure rise, (3) pressure drop across a known conductance, (4) pressure measurement at constant pumping speed and (5) comparison.

Isobaric Volume Change Calibration of Standard Leaks

A schematic diagram of the equipment used in the isobaric volume change technique of leakage rate measurement is shown in Fig. 16. One side of the leak is attached to a vacuum system; the other side is attached to a gas reservoir at atmospheric pressure. To this reservoir is attached a capillary of known cross section. A slug of indicating fluid is placed in this capillary. As gas leaks from the volume into the vacuum, the slug of fluid travels down this capillary to keep the pressure in the reservoir constant. The leakage rate is determined by measurement of the volume displaced by the travel of the slug down the capillary:

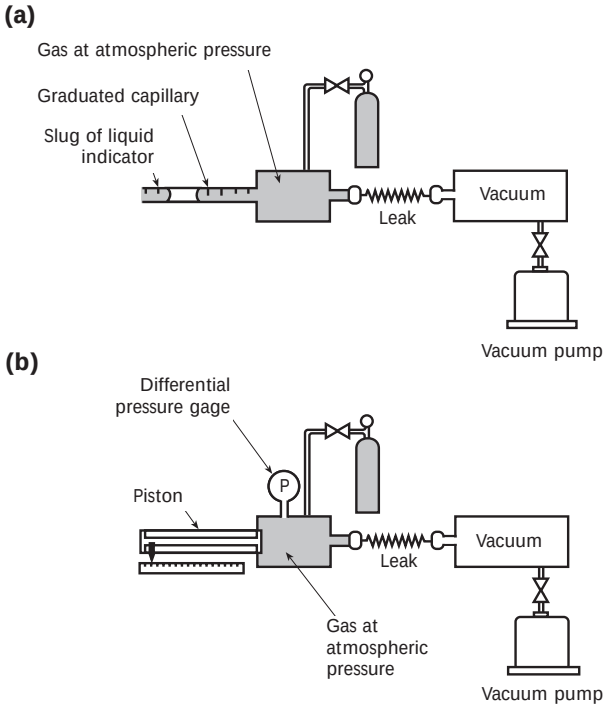
$$\textbf{(9)} \qquad Q = \frac{P\left(V_2 - V_1\right)}{t}$$

where Q is leakage rate ($Pa \cdot m^3 \cdot s^{-1}$), P is pressure in the gas volume (pascal), $V_2 - V_1$ is volume displaced during travel of the indicating fluid (cubic meter) and t is time (second).

Limitations of Isobaric Volume Change Leak Calibration

The primary limitation of the isobaric volume change technique is the size of the capillary tube involved in the volume measurement (see Fig. 16a). It is difficult to obtain a liquid that can be placed in a small capillary tube and that subsequently can be forced out the other end. For this reason, the practical limitation of the capillary tube technique of volume displacement measurement is in the range of 1×10^{-6} $m^3 \cdot s^{-1}$ (2×10^{-3} $ft^3 \cdot min^{-1}$). It would theoretically be possible to use a slightly larger capillary and to take longer periods of time between readings but errors might arise from permeation of gas either through the liquid slug or through

FIGURE 16. Leak calibration by isobaric volume change: (a) with capillary tube; (b) with differential pressure gage.



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the walls. An error might also be introduced by a change in barometric pressure or a change of ambient temperature. This becomes particularly critical in the calibration of small leaks, because a slight temperature change might produce a volume change greater than that due to efflux of gas.

Selection of Liquid for Capillary Slug That Indicates Volume Change

It is desirable that the indicating fluid not be permeable to the gas being calibrated. For this reason, mercury is the preferred indicating fluid. Because of its high surface tension, mercury cannot be placed in a small capillary. This drastically limits the size of the leak that may be calibrated with mercury. For these measurements it is desirable to use a liquid with a low vapor pressure so that the leak is not contaminated by the calibration fluid. Unfortunately, most liquids of low vapor pressure are also of high viscosity and make it difficult to obtain an accurate measurement of the flow of liquid displaced in the capillary. These fluids also tend to form bubbles at the end of the capillary. The added pressure necessary to remove the bubble of liquid from the end of the capillary prevents the experiment from being isobaric.

Piston and Differential Pressure Gage in Isobaric Volume Change Tests

Another technique for measuring volume displacement is with a piston to replace the effluent gas. In this technique, a differential pressure gage is used to measure the pressure in the gas volume and the piston is manually pushed into the volume at such a rate as to keep the pressure constant. The pressure gage need not be calibrated because the readings are made only when the differential pressure gage is at zero indication. This technique can readily measure leakage as low as 10^{-9} Pa·m^3·s^{-1} (10^{-8} std cm^3·s^{-1}).

Pressure Rise Calibration of Standard Leaks

The second technique of calibrating leaks is by means of the pressure rise technique. A leak and its gas supply are attached to an evacuated chamber of known volume in the arrangement sketched in Fig. 17. The leaking gas is allowed to accumulate in this volume and the pressure rise is measured at various intervals. The leakage may then be computed by Eq. 10:

$$\textbf{(10)} \quad Q = V \frac{dP}{dt}$$

where Q is leakage rate (Pa·m^3·s^{-1}), V is volume of evacuated chamber (m^3), P is pressure in chamber (Pa absolute), t is time (s) and dP/dt is time rate of pressure change (Pa·s^{-1}).

Limitations of Leak Calibration by Pressure Rise

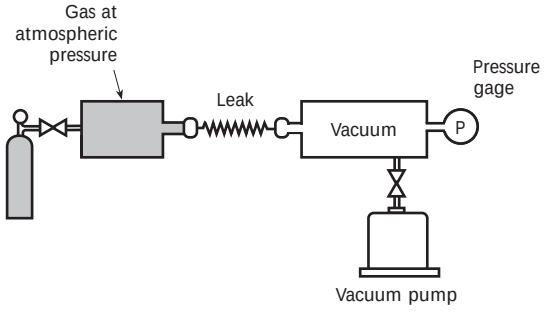
The major difficulties with the pressure rise calibration technique occur in measurement of pressure. The pressure in an evacuated system usually does not stay constant, but gradually increases due to outgassing of the walls of the chamber. The pressure rise due to this desorption must be taken into account in calculations. The type of pressure instrumentation to be used for the measurement depends on the range of pressures that are expected to be measured. Table 3 lists some gages that may be used and their ranges.

The effect of desorption on the uncertainty of the measurement will depend on the ratio of the apparent leakage because of gas desorption to that of the leakage to be measured. It should be recognized that the rate of desorption is usually not constant and will in general be a function of temperature.

TABLE 3. Gages for pressure rise leak calibration.

Gage	Pressure Range	
	Pa	(lb_f·in.$^{-2}$)
Mass spectrometer	< 10^{-3}	(< 1.5×10^{-7})
Molecular drag	10^{-4} to 10^{-10}	(1.5×10^{-8} to 1.5×10^{-14})
Capacitance diaphragm	10^{-1} to 10^{5}	(1.5×10^{-5} to 1.5×10^{1})

FIGURE 17. Leak calibration by pressure rise technique.



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Practical Example of Leak Calibration by Pressure Rise

As noted just previously, the pressure rise technique for calibrating a standard reference leak in a laboratory depends on the outgassing surface area as well as the volume of the system to be evacuated. The upper size limit for large leaks to be measured by this technique would be governed mainly by the largest size of test volume that could be realistically placed within a laboratory. Probably leaks as large as those with 1 $Pa \cdot m^3 \cdot s^{-1}$ (10 std $cm^3 \cdot s^{-1}$) leakage rates would be near the upper limit.

The size limit for small leaks measured by the pressure rise calibration technique would be governed by the accuracy of measurement of the volume of the evacuated test system and the accuracy with which the pressure change could be measured. These may place the lower limit of leak size in the range of leakage rates from 10^{-5} to 10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-4} to 10^{-5} std $cm^3 \cdot s^{-1}$).

In a practical industrial laboratory, calibration would be performed by measuring the rate of pressure rise of a well conditioned evacuated system volume when closed off from external sources of gases. The result may be a curve similar to the lower curve shown in Fig. 18.

Following that test, the reference physical standard leak would be attached to the same test volume, which would be evacuated to the same vacuum level as in the first test, with the valve closed between the chamber and the standard leak. Again, the pressure rise of the evacuated system would be measured, this time with the valve open so that air enters through the standard leak to be calibrated. The rate of pressure rise is higher with the leak in place and the curve from the second test with the leak admitting air to the evacuated chamber would be higher than the initial curve, as indicated by the higher curve of Fig. 18. The vertical difference between the two curves (with and without the leak opened to the evacuated chamber) indicates the theoretical rate of rise or pressure due to the leak.

With this number, together with the values for system volume and test time, the rate of leakage through the standard leak under calibration test can be calculated. Figure 19 shows the relation of pressure difference to the elapsed test time and approaches a linear (straight line) relationship. The leakage rate is computed in SI units from the relation

$$\textbf{(11)} \quad Q = V \frac{\Delta P}{t}$$

where V is volume (cubic meter), ΔP is pressure difference (pascal) and t is elapsed test time (second). For example, for the case shown in Fig. 19, the calculation is as follows:

$$\begin{aligned} Q &= 0.382 \times \frac{0.0169}{700} \\ &= 9.2 \times 10^{-6} \ \text{Pa} \cdot \text{m}^3 \cdot \text{s}^{-1} \\ &= 9.2 \times 10^{-5} \ \text{std cm}^3 \cdot \text{s}^{-1} \end{aligned}$$

FIGURE 18. Pressure rise as a function of time elapsed after evacuating test chamber, during calibration tests of physical reference leak.

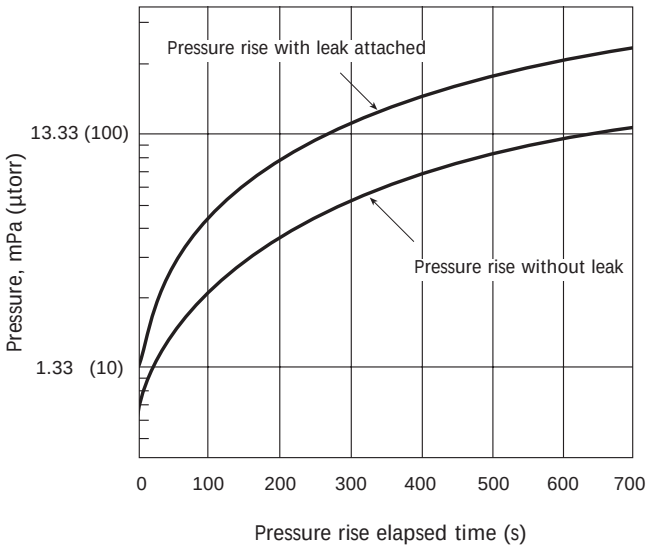
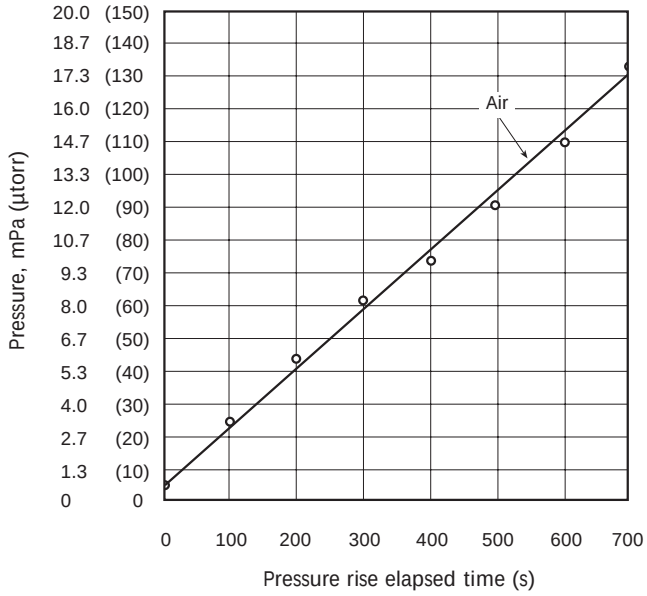


FIGURE 19. Pressure difference resulting from leakage through standard leak.



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Calibration of Standard Leaks by Pressure Drop across a Known Conductance

A third technique of measuring leakage rates is by measuring the pressure drop across a known conductance *C*. This technique is illustrated in Fig. 20. The pressure drop ($P_1 - P_2$) across a known conductance is proportional to the flow rate *Q*, as indicated by Eq. 12:

$$\textbf{(12)} \quad Q = C\left(P_1 - P_2\right)$$

With molecular flow the conductance *C* may be designed from theoretical grounds and such a conductance can be accurately constructed.

Limitations of Pressure Drop Leak Calibration Technique

The major difficulty with the pressure drop calibration technique is in obtaining accurate pressure measurements. Ionization gages have been used for the pressure measurement in evacuated systems, but their readings are often questionable. Because their sensitivities are more often in doubt, pressure drop leakage tests are also used to calibrate ionization gages.

An alternative to using an ionization gage is to use a molecular drag gage, sometimes referred to as a spinning rotor gage. This instrument is stable with time and can achieve accuracies of ±10 percent even if uncalibrated over the pressure range of 10^{-4} Pa to 10^{-1} Pa (1.5×10^{-8} to 1.5×10^{-5} $lb_f \cdot in.^{-2}$).

Calibration of Standard Leaks by Pressure Measurement at Constant Pump Speed

A fourth technique of calibrating the flow of a leak is by measuring the pressure it produces in a vacuum system that is pumped at a known speed (see Fig. 21). This is the limiting case for Eq. 12, when P_2 is zero. The equation then being used takes the form of Eq. 13:

$$\textbf{(13)} \quad Q = SP$$

where *S* is the pumping speed ($m^3 \cdot s^{-1}$) of the system (usually governed by an orifice) and *P* is the ultimate pressure (pascal) attained within the vacuum chamber while being pumped. The system is usually constructed so that the pumping speed is controlled by molecular kinetics considerations and can be rigorously calculated from theoretical grounds. The disadvantage of the pumping speed technique is, again, that the pressure of the system must be accurately measured. If the leakage *Q* and the pumping speed *S* are known, *P* can be derived using the above equation. This type of system has also been used to calibrate pressure gages.

Mass Spectrometer As Pressure Gage in Leak Calibrations

If a mass spectrometer is used as the pressure gage, some accuracy is gained because the error due to outgassing is minimized. The pumping speed system has essentially the same flow pattern as the mass spectrometer leak detector. In a mass spectrometer leak detector, Eq. 13 takes the form of Eq. 14:

FIGURE 20. Leak calibration by pressure drop across a known conductance.

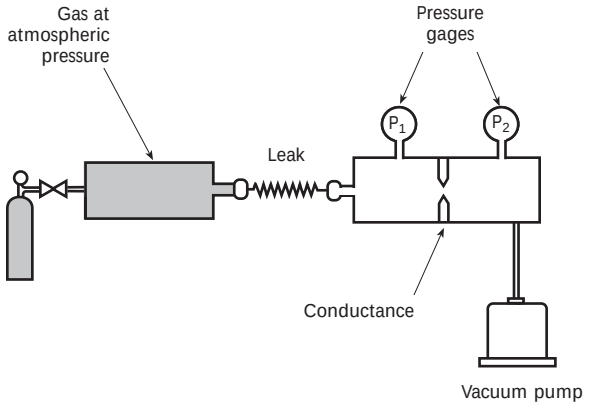
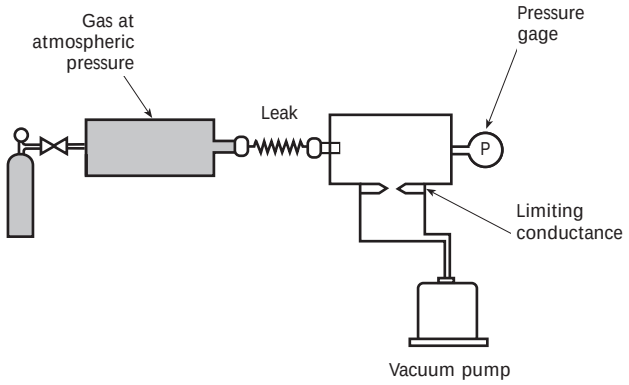


FIGURE 21. Leak calibration by pressure measurement at constant pumping speed.



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(14) $Q = SKa = C_1 S$

where Q is leakage rate ($Pa \cdot m^3 \cdot s^{-1}$); S is pumping speed, a constant ($m^3 \cdot s^{-1}$); K is conversion factor for pressure from collector current reading ($Pa \cdot mA^{-1}$); a is collector current reading in mass spectrometer (mA); and proportionality constant C_1 equals Ka (Pa). In most cases, a and K are not known, but a proportionality constant C_1, the product of these two numbers, is used. Providing that the response of the leak detector is linear, the mass spectrometer can be used to calibrate leaks by comparison to standards calibrated by other techniques.

Comparison Calibration

A fifth type of calibration is by comparison with a calibrated leak whose measurement is traceable to the National Institute of Standards and Technology. This technique can be used over a wide range of leakage rates, 10^{-11} to 10^{-3} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-10} to 10^{-2} std $cm^3 \cdot s^{-1}$) and with a wide range of gases. With a mass spectrometer type leak detector (helium only) a calibrated leak may be compared to a leak whose leakage rate is to be determined. The leakage rate is calculated with the following expression:

(15) $$Q_{unk} = Q_{std} \frac{H_{unk}}{H_{std}}$$

where Q_{unk} is the leakage rate of the unknown leak and Q_{std} is the leakage rate of the calibrated leak, H is the mass spectrometer signal corresponding to the two measured leakage rates. This equation assumes that the mass spectrometer gives a linear response to partial pressure changes and that the pumping speed of the system is stable over the testing period. To minimize uncertainties due to nonlinearities in the mass spectrometer the calibrated leak should be closely matched to the unknown leak. For the most accurate results, it is usually necessary to have the two leaks register signals in the same decade of the measuring instrument.

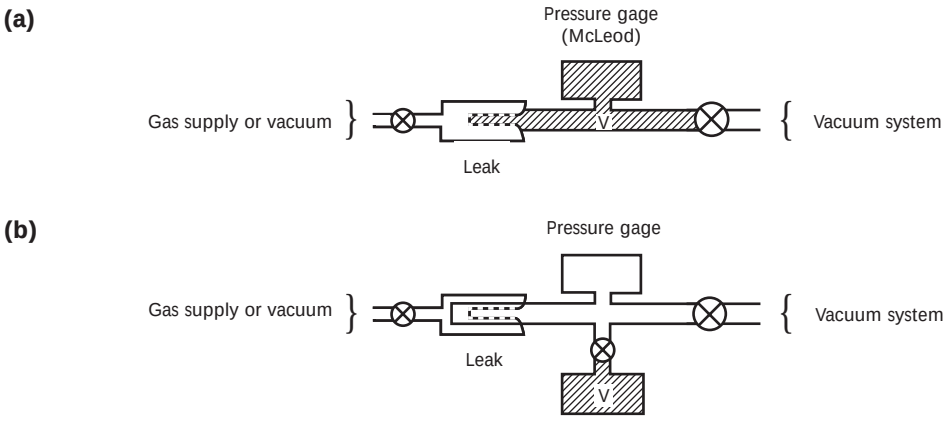
Matching Standard Leakage Rate to Permissible Leakage Rates

The standard or reference leakage rate used in leak testing should be of the approximate value of the permissible leakage rate of the test object. This must be so if the response of the detector to leakage is not linear. The smaller the standard leakage rate, the greater the difficulties associated with it. If the standard leak is substantially different from the permissible leakage (a contingency that may result from the difficulty of making small standard leaks), the response of the detector to different leakage rates becomes important.

Calibration of Standard Leaks with Different Gases

Basic leakage rate measurements are necessary for the calibration of primary standard leaks used in connection with tracer gas leakage rate measurement systems. Fortunately, controlled laboratory conditions are practical for such calibrations and time is not an essential factor. Figure 22 shows schematically two basic systems, constant

FIGURE 22. Standard leak calibration, *dQ/dt:* (a) pressure change calibration system; (b) volume change calibration system.



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volume and constant pressure. The leakage rate Q for the pressure change calibration system of Fig. 22a is given by Eq. 16:

$$\textbf{(16)} \quad Q = \frac{d(PV)}{dt} = V\frac{dP}{dt}$$

The leakage rate Q for the volume change calibration system of Fig. 22b is given by Eq. 17:

$$\textbf{(17)} \quad Q = \frac{d(PV)}{dt} = P\frac{dV}{dt}$$

It may be noted that no reservoir is shown for the leak in Fig. 22. Elimination of a fixed upstream leak reservoir has two important advantages. First, using a vacuum upstream permits an evaluation of outgassing and other extraneous sources of gas arising in the calibration system. Second, the same leak element can be calibrated for many leakage rate values for various gases simply by varying the upstream gas and pressure.

For leaks in the 10^{-9} Pa·m^3·s^{-1} (10^{-8} std cm^3·s^{-1}) range, accumulation times as long as a week have been used for increasing measured pressure change in the constant volume manifold V of the $V(dP/dt)$ calibration system. Conversely, times of the order of 100 s have been used for increasing the pressure in the known volume V from an insignificant pressure to an arbitrary pressure of 0.4 Pa (5.8×10^{-5} lb$_f$·in.$^{-2}$) in the $P(dV/dt)$ system for leaks in the 10^{-5} Pa·m^3·s^{-1} range.

The procedure for this second technique is as follows. The known volume V is evacuated to a negligible pressure, for instance less than 10 mPa (1.5×10^{-6} lb$_f$·in.$^{-2}$), and then valved off. The valve to the vacuum system is then closed; thereafter, gas from the leak is admitted to the manifold. At the instant the pressure in the manifold attains a preselected value P, a timer is started. Opening the valve to V will lower the manifold pressure temporarily, but the pressure will again increase steadily because of the continued inflow of gas from the leak. When the pressure again climbs to the value P, the time is stopped. The only difference between the conditions when starting and stopping the time is that the pressure in V increased from essentially O to P.

Closing

Calibrated leaks have a vital role in leak testing programs. Attention to the proper calibration techniques can enhance the operator's understanding of the test procedure and hence improve the reliability of the leak testing being performed.

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4

CHAPTER

Safety Aspects of Leak Testing

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PART 1. General Safety Procedures for Test Personnel

Test Personnel Dedication to Safety Procedures

The range of applications of leak testing is so wide and varied that no single set of safety rules for protection of personnel and property can be made to cover all cases. Leak testing personnel must be made aware of job hazards and be receptive to proper training to protect themselves and others working close by. On many jobs, testing must be performed at odd hours and under awkward conditions. Nightshift work, weekend work and work in unheated areas in winter and uncooled areas in summer are common. Climbing through manholes, climbing ladders and scaffolds, balancing on structural members or other awkward maneuvers may all be in a day's work.

In addition to technical abilities and training in test procedures, competent technicians must have other attributes. They must be determined to do a safe job under any circumstances. They must be willing to listen and to cooperate with the many types of personnel encountered in the field, but they must not compromise the safety aspects of their work for the convenience of themselves, their crew or someone else.

Need for Safety Training of Test Personnel

Test personnel can acquire a proper attitude and point of view toward safety only through training coupled with experience. The training program should include first aid and lifesaving techniques. In situations where irritating, toxic or corrosive dusts, gases, vapors or fluids are present, test technicians should be given special training to make sure that they are familiar with the properties of these substances and with the methods of controlling the hazards. Emergency procedures must be learned and test personnel must know where medical and hospital assistance is available at all hours. Leak testing technicians should have more thorough training in accident prevention than the regular plant or construction workers. For leak testing personnel, safety involves not a set pattern of activity but a complex and constantly changing set of problems.

The United States Department of Transportation is responsible for the rules governing training requirements for handlers of hazardous materials (HAZMAT). The *Code of Federal Regulations*[1] states the requirement that hazardous materials handlers receive training at least every two years by someone licensed to provide such training.

Hazards in Leak Testing

Precleaning of test surfaces is required for leak testing where surface contamination might prevent entry of fluid tracers. Many cleaning processes involve liquid solvents and vapors, some of which present possible hazards of flammability, toxicity or asphyxiation. Liquid leak tracers often have similar hazards, if vapors accumulate in working areas. Ventilation must be provided to prevent hazardous vapor concentrations. Electrical systems must be properly grounded and enclosed or protected to prevent ignition of flammable vapors in air. Access to test surfaces, particularly on large structures, can be hazardous if scaffolding is inadequate, lighting is insufficient or bad housekeeping creates hazards such as oily work surfaces or obstructions in passageways.

Special Safety Considerations in Testing Systems under Pressure

When a pressure or a vacuum vessel is fabricated, some means of testing this vessel must be used to predict safe performance. It is sometimes necessary to exceed the designed operating conditions during initial pressure testing. This pressurization requires many safety considerations to ensure proper protection of personnel. Greater respect for high pressure has led to increased safety emphasis, with the result that overall safety experience has been good.

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Psychological Factors and the Safety Program

The nature of leak testing work dictates that a competent safety program be used. Much of the success of such a program depends on its acceptance by those to whom it is directed. Never has there been a safety device or a safety program that some human being could not disrupt or impair. The human factors that operate at all levels in industry are perhaps the most potent factors for success or failure of a safety program. The president of a company, the safety director and the leak testing supervisor may either emphasize safety or subordinate it to production goals. Production, maintenance and testing personnel are also important contributors to safety and their full cooperation is vital. Individual differences affect personnel acceptance of a safety program. These differences must be recognized when motivating work groups to use good safety practices at all times.

The safety program must be designed with an understanding of motivation of people. To want something is to be motivated, but not to want something also requires motivation. To use a safety device to protect one's fingers from a saw shows motivation for safe practice. However, the desire to ignore a safety device that interferes with production is caused by still other motives. Conflicting motivations should also be considered in any attempt to understand human relations that influence the success of safety programs. Industry has recognized the effects that attitudes can have on production, plant morale and plant safety. As a result, management should spend considerable effort to determine the attitudes of its workers. Measuring, developing and changing attitudes constitute a major problem for personnel and psychologists and are of extreme importance to the safety program.

Personnel Safety Training Requirements

There should always be concern with safety training of personnel. The learning process starts at birth. Most early safety training is through experience, as when a child may have touched a stove and been burnt, played with a knife and been cut or fallen from a precarious treehouse and broken a bone. However, personnel testing today's vessels that hold gases, vapors and liquids at various temperatures and at pressures ranging from high vacuums (in nanopascal) to high pressures (in megapascal) cannot afford to learn safety by causing or experiencing disasters. In today's industry it is the responsibility of the employer to provide employees adequate training on safety practices for for their job responsibilities.

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PART 2. Control of Hazards from Airborne Toxic Liquids, Vapors and Particles

Toxic Gas and Vapor Sensors and Alarms

Detection and warning of the presence of toxic vapors or gases in a work area can be provided by various types of electronic instruments with detectors and alarm systems responsive to many different airborne chemicals, fumes, smoke or particulate matter. For general protective service applications, wall mounted, self-contained monitors can detect and provide audible signals of the presence of various combustible gases, fumes and microscopically sized airborne particulate contaminants. These are typically provided with pilot lights to indicate the presence of alternating current line power and standby battery power. Flashing red lights actuated when abnormal concentrations of contaminants occur. The alarm sensitivity control can be adjusted to allow compensation for the normal ambient quiescent atmospheric contamination levels.

The sensor assembly of a typical gas monitor and alarm system contains a heated semiconductor element whose resistance to current flow varies as a function of the type and quantity of gas molecules adsorbed on its surface. The heater effectively boils off adsorbed contaminants. The sensor resistance is thus primarily a function of the adsorbed gas molecules, whose number is related to their relative concentrations in the ambient air atmosphere. The sensor is designed for more than 50 000 exposures and can detect 50 $\mu L \cdot L^{-1}$ of many combustible and toxic gases and vapors, including those listed in Table 1.

Selecting Leak Testing Sites with Adequate Ventilation

When possible, testing of structures such as pressure vessels should be performed in a well ventilated area isolated far from other processes such as welding or grinding. A room is desirable with a high roof, adequately ventilated at its apex and with enough low level inlets. Conversely, a small room with a low roof and a minimum of opening for ventilation should not be used for testing with potentially dangerous tracer gases such as hydrogen.

Ventilation to Reduce Vapor Hazards in Solvent Use Areas

Many applications of leak testing in various industries have, as a prerequisite to testing, some cleaning operation. This operation often uses volatile solvents that can contaminate the air within enclosures; therefore, some consideration must be given to ventilating the working areas with explosion-proof equipment. Local exhaust systems have several inherent advantages over general ventilation for removal of atmospheric contaminants. They permit removal of hazardous vapors before they spread throughout the work area, they provide economy of air flow and they involve less heat loss. Local exhaust systems are impractical where the contaminant is usually a solvent vapor. Local exhausts may be unsuitable because there are a multitude of sources of vapor, or the source may be extensive, or the amount of ductwork to connect all the necessary hoods may be too costly or impractical.

The basic purpose of volatile solvents used in industrial cleaning operations is to dissolve or loosen contamination such as grease, dirt and other impurities and so facilitate their removal. The solvent may tend to evaporate into the atmosphere. This evaporation of volatile constituents leaves behind some physically changed substance that must be removed from test surfaces. Thus, the use of solvents in these processes involves polluting the air with vapor. The aim of the safety engineer is to keep this vapor concentration as low as possible, certainly below the toxic limit. If local exhaust systems are inadequate, such widely distributed solvent vapors can sometimes be controlled by diluting the general room atmosphere with outdoor air fast enough to keep the concentration of toxic vapor in the air of the working space within safe limits.

Ventilation Rate Calculations for Safe Use of Vaporizing Solvents

The rate of solvent evaporation can easily be ascertained, as can the chemical nature

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of the solvent. It is known that the weight of a given volume of vapor that evaporates from a liquid is proportional to its molecular weight. It is possible, then, to calculate how much air must be mixed with a solvent vapor to hold the concentration down to safe limits. Table 2, from which general ventilation can be calculated, is based on the formula of Eq. 1 (in SI units):

$$\textbf{(1)} \quad V_R = \left(2.4 \times 10^7\right) \frac{W}{M} \left(\text{VDC}\right)$$

where V_R is rate of ventilation ($m^3 \cdot min^{-1}$); W is rate of solvent evaporation ($kg_m \cdot min^{-1}$); M is molecular weight of solvent (unified atomic mass unit); and VDC is ventilation design concentration (from Table 2).

Equation 1 does not give the maximum acceptable concentration for the compound. Instead, it is the ventilation design concentration that has incorporated in it a safety factor based on toxicity, order and experience. Equation 1 converts to Eq. 2 in English units:

$$\textbf{(2)} \quad V_R = \left(4 \times 10^8\right) \frac{W}{M} \left(\text{VDC}\right)$$

where V_R is rate of ventilation ($ft^3 \cdot min^{-1}$); W is rate of solvent evaporation ($lb_m \cdot min^{-1}$); M is molecular weight of solvent (unified atomic mass unit); and VDC is ventilation design concentration (from Table 2).[2]

Neither the maximum allowable concentration (MAC) nor the threshold limit value (TLV) should be used for calculating the ventilation design concentration. The degree of vapor dilution in the working space is bound to be uneven. In addition, the concentrations must always be maintained below the MAC or TLV to provide a factor of safety. In turn, this factor of safety depends on whether the solvent vapor is to be controlled because

TABLE 1. Combustible and toxic gases and vapors detectable by area monitors and alarm systems.

Acetaldehyde	1,1 dichloroethane	2-hexanone	Nitroglycerin
Acetone	1,2 dichloroethane	Hexone	Nitromethane
Acetonitrile	Diethylamine	Hydrogen	Nitrotoluene
Acetylene tetrabromide	Diethylamino ethanol	Hydrogen bromide	Ozone
Alcohol	Diisobutyl ketone	c-hydrogen chloride	Pentane
Allyl alcohol	Dimethylamine	Hydrogen cyanide	2-pentanone
c-allylglycidylether	Dimethylaniline	c-hydrogen sulfide	Perchloroethylene
Ammonia	Dimethylformamide	Isoamyl alcohol	Petroleum distillate
Benzene	1,1 dimethylhydrazine	Isobutyl alcohol	Phenylether
Benzoyl chloride	Dinitrobenzene	Isopropyl alcohol	Propane
Benzoyl peroxide	Dinitrotoluene	Ketone	Propargyl alcohol
Butane	Dipropylene glycol	Liquid propane gas	Propylene oxide
2-butanone (MEK)	methyl ether	Methane	Propyne
2-butoxyethanol	Epichlorhydrin	Methyl acetylene	Refrigerant-11, -134a etc.
Butyl acetate	2-ethoxyethanol	Methylal	Steam
Butyl alcohol	Ethyl alcohol	Methyl alcohol	Stibine
Camphor	Ethylamine	Methylamine	Sulfur dioxide
Carbon monoxide	Ethyl benzene	Methyl n-amyl ketone	Sulfur hexafluoride
Carbon tetrachloride	Ethyl bromide	Methyl butyl ketone	Tetrachloronaphthalene
Chloroacetaldehyde	Ethyl butyl ketone	Methyl cellosolve	Tetranitromethane
Chlorobenzene	Ethyl chloride	Methyl chloride	Toluene
c-chloroform	Ethyl ether	Methyl chloroform	1,1,1 trichloroethane
1-chloro-1-nitropropane	Ethyl formate	Methylcyclohexane	1,1,2 trichloroethane
Chloropicrin	Ethylenediamine	Methylcyclohexanol	Trichloroethylene
Chloroprene	Ethyl dichloride	Methylene chloride	Trichloronaphthalene
Cumene	Ethylene oxide	Methyl ethyl ketone	1,2,3 trichloropropane
Cyclohexane	Formaldehyde	c-methyl mercaptan	Trinitrotoluene
Cyclohexanol	Furfuryl alcohol	Naphtha	Turpentine
Cyclopentadiene	Gasoline	Naphthalene	Xylene
DDT	Glycol monoethyl ether	Natural gas	
Diacetone alcohol	Heptane	Nitrobenzene	
Diazomethane	Hexachloroethane	p-nitrochlorobenzene	
Diborane	Hexane	Nitroethane	

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of its inherent toxicity or its disagreeable odor.

Example of Ventilation Rate Calculation

For example, suppose that 3 L of methyl ethyl ketone were evaporated per hour. One liter of methyl ethyl ketone requires a ventilation quantity of 1800 m^3 of air; 3 L would then require 3 times 1800 equals 5400 m^3 of air. If this is needed per hour, the ventilation rate per minute would be 5400 divided by 60 equals 90 $m^3 \cdot min^{-1}$. (Users of the English system should use a conversion of 35 $ft^3 \cdot m^{-3}$ and 2 $pt \cdot L^{-1}$, or 17 $ft^3 \cdot pt^{-1}$ for each $m^3 \cdot L^{-1}$.)

It is important to note that this example assumes there is perfect mixing of the clean air with the solvent vapor, but in practice this does not occur. The ventilation rate calculated is therefore a minimum. It should be increased depending on other factors involved, such as type and location of air diffusers, location of people in the working space and relative toxicity of the vapor.

The volume of the space in which the work is done does not enter the calculation for ventilation design concentration. This is a variance from the common practice of specifying ventilation requirements in terms of number of air changes per minute, which of course directly involves the work space volume. The rule of thumb based on room air changes per minute, thus in widespread use over many years, has been used improperly more often than properly. This is especially true when there are unwanted contaminants being released within the space.

Example of Evaluation of Health Hazard from Dilution Rate Table

The following is an example in which the degree of health hazard resulting from a solvent exposure is to be evaluated using data from Table 2. Trichloroethylene is being used in an enclosed $6 \times 6 \times 3$ m work space. In an 8 h day, 20 L of the solvent are lost through evaporation. There are two air changes per hour. Is there a potential health hazard?

Solution in metric units. The work space volume is $6 \times 6 \times 3 = 108$ m^3. Ventilation rate at two changes per hour provides $2 \times 108 = 216$ $m^3 \cdot h^{-1}$. The rate of solvent evaporation is $20/8 = 2.5$ $L \cdot h^{-1}$.

The dilution rate or ventilation ratio is 216 divided by 2.5 = 86 $m^3 \cdot L^{-1}$. The proper ventilation ratio (from Table 2) should be 2700 $m^3 \cdot L^{-1}$. Therefore, the ventilation rate is totally inadequate and a health hazard is indicated. At least 2700 divided by 86 is 31 times as much ventilation is required for the safe

TABLE 2. Dilution rates for common industrial solvents recommended for use in ventilation design (SI units), after Hemeon.[2]

Solvent	Molecular Weight[a] (M)	Density[b] ($kg \cdot m^{-3}$)	VDC[c] ($\mu L \cdot L^{-1}$)	Ventilation Ratio or Dilution Rate[d] (Quantity of Air per Unit, Solvent)				Possible Complaints If Twice VDC[c] Exceeded
				($m^3 \cdot kg^{-1}$)	($m^3 \cdot L^{-1}$)	($ft^3 \cdot lb^{-1}$)	($ft^3 \cdot pt^{-1}$)	
Acetone	58	790	150	2800	2200	46 000	38 000	Disagreeable
Benzene	78	880	——[e]	——[e]	——[e]	——[e]	——[e]	——[e]
Carbon tetrachloride	154	1580	——[e]	——[e]	——[e]	——[e]	——[e]	——[e]
Ether	74	720	75	4300	3000	72 000	54 000	Disagreeable
Ethyl alcohol	46	790	250	2100	1600	34 000	28 000	Disagreeable
Isopropyl alcohol	60	790	150	2700	2100	45 000	37 000	Disagreeable
Methanol	32	800	100	7500	6000	125 000	103 000	Toxic
Methyl-ethyl ketone	72	810	150	2200	1800	37 000	37 000	Disagreeable
Pentachloroethane	202	1670	——[e]	——[e]	——[e]	——[e]	——[e]	——[e]
PMV naphtha	110	750	200	1100	300	18 000	14 000	Disagreeable, toxic
Stoddard solvent	130	800	500	370	300	6000	5000	Disagreeable
Tetrachloroethane	168	1580	5	29 900	45 000	480 000	790 000	Toxic
Tetrachloroethylene	166	1620	100	1400	2300	24 000	40 000	Disagreeable, toxic
Toluene (toluol)	92	870	100	2600	2300	44 000	39 000	Toxic
Trichloroethane	133	1440	100	1800	2600	30 000	45 000	Disagreeable, toxic
Trichloroethylene	131	1460	100	1800	2700	30 000	45 000	Disagreeable, toxic
Xylene (xylol)	106	880	75	3000	2700	50 000	46 000	Disagreeable

a. Atomic mass units.
b. Same as $g \cdot L^{-1}$ or $mg \cdot cm^{-3}$.
c. Ventilation design concentration, not to be identified with values of maximum acceptable concentration or threshold values employed in appraising conditions since all VDCs include a factor of safety.
d. Ventilation ratio (or dilution rate) is the ratio of the volume of air (m^3 or ft^3) to the volume or weight of solvent evaporated.
e. Dilution system is not recommended in this case.

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operation of this facility. Note that this type of calculation is valid only if the air contaminant is uniformly distributed at a relatively low concentration. Where the air contaminant is localized in high concentrations, more complex means of evaluating the hazard must be used.

For users of the English system, the preceding example could be stated as follows. Trichloroethylene is used in a room of $20 \times 20 \times 10$ ft. In an 8 h day, 5 gal are evaporated; there are two air changes per hour. The solution in English units is, for room volume, $20 \times 20 \times 10 = 4000$ ft^3; for ventilation rate, $2 \times 4000 = 8000$ ft$^3 \cdot$h^{-1}.

Rate of solvent evaporation = 5 divided by 8 equals, in United States units, 0.6 gal$\cdot$h^{-1} or 5 pt$\cdot$h^{-1}.

Ventilation ratio is 8000 divided by 5 is 1600 ft$^3 \cdot$pt^{-1}. Ventilation ratio according to Table 2 is 2700 m$^3 \cdot$L^{-1} or 17 times 2700 is 46 000 ft$^3 \cdot$pt^{-1}. At least 46 000 divided by 1600 = 29 times more ventilation is required.

Evaluation of Toxicology and Health Hazards of Materials

The toxicity of a material is not synonymous with its health hazard. Toxicity is the capacity of a material to produce injury or harm. Hazard is the possibility that a material will cause injury when a specific quantity is used under specific conditions. The key elements to be considered in evaluating a health hazard are the following.

1. How much of the material is needed to produce injury?
2. What is the probability that the material will be absorbed by the body to produce injury?
3. What protective equipment is in use?

Because toxicity is not a definite physical constant but rather the degree to which a substance will affect living cells under certain conditions, it can be measured only after recognizable changes have occurred following absorption. Some changes such as impaired judgment or delayed reaction time may be produced at levels too low to cause actual cell damage. Then too, toxicity depends on the dose, rate, means and site of absorption. Other pertinent factors include the ambient temperature and the working conditions, as well as the general state of health, individual differences, tolerance and diet of individual personnel.

Estimating Toxicity Values and Lethal Doses of Toxic Materials

The first attempts at estimating the toxicity of a substance are usually made on the basis of animal experiments. Data from these experiments are expressed as lethal doses (LD) in milligrams of substance per kilogram of body weight of the test animal. The commonly used expressions are the following: *MLD, minimum lethal dose,* the smallest dose that kills one of a group of test animals; *LD50, lethal dose for 50 percent,* the dose that kills one half of a group of test animals (usually ten or more); *LD100, lethal dose for 100 percent,* the dose that kills all of a group of test animals (usually ten or more). These doses may also be expressed as *lethal concentrations (LC)* for airborne toxic substances.

Substances can then be rated according to their relative toxicity as shown in animal experiments (Tables 3 and 4).[4-6] The probable lethal dose for humans is often estimated from animal tests. These ratings are based on the results of short term exposures only. It is possible in

TABLE 3. Combined tabulation of toxicity classes, after Roehrs and Center.[3]

Commonly Used Term	LD_{50} Single Oral Dose for Rats[a] (g$\cdot$kg^{-1})	4 h Vapor Exposure Causing 2 to 4 Deaths in Six-Rat Group (µL$\cdot$L^{-1})	LD_{50} Skin Exposure for Rabbits (g$\cdot$kg^{-1})	Probable Lethal Dose for Humans	
				SI	(English)
Extremely toxic	≤0.001	>10	≤0.005	50 mg (taste)	(1 grain [taste])
Highly toxic	0.001 to 0.05	10 to 100	0.005 to 0.043	4 cm^3	(1 tsp)
Moderately toxic	0.05 to 0.5	100 to 1000	0.044 to 0.340	30 cm^3	(1 oz)
Slightly toxic	0.5 to 5.0	1000 to 10 000	0.35 to 2.81	0.5 L	(1 pt)
Relatively nontoxic	5.0 to 15.0	10 000 to 100 000	2.82 to 22.6	1 L	(1 qt)
Practically nontoxic	>15.0	>100 000	>22.6	>1 L	(>1 qt)

a. Grams of dose per kilogram of rat.
b. Parts of vapor in million parts of air.

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actual, long term chronic exposure for a substance to prove highly toxic, even though short term exposure tests indicated a low order of toxicity. However, animal experiment data are difficult to interpret and apply to human exposures. Such data are valuable only as guides to be used in estimating the gross toxicity of a substance and as leads for further investigations.

NIOSH Evaluations of Exposure to Toxic Substances

In the United States, the Department of Health, Education and Welfare (HEW), the Occupational Safety and Health Administration (OSHA) and the National Institute for Occupational Safety and Health (NIOSH) conduct critical reviews of occupational hazards, prepare criteria documents, recommend standards of exposure and list toxic effects of chemical materials. Under no circumstances can the toxic dose values presented for chemical substances be considered as being definitive values for describing safe versus toxic doses for human exposure. Concentrations of chemical substances in the work environment that may be safely tolerated can be determined only by a critical evaluation of all available pertinent data by experienced investigators.

NIOSH special occupational hazard reviews analyze and document, from a health standpoint, the problems associated with a given industrial chemical, process or physical agent and recommend the implementation of engineering controls and work practices to relieve these problems. The evaluations pertain primarily to special alleged hazards, e.g., those with carcinogenic, mutogenic, teratogenic or other reproductive effects, although they may review other effects as needed.

The permissible exposure levels of hazardous substances that have been adopted by OSHA to provide a safe, healthful work environment for all persons are cited as Occupational Standards (OSHA). These are given in an annually updated *NIOSH Registry of Toxic Effects of Chemical Substances.* NIOSH Criteria Documents contain environmental and medical recommendations related to specific substances and processes.

Management and test personnel can use NIOSH published resources to determine probabilities of hazards with new test materials, interacting combinations of chemical materials and environmental hazards. In all cases of doubt, however, reference to experts in the field for consultation and guidance is recommended.

Limitations of Safety Warnings

This volume is limited to leak testing and endeavors to provide comprehensive and useful information and data on test techniques and applications. It is not possible, within its scope, to advise users of all potential hazards and toxic or dangerous substances. In this book, only partial information and warnings can be included, so workers and test personnel or management should look up more complete data in publications from NIOSH and other sources for complete information. Qualified assistance should

TABLE 4. Guidelines for evaluating acute[a] dosages differentiating relatively toxic from nontoxic substances taking into consideration the route of administration to experimental animals and the dose causing death[b]. After Hine and Jacobson[5] and NIOSH 78-104A.[6]

Species	Rectal Intraduodenum Intracervix ($mg \cdot kg^{-1}$)	24 h Inhalation Maximum ($g \cdot kg^{-1}$)	Skin ($g \cdot kg^{-1}$)	Intraperitoneal Intrapleural ($g \cdot kg^{-1}$)	Subcutaneous Intradermal Implant ($g \cdot kg^{-1}$)	Other Parenteral[c] ($g \cdot kg^{-1}$)	Unspecified Parenteral ($g \cdot kg^{-1}$)	Unreported ($g \cdot kg^{-1}$)
Frog, gerbil, hamster	2.5	1.0	1.4	1.0	5.0	0.75	1.0	2.5
Mouse, rat, squirrel	5.0[c]	2.0	2.8	2.0	10.0[c]	1.5	2.0	5.0
Bird, chicken, duck, guinea, pig, pigeon, quail, rabbit, turkey	10.0	4.0	2.8[c]	4.0	20.0	3.0	4.0	10.0
Cat, cattle, dog, goat, horse, monkey, pig, sheep	10.0	4.0	5.6	4.0	20.0	3.0	4.0	10.0

a. Applies to those substances for which acute or short-term toxicity characterizes the response, e.g, fast-acting substances, irritants, narcosis-producing substances, and most drugs. Does not apply to substances whose characteristic response results from prolonged exposures, e.g., silica, lead, benzene, carbon disulfide, carcinogens. Concentrations more appropriately characterizing the toxicity of long- or slow-acting substances are derived from nonacute toxicity studies.
b. Calculated from experimental data (Stokinger).
c. Intravenous, intramuscular, ocular, intracerebral, intratracheal, intraplacental, intravaginal, intrarenal.

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be sought from experts in safety, legal requirements, governmental regulations, safety engineering, health and medical practice, wherever the possibility of hazards may exist. Special reference should be made by leak testing personnel and supervision to applicable plant safety rules; to procedures used in case of accidents; to local, municipal, county, state and national laws and regulations; and to qualified safety and health agencies, organizations and experts for advice on health and safety. The warnings and precautions given in this book are based on experience in industry during application of leak tests. They do not foresee the possibilities and nature of potential future accidents, nor do they include the constantly changing identifications of toxic or hazardous substances included in publications of governmental and other health and safety agencies and organizations.

Precautions with Specific Fluids

Acetone and Other Ketones

Acetone and other ketones are typical solvents and metal cleaning compounds used widely in industry. Acetone (dimethyl ketone) is a very flammable liquid that should be handled and stored with precautions against fire and explosion. In spite of the large quantities of acetone used in industry and its high volatility, there are no known documented reports of serious industrial poisoning. Experimental work has shown that acetone is a narcotic. Overexposure will lead to moderate irritation of the eyes, nose and throat and to headache, stupor and a general feeling of oppression. The absorbed acetone is eliminated slowly and the symptoms are persistent. Contact with skin and eyes should be avoided by the use of protective clothing. In areas of vapor concentration, approved respiratory protective equipment should be used.

Precautions with Halogenated Hydrocarbons

Halogenated hydrocarbons are typically colorless volatile liquids with excellent organic solvent properties and are widely used. Hydrocarbons having only one or two halogens are usually flammable and less toxic than similar hydrocarbons with complete halogen substitution. Thermal decomposition of halogenated hydrocarbon vapors occurs and poisonous gases may be formed when they come into contact with a heat source, such as a red hot surface, flame or electric arc.

The most common halogenated hydrocarbons, arranged in increasing order of ability to produce narcosis, are vinyl chloride, methyl chloride, ethyl chloride, ethylene dichloride, ethyl bromide, carbon tetrachloride, dichloromethane (also called methylene chloride), methyl chloroform (also called 1,1-trichloroethane and 1,1,2-trichloroethane), trichloroethylene, methyl bromide, tetrachloroethylene (also called perchloroethylene), pentachloroethane and tetrachloroethane (see a dictionary of commercial chemicals). Tetrachloroethane is about 40 times as strong a narcotic as vinyl chloride. An acute exposure to the more narcotic of these compounds may result in unconsciousness for a surprisingly long period, with eventual recovery. Unconsciousness for eight weeks has been reported in a case of methyl bromide poisoning. It is to be noted that the preceding listing is not in the same order as the chronic toxicity of these halogenated hydrocarbons. Chronic toxicity due to low rates of exposure over long periods of time has been the more common problem in industry.

Tetrachloroethane, the most toxic of the common chlorinated hydrocarbons, has no particular warning signs or symptoms. It can produce extremely severe poisoning from continuous exposure to fairly low concentrations. Tetrachloroethane is a very dangerous compound because inhalation of it at a concentration barely perceptible by odor can lead to extensive injury. Carbon tetrachloride, methyl chloride, dichloroethylene and trichloroethylene show decreasing chronic toxicity in approximately that order. Introduction of a bromine or iodine atom into one of the halogenated hydrocarbons generally increases the toxicity as compared to that of the corresponding chlorine compound. In contrast, introduction of a fluorine atom generally reduces the toxicity as compared to that of the corresponding chlorine compound.

The methyl compounds, particularly methyl chloride and methyl bromide, are in a special class because of their delayed action. Minor symptoms may appear during an acute exposure to these compounds; severe symptoms may appear after a delay of several hours to several days.

Precautions with Carbon Tetrachloride

Carbon tetrachloride (CCl_4) is a halogenated hydrocarbon liquid that is colorless, nonflammable and has a characteristic odor. Synonyms for carbon tetrachloride include tetrachloromethane

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and perchloromethane. Carbon tetrachloride is used as a solvent, degreaser and chemical constituent and can act to remove the natural liquid cover of human skin. With repeated contact with the skin, it can lead to a dry, scaly, fissured skin condition known as dermatitis. Chronic poisoning including liver damage comes from long, continued absorption of fairly small amounts of carbon tetrachloride over a long period. Barrier creams, gloves, protective clothing and masks should be used as appropriate where exposure occurs. The major problem in prevention of injuries from carbon tetrachloride is that of prevention of inhalation of carbon tetrachloride solvent vapor. Oxidative decomposition by flame causes it to form phosgene (a poisonous gas) and hydrogen chloride, also a poisonous gas. Carbon tetrachloride is now prohibited in many instances.

Precautions with Fluorocarbon and Refrigerant Gases

Fluorocarbons are hydrocarbons containing fluorine; they may contain other halogens in addition to fluorine. Generally these compounds are colorless nonflammable gases. Decomposition of chlorine-containing fluoromethanes, caused by contact with an open flame or hot metal, produces hydrogen chloride, hydrogen fluoride, phosgene, carbon dioxide and chlorine.

The fluorocarbons are used primarily as refrigerants, leak testing tracer gases and fire extinguishers and in degreasing of electronic equipment. They have found wide use due to their relatively low toxicity and nonflammability. Trademarks including Freon®, Genetron® and Isotron® have been used for a number of fluorocarbons used in refrigeration. The fluorocarbon compounds may produce mild irritation in the upper respiratory tract, perhaps caused by their decomposition products. Dermatitis occurs only rarely from contact with these materials.

In the United States, the Environmental Protection Agency took action to essentially ban the chlorofluorocarbons in aerosol spray cans that release the chemicals to the atmosphere with each use of the can. The law itself is written in two parts, which are integrated. The first part is administered by the Food and Drug Administration. The second part, which covers penetrants, is administered under the Toxic Substances Control Act. The exact wording appears in Parts 712 and 762 of this act and in the Federal Register of March 17, 1978. The important wording appears in paragraph 762.12(a), as follows: "After December 15, 1978 no person may process any fully halogenated chlorofluoroalkane into any aerosol propelled article. . . ."

Prevention of Personnel Exposures to Halogenated Hydrocarbons

Because exposure of testing personnel to halogenated hydrocarbons is almost invariably by inhalation, the most valuable measures to prevent poisoning are enclosure and ventilation at the point where vapor is released. However, several of the chlorinated hydrocarbons are apparently much more toxic by skin contact than has been believed. Skin contact should therefore be avoided because of the probability that where there is skin contact there will also be a severe inhalation exposure.

Precautions with Aromatic Hydrocarbons

Aromatic hydrocarbons are widely used as solvents and chemical intermediates. The basic aromatic nucleus is benzene, C_6H_6. Because of its health hazards, benzene has been replaced as a commercial solvent by toluene and other less toxic compounds. Typically, the vapor of aromatic hydrocarbons causes central nervous system depression and other effects. Vapor is absorbed through the lungs and the liquid may be absorbed through the skin. Repeated and prolonged skin contact may cause defatting of the skin, which leads to dermatitis. Chronic benzene poisoning can be fatal.

Precautions with Methyl Alcohol

Methyl alcohol (CH_3OH) is a colorless, volatile liquid with a mild odor. It is used in synthesis of many chemicals and as an industrial solvent. Contact of methyl alcohol with the skin can produce mild defatting and a mild dermatitis that can be avoided by use of barrier creams and protective clothing. Methyl alcohol is virtually nonirritating to the eyes or upper respiratory tract at concentrations in air below 2000 $\mu L \cdot L^{-1}$; it is difficult to detect by odor at less than this level.

Methanol (methyl alcohol) poisoning is usually produced by swallowing the liquid or inhaling high concentrations of vapor in an enclosed area. The signs of poisoning include headache, nausea, vomiting, violent abdominal pains, aimless and erratic movements, dilated pupils, sometimes delirium and such eye symptoms as pain, tenderness on pressure and, occasionally, blindness. Direct action of the liquid or the vapor on the skin and mucous membranes may produce an irritation and inflammation.

One of the peculiarities of methanol poisoning is its exceptionally severe action

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on the optic nerve. About one half of all the serious cases of methanol poisoning result in some impairment of vision. This loss is usually permanent and may vary from dimness or blind spots scattered through the visual field to total blindness.

Precautions with Glycols and Glycol Derivatives

Glycols are dihydric alcohols, which are colorless, odorless liquids. Glycols are soluble in water and in alcohol, have high boiling points, have low freezing points and are used as solvents and antifreeze. These compounds have relatively low toxicity and the major hazard appears when the liquids are heated during processing.

Precautions with Ethylene Glycol Ethers

Ethylene glycol ethers are only mildly irritating to the skin. Vapors may cause conjunctivitis and irritation of the upper respiratory tract. Temporary corneal clouding may also result and may last several hours. Acetate derivatives cause greater eye irritation than the parent compounds. The butyl and methyl ethers may penetrate the skin readily. Symptoms from repeated overexposure to glycol ether vapors are fatigue and lethargy, headache and tremor. Glasses and protective clothing can be used to prevent skin absorption. Respiratory protection maybe needed if ventilation is poor or glycol compounds are heated or atomized.

Precautions with Petroleum Derivatives

Naphtha is a rather indefinite term for any one of a number of solvent mixtures derived from petroleum. One should define it more carefully before attempting to assess the hazard. The naphthas are irritating to the skin, conjunctiva and mucous membranes of the upper respiratory tract. Skin chapping and photosensitivity may develop after repeated contact with liquid naphtha. If confined by clothing against the skin, the naphthas may cause skin burn. Workers should use barrier creams, protective clothing, gloves and masks where exposure to naphtha vapor is likely. Sufficient quantities of naphtha cause central nervous system depression. Symptoms include inebriation, followed by headache and nausea. In severe cases, dizziness, convulsions and unconsciousness may result. If benzene is present, coal tar naphthas may produce leukemia.

Precautions with Stoddard Solvent

Stoddard solvent is a registered commercial standard of the U.S. Department of Commerce for a dry cleaning solvent. Its specifications are that it has a flash point of 37.8 to 43.3 °C (100 to 110 °F), evaporates without residue and consists of aliphatic, saturated materials and, in some formulations, 15 to 20 percent aromatics. The fire hazard is about that of kerosene. It is available under a number of trade names.

Precautions with Toluene

Toluene is seldom a source of acute poisoning, although its inherent acute toxicity is somewhat higher than that of benzene. It is a flammable, colorless liquid of rather strong aromatic odor that serves somewhat as a warning of high concentration. At concentrations of 500 to 1000 $\mu L \cdot L^{-1}$, toluene is strongly irritating to the eyes and respiratory system. In higher concentration, it is a narcotic and the signs of acute poisoning are headaches, drunkenness, nausea, vomiting and ultimately unconsciousness. Toluene does not appear to produce the severe and often fatal depression of the blood forming organs seen in chronic benzene poisoning. In case of acute exposure to toluene, the person should be taken to fresh air as soon as possible. Oxygen should be given and, if breathing has stopped, artificial respiration should be administered immediately. A physician should be called at once.

Precautions with Trichloroethylene

Trichloroethylene is a halogenated hydrocarbon used primarily as a degreasing compound. It has no flash point as such, but at elevated temperatures and with a high energy ignition source, such as a welding arc, its vapors can and will explode. Toxic decomposition products, mainly hydrogen chloride with some phosgene, both highly poisonous gases, may also be formed under these conditions. Phosgene may be formed inside a cigarette when smoking in an area where trichloroethylene vapors are present.

Trichloroethylene may have a depressant action or, as with other chlorinated hydrocarbons, cause alteration of the heart rhythm, or lead to addiction. Although some absorption may occur through the skin, trichloroethylene has mainly a defatting and dermatitis-producing skin effect.

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Precautions with Xylene

Xylene, $C_6H_4(CH_3)_2$, is a mixture of isomers and may contain numerous other solvent compounds. It is used as a solvent and is specified in some tests to detect the water content of penetrant materials. Xylene vapor may cause irritation to the eyes, nose and throat. Repeated or prolonged skin contact may cause drying and defatting of the skin, which may lead to dermatitis. Liquid xylene is irritating to the eyes and mucous membrane. Aspiration of a few milliliters may cause severe effects. Repeated exposure of the eyes to high concentrations of xylene vapor may cause irreversible eye damage.

When xylene vapor concentrations exceed allowable standards, full face masks with organic vapor cannisters or air supplied respirators should be furnished. Impervious protective clothing and gloves should be worn by personnel exposed to liquid xylene. Xylene wet clothing should be changed quickly. Goggles or safety glasses are advised. Barrier creams may be useful.

Hazards of Oxygen Deficient Atmospheres

Oxygen deficiency designates an atmosphere having less than the percentage of oxygen found in normal air. Normal air contains about 21 percent oxygen at atmospheric pressure. When the oxygen concentration in air is reduced to approximately 16 percent, many individuals become dizzy, experience a buzzing in the ears and have a rapid heartbeat.

In addition to tests for toxicity, the oxygen content of the atmosphere of a vessel or similarly confined space suspected of being oxygen deficient should be determined by preentry and subsequent tests made with instruments approved for the purpose by the United States Bureau of Mines. No one should enter or remain in a vessel or enclosed space that tests show has less than 16 percent oxygen in its atmosphere at any time unless wearing approved respiratory protective equipment such as a fresh air hose mask or self-contained or self-generating breathing apparatus. Various types of self-contained compressed air breathing apparatus, approved by the U.S. Bureau of Mines, have proved satisfactory in oxygen deficient atmospheres. They are especially useful where it is difficult to run an air supply hose line.

Precautions with Krypton-85 Gas

Krypton-85 gas is used in leak test pressurization systems in concentrations near 0.01 percent in nitrogen or air. The two toxological effects from this inert gas are asphyxiation and radiation exposure.

To satisfy federal and state licensing requirements in the United States, the pressurization systems are provided with a room enclosure; an exhaust system for typically 3 to 5 min room air exchange; a series of interlocking safety circuits for the proper exhaust air flow; and the detection of any radioactive gas in the room or exhaust.

The regulatory agencies monitor and enforce these requirements as well as continuous monitoring film badges to document worker and room exposure levels. A total dump of a typical krypton-85 leak testing system would require immediate operator evacuation of the machine enclosure, which would typically result in *nondetectable* radiation exposure as measured on state-of-the-art film badges.

Precautions with Dry Powder Developers

Dry powder developers as used in some liquid leak tracers are subject to dusting and other behavior characteristics of dry powder materials. Safety procedures such as the following should be observed.

1. Avoid continued excessive inhalation.
2. Use a well fitting dust mask and adequate ventilation.
3. Wear eye protection when filling or emptying a hopper.
4. Any dry powder material can build static electricity charges when subjected to the friction of mixing, sliding or conveying. Proper precautions such as adequate electrical grounding of equipment and not having flammable liquids in the area should be taken.

For further information, refer to NFPA 77-1993, *Recommended Practice on Static Electricity*.[7]

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PART 3. Flammable Liquids and Vapors

Definition of Terms Characterizing Flammable Liquids and Vapors

Flammable liquids are usually subdivided into classes. As defined by the National Fire Protection Association, a flammable liquid is any liquid having a flash point below 60 °C (140 °F) and having a vapor pressure not exceeding 275 kPa absolute (40 $lb_f \cdot in.^{-2}$) at 37.8 °C (100 °F).

Combustible liquids are those with flash points in the range of 60 to 93 °C (140 to 200 °F). Although they do not ignite as easily as flammable liquids, they can ignite under certain circumstances and so must be handled with caution. The more common flammable and combustible liquids are various hydrocarbons, alcohols and their byproducts. They are chemical combinations of hydrogen and carbon; the combination may also contain oxygen, nitrogen, sulfur and other elements.

Factors Influencing Hazards of Flammable Liquids

Flammable liquids vaporize and form flammable mixtures when they are in open containers, when leaks or spills occur or when the flammable liquids are heated. The degree of danger depends on the following: (1) the flash point of the liquid, (2) the concentration of vapors in the air (whether the mixture of vapor and air is in the flammable range) and (3) the possibility of an ignition source at or above a temperature sufficient to cause the mixture to burst into flame.

Precautions for Flammable Liquids

In the handling and use of flammable liquids, exposure of large liquid surfaces to air should be prevented. It is not the liquids themselves that burn or explode, but rather the vapor-and-air mixture formed when liquids evaporate. Therefore, flammable liquids should be handled and stored in closed containers. Low flash liquids in use should be covered or enclosed to avoid evaporation into the atmosphere. In both cases, the fluids should be enclosed wherever feasible. When the fluid is exposed to air for a specific operation, it should again be covered or enclosed as soon as possible.

Flash Point of a Flammable Liquid

The flash point of a liquid is the lowest temperature at which it gives off enough vapor to form flammable mixtures with air and to produce a flame when a source of ignition is brought close to the surface. Other properties are factors in determining the hazards of flammable liquids, but the flash point is the principal factor. The relative hazard increases as the flash point is lowered. The significance of this property becomes more apparent when liquids of different flash points are compared.

Examples of Flash Points of common Liquid Fuels

Kerosene and number 1 fuel oil have flash points of about 43 to 74 °C (110 to 165 °F) but ASTM D 396, *Specification for Fuel Oils*,[8] will permit a flash point as low as 38 °C (100 °F) for number 1 fuel oil. At ordinary room temperatures of 22 °C (72 °F), these oils do not give off dangerous quantities of vapor. On the other hand, gasoline gives off vapor at a rate sufficient to form a flammable mixture with air at temperature as low as –45 °C (–50 °F).

Any flammable liquid, when heated to a temperature above its flash point, can produce vapors in sufficient quantity to produce an explosive mixture in the air. For example, when heated, heavy fuel oil may produce flammable vapors just as readily as gasoline does at –20 °C (–4 °F).

Autoignition Temperature

Autoignition temperature is the lowest temperature at which a flammable gas or vapor-and-air mixture will ignite under defined conditions without an external source of ignition. Flammable vapors and gases in oxygen will spontaneously ignite at a lower temperature than in air and their autoignition temperature may be influenced by the presence of catalytic substances.

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Flammability Limits of Vapor Concentrations

Flammable liquids have a minimum concentration of vapor in air below which propagation of flame does not occur on contact with a source of ignition. There is also a maximum proportion of vapor or gas in air above which propagation of flame does not occur.

The extremes of vapor or gas concentration with air which, if ignited, will just propagate flame, are known as the lower and upper flammable limits. These are usually expressed in terms of percentage by volume or weight of gas or vapor in air. These limits are also commonly referred to as, respectively, the lower and upper explosive limits.

A mixture with less than about 1.0 percent by weight of gasoline vapor is too lean and propagation of flame will not occur on contact with a source of ignition. Similarly, if there is more than about 8 percent of gasoline vapor, the mixture will be too rich. Other gases such as hydrogen, acetylene and ethylene have a wider range of flammable limits.

Flammability Ranges (Explosive Range)

Flammable range is the difference between the lower and upper flammable limits, expressed in terms of percentage by volume of vapor or gas in air. It is also often referred to as the explosive range. For example, the limits of the flammable range of gasoline are generally taken as 1.4 to 7.6 percent, which is relatively narrow. Thus, a mixture of 1.4 percent gasoline vapor and 98.6 percent air is flammable, as are all the intermediate mixtures up to and including 7.6 percent gasoline vapor and 92.4 percent air. The range is the difference between these limits, or 6.2 percent.

Effects of Diffusion Rate, Vapor Pressure and Volatility

Rate of diffusion is the tendency of one gas or vapor to disperse into or mix with another gas or vapor. This rate depends on the density of the vapor or gas as compared with that of air. Whether a vapor or gas is lighter or heavier than air determines to a large extent the means of solving ventilation problems.

Vapor pressure is the partial pressure (in kilopascal or in $lb_f \cdot in.^{-2}$) exerted by the vapor of a volatile liquid, when in equilibrium with the surface of the liquid, as determined by standard ASTM D 323, *Test Method for Vapor Pressure of Petroleum Products (Reid Method)*.[9]

Volatility is the tendency or ability of a liquid to vaporize. Such liquids as alcohol and gasoline, because of their well known tendency to evaporate rapidly, are called volatile liquids.

Boiling Points of Flammable Liquids

The *boiling point* of a liquid is that temperature at which the vapor pressure of the liquid equals the atmospheric pressure. Increasing the liquid temperature causes vapor to be given off more readily. Liquids with low boiling points generally volatilize more readily than those with higher boiling points. However, there is not consistent relationship between boiling point and evaporation rate.

Definitions for Vapor Volume and Evaporation Rate

Vapor volume is the number of liters of solvent vapor formed by evaporation of 1.0 L of liquid at standard temperature (20 °C). In English units, the vapor volume is the number of cubic feet of solvent vapor formed by the evaporation of 1 gal (imperial or United States gallon), of a liquid at 68 °F. One can always find vapor volume by using the mole (an amount of gas or liquid whose weight in grams equals its molecular weight.) This number of grams, equal to the molecular weight of the substance (at 0 °C and 101.3 kPa), evaporates to 22.4 L at standard temperature and pressure.

Evaporation rate is the ratio of time required to evaporate a measured volume of liquid to the time required to evaporate the same volume of a reference liquid under ideal test conditions. The higher the ratio, the slower the evaporation rate.

Containers for Flammable Liquids

Portable containers should be provided with flame arresters installed in the vent or opening. If a number of different flammable liquids are handled, safety cans should have distinct stripes, or identification lettering should be placed on them so as to reserve certain cans for their respective liquids and to help reduce the chance of the liquids being mixed. Safety can caps should be regularly inspected for proper operation and sealing.

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Restriction of Smoking and Lighters in Flammable Material Areas

Smoking and carrying of lighters, strike-anywhere matches and other spark producing devices should be prohibited in buildings or areas where flammable liquids are stored, handled or used. The extent of the restricted area will depend on the type of products handled, the design of the building design, local conditions and compliance with local, state and federal regulations for flammable material areas.

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PART 4. Electrical and Lighting Hazards

Hazards of Static Electricity with Flammable Materials

Static electricity is an accumulation of motionless charges generated by the contact and separation of dissimilar materials. For example, static electricity is generated when a fluid flows through a pipe or from an orifice into a vessel and may set up high voltages. The principal hazards created by static electricity are those of fire and explosion caused by spark discharges occurring in the presence of flammable or explosive vapors, gases or dust. A spark between two bodies occurs when there is no good electrical conductive path between them. Hence, grounding and bonding of flammable liquid containers is necessary to prevent static electricity from causing a spark.

Avoidance of Sources of Ignition of Flammable Gases and Vapors

When using potentially explosive gases, the test area should be free from obvious sources of ignition. Smoking should be prohibited and signs should be posted to warn of the hazards. Electrical equipment may also present a problem. If there is a possibility that, in the event of leakage, such equipment will be in an explosive environment, then either the equipment should be repositioned outside the danger area or specifically chosen equipment should be used.

Although hydrogen presents the most severe risk, the above precautions are also relevant if other flammable tracer gases are used. When large components are tested, or when large volumes of hydrogen are used, it may be advisable to provide monitors that give a continuous indication of the hydrogen and air content in the test area. Intrinsically safe detectors are available. Gas monitoring may also be advisable when high vacuum vessels are being chemically cleaned before evacuation. Cleaning techniques often include washing with benzene, acetone or alcohol. The interior of the vessel as well as the environment may contain an explosive mixture. Extreme precautions should be taken when using these materials.

A discharge of static electricity is a possible cause of ignition, so all metal parts likely to become charged should be grounded. When testing with gases such as hydrogen, it would also be sensible for personnel to avoid wearing clothing that might produce static charges and for them to wear shoes with conducting soles. Another precaution is the use of reduced sparking or nonsparking tools.

Bonding and Grounding to Prevent Electric Sparks

A point of great danger from a static spark is the place where a flammable vapor may be present in the air, such as at the outlet of a flammable liquid fill pipe or a delivery hose nozzle. Static spark ignition sources are prevented by bonding or grounding or both so they have the same static voltage or potential.

The terms bonding and grounding often have been used interchangeably because of poor understanding of the distinct functions indicated. Bonding is done to eliminate a difference in potential between objects. The purpose of grounding is to eliminate a difference in potential between an object and ground. Bonding and grounding are effectively applied only to conductive bodies. The human body is a conductive body that may differ in potential from ground or other bodies, so that it may also serve as a source of spark ignition.

Although bonding will eliminate a difference in potential between the objects that are bonded, it will not eliminate a difference in potential between these objects and the earth unless one of the objects possesses an adequate conductive path to earth. Therefore, bonding will not eliminate the static charge but will only equalize the potential between the objects bonded.

Electrical Power Hazards

Electricity as a source of power is, in some ways, less hazardous than steam or other energy sources. However, failure to take suitable precautions in its use creates conditions that are certain to result in bodily harm or property damage or both. Although there have been advances in the

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control of electrical hazards, industry still has many injuries and fatalities from preventable causes. Machine tools can, with minimum expense and difficulty, be arranged for maximum safety and efficiency. There are, however, certain hazards in the installation, maintenance and use of electric wiring and equipment. Control of most of these hazards is neither difficult nor expensive, but ignoring or neglecting them may lead to serious accident.

Electrical Injury and Fatal Levels of Body Current

Current flow is the factor that causes injury in electric shock. The severity of electric shock injury is determined by the amount of current flow through the victim. Experimental and field data from authoritative sources indicate that, in general, an alternating current of 0.1 A at commercial frequency (60 Hz) may be fatal if it passes through the vital organs. Similarly, it is estimated that a current value of 0.02 A is the limit at which an individual can still release himself from an object held by the hand. Such current flow may readily result from body contact with low voltage sources of ordinary lighting or power circuits.

Limiting Current Flow to Human Body

Because current flow depends on voltage and resistance, these factors are important. Other factors affecting the amount of injury are the parts of the body involved, the duration of current flow through the victim and the frequency with alternating current. Resistance to current flow is mainly to be found in the skin surface. Callous or dry skin has a fairly high resistance, but a sharp decrease in resistance takes place when the skin is moist. Once the skin resistance is broken down, the current flows readily through the blood and body tissues. Grounding conditions often determine resistance to current flow from the human body to earth or grounded structures.

Whatever protection is offered by skin resistance decreases rapidly with increase in voltage. High voltage alternating current at 60 Hz causes violent muscular contraction, often so severe that the victim is thrown clear of the circuit. Although low voltage also results in muscular contraction, the effect is not so violent. The fact, however, that low voltage often prevents the victim from freeing himself from the circuit makes exposure to it dangerous.

Effects of Electric Current on Human Body

Death or injury by electric shock may result from the following effects of current on the body.

1. Electric current may cause contraction of the chest muscles, which may interfere with breathing to such an extent that death will result from asphyxiation when the exposure is prolonged.
2. Electric current may cause temporary paralysis of the nerve center, which may result in failure of respiration, a condition that often continues until long after the victim is freed from the circuit.
3. Electric current may interfere with normal rhythm of the heart, causing ventricular fibrillation. In this condition, the fibers of the heart muscles, instead of contracting in a coordinated manner, contract separately and at different times. Blood circulation ceases and death ensues, because apparently the heart cannot spontaneously recover from this condition. It has been estimated that 0.1 A flowing through the body cavity (chest) is sufficient to cause ventricular fibrillation.
4. Electric current may suspend heart action by muscular contraction (on contact with heavy current). In this case, the heart may resume its normal rhythm when the victim is freed from the circuit.
5. Electric current may cause hemorrhages; destruction of nerves, muscles or other tissues; or extensive skin burn from heat due to heavy current or electric arcs.

In general, the longer the current flows through the body, the more serious may be the result. Considerable current is likely to flow from high voltage sources and in general only very short exposure can be tolerated if the victim is to be revived.

Injuries from electric shock are less severe when the current does not pass through or near nerve centers and vital organs. In most electric accidents in industry, the current flows from hands to feet. Because such a path involves both the heart and the lungs, results are usually serious.

Treatment of Victims of Electric Shock

Statistics indicate that only a small percentage of those who recover from electric shock show permanent disability.

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In many cases, the victim may be saved by prompt application of cardiopulmonary resuscitation because a common result in electrical accidents is failure of that part of the nervous system that controls breathing. Therefore, it is essential that persons working with electrical power equipment be instructed in the modern technique of mouth-to-mouth or mouth-to-nose resuscitation and cardiopulmonary resuscitation as developed by the American Heart Association. Immediate treatment should be applied to victims of electric shock and should be continued until they revive or until death is diagnosed by a physician or until rigor mortis sets in.

Hazards of Electric Arcs

Another type of injury is burns from electric arc flashes or from human contact with energized electric power equipment. Such burns are usually deep and slow to heal and may involve large areas of the body. Even welding arcs are also sources of arc flashes. Hot weld metal, welding slag and electrode stub ends can produce severe burns if touched. Side shielded safety glasses, glasses that do not transmit ultraviolet radiation and proper use of welding helmets all help avoid welding arc flash injuries to the eye.

Hazards of Electrical Extension Cords

Extension cords should be of a type listed by the Underwriter's Laboratories and should be labeled to show that they meet all requirements of the *National Electrical Code*.[10] They should be inspected regularly. Kinking or excessive bending of the cord should be avoided to prevent the wire strands from breaking. Broken strands may pierce the insulated covering and become a shock or short circuit hazard. Old insulation on extension cords often becomes brittle and creates a shock or short circuit hazard. Ordinary twisted lamp cord should never be used for extension cords or lamps in vessels or on damp or metallic floors and should never be used where it will be exposed to mechanical wear.

Cord for use with portable power tools and equipment is made in several grades, each of which is designed for a specific type of service. Rubber sheath cord should be used with portable electric tools and with extension lamps in vessels or other grounded enclosures. Special types of synthetic rubber or plastic covering should be considered when the cord is to be used in areas where it may come in contact with oils or solvents. Double insulated electrical tools should be selected for maximum safety.

Explosion-Proof Electrical Fittings

When using potentially explosive gases, the test area should be free from obvious sources of ignition. Smoking should be prohibited and signs should be posted to warn passersby of the hazards. Electrical equipment may also present a problem. If there is a possibility that, in the event of leakage, such equipment will be in an explosive environment, then either the equipment should be repositioned outside the danger area, or else specifically chosen, safe, explosion-proof equipment should be used.

Standard electrical fittings, considered safe for ordinary application, are obviously unfit for installation in locations where flammable gases and vapors or other easily ignitable flammable materials are present. Sparks and electric arcs originating within electrical switches and fittings have been the igniting medium in costly fires and explosions.

Selection of Electrical Fittings for Hazardous Locations

Before fittings are selected for a hazardous location, it is necessary to determine the exact nature of the flammable materials present. For instance, an electrical fitting, found by test to be safe for installation in an atmosphere of combustible dust, may be unsafe for operation in an atmosphere containing flammable vapors or gases.

It is impossible to prevent highly flammable gases from entering the interior of either an explosion resistant or an ordinary wiring system. They will eventually enter the entire line through the joints and through the breathing of the conduit system caused by temperature changes. Furthermore, gaseous vapors will fill every crevice whenever covers are removed. For these reasons, it is impossible to provide an entirely vapor-proof switch unit or to regulate temperatures or keep the air free from flammable gases inside the electrical fittings.

To protect that area classified as a hazardous location, it is necessary to have positive confinement of the arc, heat and explosion within the internal limits of explosion-proof fittings. These fittings are constructed to completely imprison the dangerous arcing, intense heat and subsequent explosion so that the gas laden air outside does not become ignited.

Protective Enclosures for Electrical Apparatus

A useful substitute for explosion-proof equipment is to enclose nonexplosion-

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proof apparatus in metal boxes and pass a stream of nitrogen or even air into the box to maintain it slightly above atmospheric pressure. However, it should be kept in mind that all equipment can be hazardous and should only be used with due regard to the hazards involved. Put only that equipment in the test area that must be there. Where possible, use air operated equipment instead of electrical equipment.

It is necessary that electrical equipment be explosion-proof throughout the entire building and not solely in the test area, in cases where explosive vapors may travel to other parts of the building should a leak occur. Under some circumstances, the test area can be sealed to prevent escape of vapors to other areas.

Lighting as a Factor in Industrial Safety

The proportion of industrial accidents attributable to poor lighting has been estimated to be from 15 to 25 percent. Good lighting contributes greatly to safety, as well as increasing efficiency and morale. Daylight is an ideal type of illumination. For the most effective use of daylight, a definite relationship of floor to window must be maintained. Sudden transitions from brightly lighted to dim areas and vice versa are dangerous; the result is momentary blindness due to the lag in eye accommodation. Gradations of light between areas of different intensities will remedy this difficulty.

Artificial Lighting

Artificial lighting has become so accepted as an element of modern life that its original supplementary character has been largely forgotten. Artificial lighting has become the major source of illumination because natural light is undependable, especially in the winter when work schedules do not coincide with daylight. For continuous shift operation, artificial light is essential. For other types of operation, it must be relied on from 20 to 50 percent of the total working hours, excluding overtime work or night work.

General Lighting

General lighting is the base or minimum amount of light required. It has been defined as uniform distribution of light to produce equivalent seeing conditions throughout an interior. Localized general lighting sources usually are arranged 3 m (about 10 ft) or more above the work to prevent too great a contrast in brightness between the more highly lighted work area and the adjacent areas and to provide sufficient light for general safety and for ordinary visual needs.

Light intensity on a surface varies inversely with the square of the distance between the surface and a small area source of light. A source 3 m (about 10 ft) above a surface would give four times more light to the work area than would the same source 6 m (about 20 ft) high. Where visual needs are more critical, additional lighting can thus be provided by fixtures placed fairly close to the area needing more light.

Precautions with Ultraviolet Sources Used for Inspection with Fluorescent Leak Tracers

Fluorescent penetrants and leak tracers require intense illumination with ultraviolet and near ultraviolet radiation sources to make test indications visible. Properly enclosed, shielded and filtered ultraviolet radiation sources used for inspection emit radiation in the 320 to 400 nm wavelength range, well above the more hazardous shorter wavelength ranges of hard ultraviolet radiation. Failure to use proper filters and lamp enclosures could permit such hard ultraviolet radiation from mercury vapor arc lamps, welding arcs and fluorescent tubular ultraviolet lamps to escape. The following discussion lists hazards and precautions for control of ultraviolet radiation and notes its physiological effects.

Effects of Hard Ultraviolet Radiation

Hard (short wavelength) ultraviolet radiation has long been known to produce physical, chemical and physiological effects, so some evaluation of these effects and the degree of hazard involved in ultraviolet radiation is in order.

Physically, ultraviolet radiation is the portion of the electromagnetic spectrum with wavelengths between those of visible light and X-rays. Therefore, as might be expected, the long wave portions behave very much like visible light and the short wave portions have some of the properties of X-rays. The middle ranges have properties of their own that are not common to other portions of the spectrum.

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Filters for Transmission or Absorption of Ultraviolet Radiation

Ultraviolet radiation can be transmitted, absorbed and refracted or bent just like visible light, although usually by substances other than those normally used for visible light. For instance, ordinary window glass transmits quite well in the longer wavelengths, but becomes opaque to wavelengths shorter than 310 nm. Thus, it will transmit ultraviolet radiation but absorb the shorter, more harmful wavelengths. Therefore, ordinary glass is a good protective shield against hard ultraviolet radiation.

A number of suitable filters and glass types will remove all ultraviolet radiation while permitting visible light to pass. In cases where short wave ultraviolet radiation must be transmitted, special glasses are available. Some glass will transmit wave lengths as short as 280 nm and other glass to 230 nm. Below this point, quartz, particularly in the crystalline form, must be used.

Reflection of Ultraviolet Radiation

Ultraviolet can also be reflected, but often by materials different from those used to reflect visible light. Most white metals reflect ultraviolet radiation although not as strongly as they reflect visible light. Silver is an exception, reflecting to about 360 nm, with absorbing shorter wavelengths. Aluminum and polished iron are good ultraviolet reflectors. Some white pigments such as magnesium oxide, aluminum oxide and calcium carbonate are good reflectors, whereas others such as titanium dioxide and zinc oxide are poor ultraviolet reflectors. Dark visible colors, particularly greens, browns and reds, are usually poor reflectors and good absorbers of ultraviolet. These factors should be kept in mind when designing ultraviolet radiation inspection booths.[11]

Chemical Reactions Excited by Hard Ultraviolet Radiation

Ultraviolet radiation is also chemically active and accelerates many reactions. Of particular importance are oxidation and molecular breakdown. Oxidation is a primary cause for the breakdown of paint vehicles and the fading of dyes and other colorants. Powerful ultraviolet will also bread down or otherwise alter many molecules even without the presence of oxygen, so oxidation is not the only chemical action it produces. Ultraviolet radiation, being at the long wave end of the range, is probably only slightly more chemically active than visible light. However, as with visible light, long exposure to high intensity ultraviolet radiation can be expected to have its effect.

Chemical Reactions Caused by Ozone

Hard (short wave) ultraviolet radiation also produces ozone, which itself is a strong oxidant. Ozone (O_3) is a bluish gas with a characteristic pungent odor and is found naturally in the atmosphere as a result of the action of solar radiation and lightning in electrical storms. It is also formed in corona discharges around high voltage conductors and is generated by X-ray and ultraviolet radiation, electric arcs (including welding arcs), mercury vapor lamps and linear accelerators.

Physiological Effects of Hard Ultraviolet Radiation

Physiologically, ultraviolet radiation can produce a variety of effects, depending strongly on the wavelength. Short wave ultraviolet radiation, as previously stated, produces ozone. Ozone is a very toxic compound that may cause death due to lung congestion and edema. Its maximum allowable concentration is 0.1 part per million (0.2 $mg \cdot m^{-3}$). Ozone is produced essentially at wavelengths below 260 nm. The properly filtered mercury arc ultraviolet radiation sources used with fluorescent leak tracers do not produce ozone.

Ultraviolet radiation also has a germicidal effect and is used for sterilization. This effect reaches a maximum at 260 nm and falls off rapidly to nearly zero at 320 nm. The action is effective on almost all bacteria as well as some fungi and molds. Thus, ultraviolet radiation is a very useful tool for disinfecting surfaces as well as room air while it passes through enclosed ventilating systems. Sterilizing lamps should not be placed where human eyes or skin can be exposed to their radiation.

Skin Inflammation Caused by Ultraviolet Radiation

Another well known effect of ultraviolet radiation is the production of erythema or skin inflammation, commonly known as

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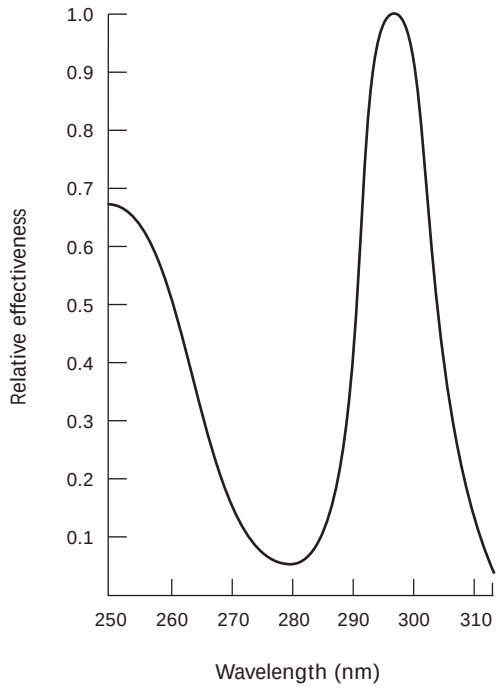
sunburn. This effect is produced strongly by certain wavelengths and not at all by others, as shown in Fig. 1. Thus, the short germicidal wavelengths produce considerable inflammation, whereas certain middle wavelengths are relatively ineffective. For those using ultraviolet radiation for inspection, the important fact is that there is essentially no erythemal effect above 320 nm. Because the light used for inspection is essentially 365 nm, inspectors do not become sunburned from their work with properly filtered and shielded black lights.

One of the serious concerns about possible effects of any radiation is its tendency to produce cancer. The United States government in its role as a consumer protection agency has conducted studies on carcinogenic and other health hazards of ultraviolet radiation. This work is summarized in the document *Criteria for a Recommended Standard for Occupational Exposure to Ultraviolet Radiation,*[12] which concludes that cancer can be produced by long exposure to sunlight rich in midrange ultraviolet. However, the cancer producing effect is directly proportional to the erythemal or sunburn effect. Therefore, the ultraviolet radiation used for inspection purposes is not a probable cause of cancer.

FIGURE 1. Standard curve for erythemal effectiveness of various wavelengths of hard ultraviolet radiation. Note that radiation used in inspection with fluorescent leak tracers lies in the range of 360 nm and above and does not have significant hazardous effects.



Eye Irritation Caused by Ultraviolet Radiation

Eye irritation is one further physiological effect due to ultraviolet radiation. There are two types of irritation. The first is a bluish haze noted when the eyes are exposed to ultraviolet, particularly of the longer wavelengths. This is irritating, causing headaches and, in extreme cases, nausea but is otherwise not harmful. It is caused by fluorescence of certain portions of the eye when exposed to ultraviolet radiation.

The second type of irritation is photokeratitis followed by conjunctivitis. This is essentially snow blindness. It includes a feeling of sand in the eyes, allergy to light, tear formation and finally blindness. These symptoms usually begin 6 to 12 h after exposure and last from 6 to 24 h, with all symptoms disappearing in 48 h. There is not cumulative effect but, on the other hand, no tolerance is developed from repeated exposure as is the case with sunburn. This effect is caused only by the wavelengths shorter than 310 nm and so should be no problem in inspection operations as long as the light is passed through the normal filters.

Protective Glasses to Shield Eyes from Ultraviolet Radiation

Should eye irritation be a problem, yellow tinted eye glasses will offer complete protection, particularly if equipped with side shields. Such glasses are sold as shooter's glasses and can be provided by local oculists.

Recommended Limits for Personnel Exposure to Ultraviolet Radiation

In *Criteria for a Recommended Standard for Occupational Exposure to Ultraviolet Radiation,*[12] recommended limits for personnel exposure to ultraviolet radiation in the 314 to 400 nm range were listed as 1.0 $mW \cdot cm^{-2}$ for exposures exceeding 1000 s and 100 $mW \cdot cm^{-2}$ for exposures under 1000 s (about 16 min).

The OSHA Environmental Standard was 10 $mW \cdot cm^{-2}$ over any 1 h period.

Recommended Limits for Exposure to Krypton-85 Gas

Although regulations in some of the United States vary in specific exposure limits, the *Code of Federal Regulations,* Title 10, Part 20,[1] sets standards for the

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allowable *cumulative annual exposure:* 1 mSv (100 mrem) for the general public, 50 mSv (5 rem) for the whole body, 150 mSv (15 rem) for the lens of the eye, 0.5 Sv (50 rem) for the skin and extremities and 5 mSv (500 mrem) for an embryo or fetus.

The prevailing industrial philosophy is that *any unnecessary exposure should be prevented*. The gamma radiation from krypton-85 has a 514 keV energy and represents 0.46 percent of the emission from krypton-85 gas. This is considered to be a very week photon with potential for very little tissue damage. The beta particle emitted by krypton-85 is quite weak and, when the gas is leaked into a hermetic device, the beta particles rarely can penetrate the walls of the device. In actual leak testing, the krypton-85 gas that has leaked into the device is measured by detecting the total radiation seen through the walls of the device using highly sensitive scintillation detectors.

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PART 5. Safety Precautions with Leak Testing Tracer Gases

Tracer Gas Hazards in Leak Testing

Tracer gas safety aspects such as flammability, asphyxiation or specific physiological effects as well as the possibility of pressure vessel explosions must be considered. So long as the nondestructive test engineer and the leak test technician are aware of these considerations from the start, it is possible to leak test a vessel with minimum inconvenience or danger.

Most tracer gases are not toxic. However, if a question exists about the toxicity of any particular gas, a competent authority should be consulted. Many tracer gases will not support human life. If such tracer gases replace oxygen in a vessel, this vessel cannot be entered without proper ventilation. In this case, proper ventilation consists of a gas mask that contains its own air-oxygen gas supply.

The oxygen required for breathing may be accidentally removed from an area. For example, if one of the halogenated hydrocarbons is used as a tracer gas, it may stagnate and settle to the lowest area. If a technician is attempting to use a detector probe in this low area, the tracer gas that settles may eventually displace enough of the air to produce asphyxiation. To avoid this condition, adequate ventilation must be provided. However, this ventilation must be performed carefully. If the tracer gas is removed too rapidly from the place where it is escaping from the vessel, leakage location may be difficult.

To aid in a better understanding of the safety aspects, the following data are presented below for several tracer gases that may be used. In addition, information is given on the availability of personnel protection indicators and area contamination monitors that can provide warning indications of dangerous accumulations of toxic gases or vapors.

Personnel Protection Badges to Warn of Excessive Exposure to Toxic Gases

Personnel protection indicators (PPIs) are plastic badges with pocket clips that have sensors that react chemically with concentrations of various gases or vapors used as tracers in leak testing. They provide forewarning of excessive exposure to the toxic substances by means of color changes, as listed in Table 5. These personnel protection indicators are sensitive to the accumulated personal exposure of the badge wearer to the concentration of gas in the leak testing area. The Occupational Safety and Health Administration of the United States defines the critical exposure period to be an 8 h shift. A color change of the protective badge at any time during an 8 h shift indicates that the badge wearer has received his or her maximum safe exposure. Table 5 lists the concentrations of toxic gas or vapor in air, which are designated as the critical accumulations. Also listed in Table 5 are the color changes that occur on exposure of personnel protection badges to the specific tracer gases for which they are sensitive.

Although the personnel protection indicator badges are normally worn on

TABLE 5. Selection guide for personnel protection indicators for toxic gases and vapors accumulating in leak testing areas. Data apply to both personnel protection and area contamination monitors.

Toxic Substance	Warning Concentration ($\mu L \cdot L^{-1}$)	Color Change
Ammonia	15	Brown to white
Carbon monoxide	50	White to Black
Chlorine	2	White to yellow
Hydrazine	5	White to yellow
Hydrogen sulfide	5	White to brown
Nitrogen dioxide	1	White to yellow
Ozone	0.1	White to brown

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breast pockets so supervisory personnel and coworkers can easily see the status of the indicator, a person working alone can monitor his or her own status more easily by clipping the badge to his or her belt. Replacement color change buttons are available to be inserted into these badges because the color changes occurring on exposure are permanent. Continuous use of suitable personnel protection indicators would be appropriate during leak testing operations. In addition, such leak testing areas can be monitored by area contamination monitors, as described next.

Contamination Monitoring of Excessive Accumulations of Toxic Gases

Area contamination monitors (ACMs) for atmospheric accumulations of gases and vapors such as ammonia, chlorine, hydrazine, hydrogen sulfide, nitrogen dioxide or ozone are self-adhesive filter papers that chemically react to concentrations of various gases or vapors. Indicating by means of color changes listed in Table 6, these area monitoring indicators are normally mounted on walls or bulkheads that are easily seen by supervisory personnel and by leak testing workers. Ideally, the monitors should be placed opposite an entrance door with a window (within buildings) or at locations where they are visible prior to entry in open areas, so that personnel can see their indications and do not enter any contaminated areas unnecessarily.

By contrast, the area contamination monitors for carbon monoxide is a triangular wall mounting plaque. The propane monitor is a vial of crystals. Both of these monitoring indicators change color, as indicated in Table 6, when excessive accumulations of the specific toxic gas are present.

Each of the contamination monitors listed in Table 6 indicates the accumulated exposure to the specific gas to which the work area has been exposed during the 8 h measurement period set by the Occupational Safety and Health Administration. A color change at any time during this 8 h interval indicates that anyone in the area is being exposed to a toxic gas concentration in excess of the safe maximum.

TABLE 6. Selection guide for area contamination monitors for toxic gases and vapors accumulating in leak testing areas.

Toxic Substance	Critical Concentration ($\mu L \cdot L^{-1}$)	Color Change
Ammonia	15	Brown to white
Carbon monoxide	50	White to Black
Chlorine	2	White to yellow
Hydrazine	5	White to yellow
Hydrogen sulfide	5	White to brown
Nitrogen dioxide	1	White to yellow
Ozone	0.1	White to brown
Propane	0.001	Purple to yellow

Portable Electronic Instrument for Locating Small Combustible or Toxic Gas Leaks

Figure 2 shows a portable, hand held electronic sensing instrument with pointing indicator; the instrument is used both for personnel protection and as a tracer gas detector in leak testing. It detects all combustible and many noncombustible toxic gases and vapors, including the following: acetone, alcohol, ammonia, benzene, butane, carbon monoxide, carbon tetrachloride, ethane, ethylene oxide, gasoline, hydrogen,

FIGURE 2. Portable personnel protection monitor and detector for leak testing of certain combustible or toxic gases.

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turpentine, hydrogen sulfide, liquid propane gas, methane, methyl ethyl ketone, naphtha, natural gas, propane, steam, sulfur dioxide, toluene, trichloroethylene and xylene.

This instrument does not detect carbon dioxide. It is a low cost, simple leak tracer designed to locate small leaks. A flexible 1 m (3 ft) extension hose can be used to sniff leaks in less accessible locations behind pipes or around complex pipe connections. Its use is often more convenient than using bubble tests and the small battery operated hand held detection and indicating instrument is often more feasible than larger electronic instruments requiring connections to alternating current power outlets. Its sensor is reported to detect 50 $\mu L \cdot L^{-1}$ of gas or vapor contaminant in atmospheric air and is designed for over 50 000 exposures to gases.

Precautions with Ammonia Gas

Ammonia (NH_3) is used as a tracer gas for many chemical indicator leak tests. At room temperature and atmospheric pressure, ammonia is a colorless, alkaline gas having a pungent odor, which provides ample warning of its presence. Ammonia gas is irritating to the eyes and to moist skin. However, concentrations of ammonia gas in air in the concentration range below 50 $\mu L \cdot L^{-1}$, although not harmful, are a considerable nuisance, so that people tend to avoid them. It is therefore unlikely that an individual would unknowingly become overexposed to ammonia gas.

Physiological Effects of Ammonia Gas

Table 7 lists the physiological effects of various concentrations of ammonia. The corrosive action of high concentrations (above 700 $\mu L \cdot L^{-1}$) can cause extensive injuries to the eyes, including severe irritation, hemorrhaging and swollen lids. If not treated immediately, partial or total loss of sight may result. The mucous lining of the mouth, throat, nose and lungs is particularly sensitive to ammonia attack.

Precautions with Anhydrous Liquid Ammonia

Contact with anhydrous liquid ammonia is intensely irritating to the mucous membranes, eyes and skin. Contact with the skin will produce severe burns and the freezing effect due to rapid evaporation of liquid ammonia from the skin surface can cause frostbite. Anyone working with liquid ammonia must wear rubber gloves, chemical protection clothing and goggles and a rubber or plastic apron.

Hazards of Explosion or Ignition with Ammonia

Ammonia cylinders should never be directly heated by steam, direct electric coils or flames. Uncontrolled heating of a cylinder can cause the liquid to expand to a point where dangerous pressures will be developed. Heating is done in a thermostatically controlled water or oil bath. In no case should the temperature be allowed to exceed 50 °C (120 °F).

Ammonia represents a possible flammability hazard. A mixture of air and ammonia containing from 15 to 28 percent ammonia by volume will ignite when sparked or exposed to temperatures exceeding 50 °C (120 °F). Therefore, flames and sparks should not be allowed in the area where ammonia is being used.

As another noteworthy consideration, ammonia can combine with mercury to form explosive compounds. Therefore, instruments containing mercury (such as manometers) should not be used where they will be exposed to ammonia.

Precautions with Argon Gas

On some occasions, argon (Ar) is used as a leak tracer gas. It is the most abundant member of the rare gas family, which consists of helium, neon, argon, krypton and xenon. All of these gases are monatomic and are characterized by their extreme chemical inactivity. Argon, a

TABLE 7. Physiological effects of various concentrations of ammonia gas (NH_3).

Atmospheric Concentration ($\mu L \cdot L^{-1}$)	Physiological Effects
20	First perceptible odor.
40	A few individuals may suffer slight eye irritation.
100	Noticeable irritation of eyes and nasal passages after few minutes' exposure.
400	Severe irritation of the throat, nasal passage and upper respiratory tract.
700	Severe eye irritation. No permanent effect if the exposure is limited to less than 0.5 h.
1700	Serious coughing, bronchial spasms; less than 0.5 h of exposure may be fatal.
5000	Serious edema, strangulation, asphyxia, fatal almost immediately.

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colorless, odorless and tasteless gas, is nontoxic. However, argon can act as a simple asphyxiant by displacing the amount of air necessary to support life.

Precautions with Carbon Dioxide Gas

Carbon dioxide (CO_2) is a nonflammable, colorless, odorless and slightly acid gas which is about one and one half times as dense (heavy) as air. The normal concentration of carbon dioxide in the atmosphere is 0.03 percent, or 300 $\mu L \cdot L^{-1}$. Gaseous carbon dioxide is not a chemically active compound as such and high temperatures are generally required to promote its chemical reactions. However, aqueous solutions of carbon dioxide are acidic and many reactions occur readily.

When it replaces breathable air, carbon dioxide acts as a simple asphyxiant. Because it is heavier than air and does not diffuse readily, pure carbon dioxide may collect in confined, unventilated areas or in lower regions of large vessels. Gaseous carbon dioxide is the regulator of the breathing function. An increase in the amount of carbon dioxide inhaled will cause an increased rate of breathing. The body, while exercising, will burn more oxygen and the product of this combustion will be higher concentrations of carbon dioxide. These higher concentrations of carbon dioxide produce higher rates of breathing listed (Table 8).

TABLE 8. Physiological effects of carbon dioxide gas in air.

Carbon Dioxide Gas in Air ($mL \cdot L^{-1}$)	Increased Lung Ventilation
1 to 10	Slight and unnoticeable
20	50 percent
30	100 percent
50	300 percent (breathing becomes laborious)

Concentrations of 10 percent (100 000 $\mu L \cdot L^{-1}$) of carbon dioxide in breathing air can produce unconsciousness; concentrations of 10 to 25 percent may cause death with exposures of several hours. A concentration of 5 percent may produce shortness of breath and headache. Continuous exposure to 1.5 percent carbon dioxide may cause changes in some physiological processes.

Characteristics of Refrigerant-12 Gas

The halogen tracer gas dichlorodifluoromethane (CCl_2F_2) was widely used in the 1980s. This was the refrigerant-12 gas used in air conditioners. It is a colorless, nonflammable gas at normal temperatures and pressures. In concentrations of less than 20 percent (by volume), refrigerant-12 is odorless. At high concentrations, its odor is mild and somewhat ethereal and similar to that of carbon tetrachloride. Refrigerant-12 is readily liquefied and is usually supplied in steel cylinders as a liquefied gas under its own vapor pressure of about 480 kPa (70 $lb_f \cdot in.^{-2}$ gage) at 21 °C (70 °F). Refrigerant-12 gas has also been known by several trade names, including Freon® 12. Its extensive use as a propellant for spray cans has been discontinued in the United States. Its manufacture in and its importation into the United States have been banned. However, if this gas is sprayed on very hot metallic surfaces or in the presence of flames, it can dissociate to form deadly toxic gases such as phosgene.

Refrigerant-12 gas is practically nontoxic. It shows no toxic effects in guinea pigs in concentrations up to at least 20 percent by volume for 2 h exposures. In higher concentrations, refrigerant-12 may produce some physiological action, caused primarily by oxygen deficiency. The generally accepted maximum allowable refrigerant-12 concentration for an 8 h daily exposure of personnel is 1000 $\mu L \cdot L^{-1}$.

Precautions with Helium Gas

Helium (He) is widely used as a tracer gas in leak testing with the mass spectrometer leak detector. It is the lightest member of the rare gas family and is a chemically inert, colorless, odorless and tasteless gas. Helium is not toxic but can act as an asphyxiant by displacing the air necessary to support life. Because of its low density, helium tends to rise to the top regions of closed vessels or enclosures, where it could lead to asphyxiation of workers at these elevations.

Characteristics of Hydrogen Chloride Gas

To some degree, hydrogen chloride (HCl) has also been used as a tracer gas. Anhydrous hydrogen chloride is a colorless, pungent, nonflammable,

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corrosive gas with a suffocating odor. It is heavier than air, soluble in water and fumes strongly in moist air. The aqueous solution is known as hydrochloric acid (or muriatic acid) and may contain as much as 38 percent hydrogen chloride. Hydrogen chloride is supplied in cylinders in the form of a gas over a liquid. The cylinder pressure is about 4.2 MPa (610 $lb_f \cdot in.^{-2}$ gage) at 21 °C (70 °F). As long as liquid is present in the cylinder, this pressure remains fairly constant. When the liquid phase is exhausted, cylinder pressure drops rapidly.

Physiological Effects of Hydrogen Chloride Gas

Hydrogen chloride is a highly toxic gas that severely irritates the upper respiratory tract and is corrosive to the eyes, skin and mucous membranes. It may produce dermatitis on repeated exposures. Eye contact may result in reduced vision or blindness. Ingestion may be fatal. Hydrogen chloride concentrations of 0.13 to 0.2 percent (1300 to 2000 $\mu L \cdot L^{-1}$) in air are lethal for human beings in exposure lasting only a few minutes. The maximum hydrogen chloride concentration that can be tolerated for exposures of 60 min is in the range of 0.005 to 0.01 percent (50 to 100 $\mu L \cdot L^{-1}$). However, the unpleasant effects of hydrogen chloride provide adequate warning, leading to prompt voluntary withdrawal of personnel from hydrogen chloride contaminated atmospheres.

Precautions with Hydrogen Chloride Gas

Personnel who handle hydrogen chloride gas must wear protective clothing such as rubber or plastic aprons, rubber gloves and suitable gas tight safety goggles. Appropriate gas masks with cannisters or supplied air respirators should be provided when hydrogen chloride vapor concentrations are excessive. Woolen outside clothing or other acid-resisting fabrics are also recommended for personnel handling hydrogen chloride. Personal hygiene and showering after each work shift should be encouraged. When hydrogen chloride is supplied from cylinders, users should always shut off their hydrogen chloride lines from the use end, closing valves successively backward to the cylinder.

Dry gaseous hydrogen chloride is essentially inert to metals and does not attack the commonly used structural metals under normal conditions of use (room temperature and atmospheric pressure). In the presence of moisture, however, hydrogen chloride will corrode most metals (other than silver, platinum or tantalum). When hydrogen chloride is used at higher pressures, it is necessary to use extra-heavy steel pipe throughout. No galvanized pipe or bronze valves should be used.

Precautions with Hydrogen Gas

Hydrogen (H_2) is colorless and odorless and is the lightest gas known. It is nontoxic but can act as an asphyxiant by displacing the necessary amount of air required to support life. Because hydrogen is much lighter than air, it tends to collect near the top of closed vessels. Hydrogen, in combination with air or oxygen, can explode with great violence. Hydrogen gas, although relatively inactive at ambient temperatures, reacts with almost all the other elements at high temperatures and is considered to be a very dangerous tracer gas. For this reason, hydrogen should be avoided if at all possible.

When large vessels are tested or when large volumes of hydrogen are used, it may be advisable to provide monitoring equipment that gives a continuous indication of the hydrogen and air content in the test area. Intrinsically safe detectors are available. This precaution may also be advisable when high vacuum vessels are in the process of being chemically cleaned before evacuation because the vessel interior as well as the surrounding environment may contain an explosive mixture.

Precautions with Radioactive Krypton-85 Gas

Krypton gas is completely chemically inert and, thereby, forms no chemical combinations with any material used in the tested components. Radioactive krypton-85 tracer gas, due to its chemical inertness, does not participate in any metabolic processes in the body if inhaled or ingested in any way. If accidentally inhaled for a short time, normal breathing of noncontaminated air will rapidly remove the radioactive krypton gas from the lungs and body tissue into which it might be diffused. With an adequate ventilating system, proper gamma ray shielding of storage tanks and reasonable care, krypton-85 tracer gas can be handled with negligible risk to the operators.

Krypton-85 has a radioactive half life or 10.76 yr. Over 99 percent of the disintegrations give no gamma rays but emit beta particles with a maximum energy of 0.67 MeV. Only 0.7 percent of

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the disintegrations yield 0.514 MeV gamma rays. The primary usefulness of krypton-85 for leak testing depends on this small proportion of gamma emitting disintegrations, reinforced in some applications by the emission of low energy bremsstrahlung or very soft X-rays.

Many industrial hand held portable survey meters are used to detect the presence of trace quantities of krypton-85 gas. The high percentage of beta particle emission allows for detection of nanocuries of krypton-85 gas in the air. Additionally, all equipment approved to handle krypton-85 gas is required to have air monitors in continuous operation to detect any airborne krypton-85 gas and initiate an alarm.

Precautions with Methane Gas

Methane is sometimes used as leak testing tracer gas. Natural gas consists primarily (85 percent) of methane. Methane gas (CH_4) in its pure state is flammable, colorless, odorless and tasteless and is not considered toxic. It can act as a simple asphyxiant where, present in high concentrations, it displaces the oxygen necessary to sustain life. As an example, coal miners frequently breath air containing 9 percent methane and do not appear to suffer. When concentration increases above this point, pressure on the forehead and eyes is noticed. However, this pressure disappears again on breathing fresh air. Methane in mixtures with air or oxygen burns rapidly. Ignition leads to explosions similar to many coal mine explosions. Incomplete combustion of methane gas may produce carbon monoxide, a toxic gas.

Precautions with Nitrogen Gas

Nitrogen (N_2) is not often used as a tracer gas but may be used to backfill vacuum vessels or may be mixed with a tracer gas and introduced into a vessel before a pressure leak test. Nitrogen gas comprises about 79 percent by volume of the air. It will not burn and will not support combustion. It is nontoxic; however, nitrogen can act as an asphyxiant by displacing the amount of air necessary to sustain life. This gas is extremely inert, except when heated to very high temperatures where it combines with metals to form nitrides. At pressures of 400 kPa (4 atm) or higher, the gaseous nitrogen in normal air induces a narcotic action evidenced by decreased ability to work, mood changes and frequently a mild to marked euphoria. These responses are similar to those associated with alcoholic intoxication.

Precautions with Nitrous Oxide

Nitrous oxide (N_2O) is used as a tracer gas in the performance of some leak tests, such as those using the infrared leak test method. Nitrous oxide is a colorless, nonflammable gas with a slightly sweetish taste and odor. It is nontoxic and nonirritating and must not be confused with other nitrogen oxides that can be harmful. Nitrous oxide is a rather weak anesthetic and must be inhaled in high concentrations, mixed with air or oxygen, when regularly used as an anesthetic in medicine and dentistry. Medical and dental personnel who repeatedly inhale this gas over a long period of time are known to suffer nerve damage. When inhaled without oxygen, nitrous oxide is a simple asphyxiant. Inhalation of small amounts of nitrous oxide often produces a type of hysteria, which accounts for its common name of *laughing gas*.

It is to be recognized that most other nitrogen oxides can be harmful. California's Occupational Safety and Health Administration standard for all nitrogen oxides combined is a concentration of 5 $\mu L \cdot L^{-1}$ ceiling for an 8 h occupational standard. The California ambient air standard for nitrogen-oxygen pollutants is 0.25 $\mu L \cdot L^{-1}$ for 1 h. The Federal standards in 1978 for nitrogen oxides, determined as time weighted averages (TWAs), are 25 $\mu L \cdot L^{-1}$ or 30 $mg \cdot m^{-3}$ for nitric oxide (NO), 5 $\mu L \cdot L^{-1}$ or 9 $mg \cdot m^{-3}$ for nitrogen dioxide (NO_2) and 2 $\mu L \cdot L^{-1}$ or 5 $mg \cdot m^{-3}$ for nitric acid (HNO_3).

Precautions with Oxygen

Even though oxygen (O_2) is not often used as a tracer gas, there should be full awareness of its potential hazards. Oxygen is a colorless, odorless, tasteless gas and its outstanding properties include its ability to sustain animal life and to support combustion. Inhalation of 100 percent oxygen at atmospheric pressure (100 kPa or 1 atm) will irritate the throat although symptoms of oxygen poisoning do not occur if the exposure is relatively short. Long periods of exposure to higher oxygen pressures can adversely affect neuromuscular coordination and the power of attention. Inhalation of oxygen when its partial pressure exceeds 200 kPa (2 atm) may result in the signs and symptoms of oxygen poisoning. These

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include tingling of fingers and toes, acoustic hallucination, confusion, muscle twitching (especially about the face) and nausea. The final result of such exposure may be convulsion, which ceases as soon as exposure to high partial pressures of oxygen is terminated. Note that carbon dioxide enhances the toxicity of oxygen and the narcotic effect of nitrogen.

Precautions against Oxygen Fires and Explosions

Pressurized oxygen reacts violently with oil, grease, fuel gases or metallic particles, often producing flames or violent explosions. The cylinders in which gaseous oxygen is supplied are often pressurized to 14 or 15 MPa (2.2×10^3 $lb_f \cdot in.^{-2}$ gage). Thus, oil, grease or readily combustible materials should never be allowed to come into contact with interiors of oxygen cylinders, valve, pressure regulators and fittings. These components should never be lubricated with oil, grease or other combustible substances containing hydrocarbons.

Oxygen gages, regulators and fittings should never be used for compressed air (which may contain lubricants from air pumps). Similarly, gages regulators and fittings used with air or other gases should never be used on oxygen systems, for fear of violent explosions. It is also advisable never to use manifolds for pressurized oxygen systems unless these are designed and constructed with the advice and control of a qualified engineer. Manifolds must comply with applicable regulations and safety procedures. Cylinders of oxygen should not be stored near cylinders of acetylene or fuel gases.

Characteristics of Sulfur Dioxide

Sulfur dioxide (SO_2), through extremely undesirable, is sometimes used in the leak testing of welded pressure vessels. It is a highly irritating, nonflammable, colorless gas at room temperature and atmospheric pressure. Liquid sulfur dioxide may cause skin and eye burns on contact with these tissues as a result of the freezing effect of sulfur dioxide liquid on the skin or eyes. Sulfur dioxide is also a highly irritating gas in the vapor form, but is readily detectable in concentrations of 1 to 3 $\mu L \cdot L^{-1}$ and so provides ample warning of its presence.

Slight tolerance, at least up to the odor threshold and general acclimatization are common. Sensitization in a few individuals, particularly young adults, may develop following repeated exposure. In higher concentrations, the severely irritating effects of gaseous sulfur dioxide make it unlikely that any person would be able to remain in such a contaminated atmosphere unless he or she were unconscious or trapped. The adverse effects of sulfur dioxide are heightened by the presence of dust, dirt, soot or other particulates in the air. If particulates are high in concentration in the air, even a little sulfur dioxide can cause illness.

Chronic exposure to sulfur dioxide may result in fatigue, altered sense of smell and chronic bronchitis symptoms. Short acute exposure to sulfur dioxide gas has severe effects. A concentration of 8 to 12 $\mu L \cdot L^{-1}$ causes throat irritation, coughing, constriction of the chest, tears and smarting of the eyes; a concentration of 150 $\mu L \cdot L^{-1}$ causes extreme irritation and can be tolerated for only a few minutes; and a concentration of 500 $\mu L \cdot L^{-1}$ causes a sense of suffocation because it is so acutely irritating. Acute overexposure to sulfur dioxide may result in death from asphyxiation.

Precautions with Sulfur Dioxide

Sulfur dioxide should be handled only in a well ventilated area, preferably using a hood with forced ventilation. Personnel handling sulfur dioxide should wear chemical safety goggles or plastic face shields (or both), approved safety shoes and rubber gloves. Additional gas masks, airline gas masks and self-contained breathing apparati should be at hand for emergencies. Instant acting safety showers should be available in convenient locations.

Where sulfur dioxide gas is excessive, the worker should be supplied with a full face piece cartridge, canister respirator or supplied air respirator. Goggles, protective clothing and gloves should be worn if splashes of liquid are likely. In areas of splash or spill, impervious clothing should be supplied. If work clothes are wetted by sulfur dioxide, they should be removed promptly and the skin area washed thoroughly.

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PART 6. Safety Precautions with Compressed Gas Cylinders

Handling and Use of Compressed Gas Cylinders

Most of the gas used for leak testing is purchased in cylinders, which should be constructed and maintained in accordance with regulations of the Interstate Commerce Commission. The contents should be legibly marked on each cylinder in large letters.

Serious accidents may result from the misuse, abuse or mishandling of compressed gas cylinders. Technicians assigned to the handling of pressurized cylinders should be carefully trained and work only under competent supervision. Observance of the following rules will help control hazards in the handling of compressed gas cylinders.

1. Accept only cylinders approved for use in interstate commerce for transportation of compressed gases.
2. Do not remove or change numbers or marks stamped on cylinders.
3. Never move cylinders unless the protective cap is in place. Because of their shape, smooth surface and heavy weight, cylinders are dangerous to carry by hand and some type of carrying device should be used when they must be moved without the aid of a cart. Cylinders may be tilted and rolled on the bottom edge, but they should never be dragged.
4. Protect cylinders from cuts or abrasions.
5. Do not lift a compressed gas cylinder with an electromagnet. Where cylinders must be handled by a crane or derrick when testing field erected vessels, carry them in a cradle or similar device. Take extreme care that they are not dropped. Do not use slings or chains.
6. Do not drop cylinders or let them strike each other violently.
7. Do not use cylinders for rollers, supports or any purpose other than to contain gas.
10. When empty cylinders are to be returned to the vendor, mark them *EMPTY* or *MT* with chalk. Close the valves and replace the valve protection caps.
11. Load cylinders to be transported so as to allow as little movement as possible. Secure cylinders to prevent violent contact or upsetting.
12. Always consider cylinders as full and handle them with corresponding care. Accidents have resulted when containers under partial pressure were thought to be empty.
13. Use of safety chains to secure cylinders during use to prevent accidental falling is required practice by the Occupational Safety and Health Administration.

Precautions for Storage of Compressed Gas Cylinders

Store compressed gas cylinders with protective caps properly installed in safe, dry and well ventilated places prepared and reserved for this specific purpose. Cylinders should be stored on a level, fireproof floor and should be chained in place or provided with barriers to prevent them from falling over. Flammable substances such as oil and volatile liquids should not be stored in the same area as pressurized gas cylinders. Cylinders should not be stored near arc welding areas, elevators, gangways, stair wells or other places where they could be knocked over, arc gouged or damaged. Cylinders are not designed for temperatures in excess of 55 °C (130 °F). Accordingly, they should not be stored near sources of heat such as radiators or furnaces, nor near highly flammable substances like gasoline.

Cylinder storage should be planned so that cylinders will be used in the order in which they are received from the supplier. Empty and full cylinders should be stored separately, with empty cylinders being plainly identified as such to avoid confusion. Group together cylinders that have held the same contents.

Precautions in Indoor Storage of Oxygen and Fuel Gas Cylinders

Cylinders of oxygen must not be stored indoors close to cylinders containing flammable gases. Unless they are stored apart, oxygen cylinders and flammable gas cylinders must be separated by a fire resistive partition. A direct flame or

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electric arc should never be permitted to contact any part of a compressed gas cylinder.

Acetylene and liquefied fuel gas cylinders should be stored with the valve end up. The total capacity of acetylene cylinders stored inside a building should be limited to 60 m^3 (2000 ft^3) of gas, exclusive of cylinders in use or connected for use. Quantities exceeding this total must be stored in a special room, located in a separate building or outdoors and built in accordance with the specifications of NFPA 51, *Standard for the Design and Installation of Oxygen-Fuel Gas Systems for Welding, Cutting, and Allied Processes.*[13]

Storage rooms for cylinders containing flammable gases should be well ventilated to prevent the accumulation of explosive concentrations of gas. No source of ignition will be permitted; smoking must be prohibited. Wiring should be in conduit. Electric lights should be in fixed positions and enclosed in glass or other transparent material and equipped with guards to prevent breakage. (Note that glass enclosures, electrical conduit and conventional switch and receptacle boxes used in electrical wiring systems do not prevent entry of gases into their enclosures.) Therefore, electrical switches, which are subject to sparking or arcing during operation, should be located outside the room in which flammable gases are stored.

Precautions in Outdoor Storage of Gas Cylinders

One common type of storage house consists of a shed roof with side walls extending about halfway down from the roof and a dividing wall between cylinders of one kind of gas and those for another gas. To prevent rusting, cylinders stored in the open should be protected from contact with the ground and against extremes of weather, accumulations of ice and snow in winter and continuous direct rays of the sun in summer.

Safe Procedures for Using Cylinders of Compressed Gases

Safe procedures for compressed gas cylinders include the following.

1. Use cylinders in the upright position and secure them to prevent them from being accidentally knocked over.
2. Unless the cylinder valve is protected by a recess in the head, keep the metal cap in place to protect the valve when the cylinder is not connected for use. A blow on an unprotected valve might cause gas under high pressure to escape.
3. Make sure the threads on a regulator or union correspond to those on the cylinder valve outlet. Do not force connections that do not fit.
4. Open cylinder valves slowly. A cylinder not provided with a handwheel valve should be opened with a spindle key, a special wrench or other tool provided or approved by the gas supplier.
5. Do not use a cylinder of compressed gas without a pressure reducing regulator attached to the cylinder valve, except where cylinders are attached to a manifold, in which case the regulator should be attached to the manifold header.
6. Before making connection to a cylinder valve outlet, except that of a hydrogen cylinder, crack the valve for an instant to clear the opening of particles of dust and dirt. Always point the valve and opening away from the body and not toward anyone else. Operators should wear safety glasses.
7. Use regulators and pressure gages only with gases for which they are designed and intended. Do not attempt to repair or alter cylinders, valves, regulators or attachments. This work should be done only by the manufacturer.
8. Unless the cylinder valve has first been closed tightly, do not attempt to stop a leak between the cylinder and the regulator by tightening the union nut.
9. Combustible gas cylinders in which leaks occur should be taken out of use immediately and handled as follows: (a) Close the valve and take the cylinder outdoors well away from any source of ignition. Properly tag the cylinder and notify the supplier. A regulator attached to the valve may be used temporarily to stop a leak through the valve seat. (b) If the leak occurs at a fuse plug or other safety device, take the cylinder outdoors well away from any source of ignition, open the cylinder valve slightly and permit the gas to escape slowly. Tag the cylinder plainly. Post warnings against approaching with lighted cigarettes or other sources of ignition, promptly notify the supplier and follow its instructions for returning the cylinder.
10. Do not permit heavy objects, sparks, molten metal, electric currents, excessive heat or flames to come in contact with cylinders or attachments.
11. Never use oil or grease as a lubricant for valves or attachments of oxygen cylinders. Keep oxygen cylinders and fittings away from oil and grease and do not handle such cylinders or

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apparatus with oily hands, gloves or clothing. Signs should be posted where oxygen is stored, prohibiting oil, grease or other lubricants on oxygen equipment.
12. Never use oxygen as a substitute for compressed air in pneumatic tools or to start internal combustion engines or for pressurizing a system for testing or for dust removal. Use it only for the purpose for which it is intended.
13. Never bring gas cylinders into vessels or unventilated rooms.
14. Do not fill cylinders except with the consent of the owner and then only in accordance with regulations. Do not attempt to mix gases in a compressed gas cylinder or to use it for purposes other than those intended by the supplier.
15. Secure all gages and hoses with proper size wrenches, not slip jaw pliers.
16. Do not overtighten or strip threads on cylinder attachments.

Safety Precautions with Valves or Regulators on Gas Cylinders

Regulators or reducing valves must be used on gas cylinders to maintain a uniform gas supply. Technicians should stand to one side and away from regulator gage faces when opening cylinder valves. Always wear safety glasses to protect eyes from ejected particles.

Only regulators listed or approved by agencies such as Underwriters' Laboratories, Incorporated, should be used on cylinders of compressed gas. Each regulator should be equipped with both a high pressure (contents) gage and a low pressure (working) gage.

Safety Precautions with Oxygen Pressure Regulators

High pressure oxygen gages should have safety vent covers to protect the operator from broken glass in case of an internal explosion. Each oxygen gage should be marked *OXYGEN—USE NO OIL*.

Serious, even fatal accidents, have resulted when oxygen regulators have been attached to cylinders containing combustible gas or vice versa. To guard against this hazard, it has been customary to make connections for oxygen regulators with right hand threads and those for combustible gases such as acetylene with left hand threads, to mark the gas service on the regulator case, and to paint the two types of regulators different colors. Cylinder valve outlet threads have been standardized for most industrial and medical gases by the American National Standards Institute, recommending different combinations of right hand and left hand threads, internal and external threads and different diameters to guard against wrong connections. Standards are being rapidly adopted whenever gas manufacturers and industrial users reach agreement to change both valve outlets and regulator connections. Adaptors are used in the interim until the changes are completed.

The regulator is a delicate apparatus and should always be handled carefully. It should not be forced, dropped or pounded. Regulators should be sent to the manufacturer for repairs and testing by skilled personnel.

Safety Procedures for Leaky or Anomalous Regulators

Leaky or creeping regulators are a source of danger and should be withdrawn from service at once for repairs. If a regulator shows a continuous creep, indicated on the low pressure (delivery) gage by a steady buildup of pressure when the outlet valves are closed, the cylinder valve should be closed and the regulator removed for repairs. If the regulator pressure gages have been strained so that the pointers do not register properly, the regulator must be repaired at once. When regulators are connected but are not in use, the pressure adjusting device should be released. Cylinder valves should never be opened until the regulator is drained of gas and the pressure adjusting device on the regulator is fully released.

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PART 7. Safety Precautions in Pressure and Vacuum Leak Testing

Safety Considerations in Leak Testing

When a pressure or a vacuum vessel is fabricated, some means of testing must be used to predict safe performance of the vessel. It is sometimes necessary to exceed the designed operating conditions during initial pressure testing. This requires many safety considerations to ensure proper protection of personnel. (Hazards related to toxic or flammable solvent vapors and tracer gases in leak testing should also be given careful consideration.)

Explosion and Implosion Hazards in Pressure and Vacuum Leak Testing

Pressurized vessels can fail by explosion because of the energy stored in air or nonflammable gases used to pressurize systems during leak testing. In systems that are evacuated during leak testing, implosion (violent collapse) failures can result from external (atmospheric) pressures applied to structures not designed for such loading. Where flammable tracer gases are used in leak testing in the presence of air or oxygen, violent combustion or explosive chemical reactions can occur. These hazards must be foreseen and carefully controlled to ensure safety during leak testing.

Precautions in Selecting Sites for Leak Testing

Major factors determining the size, shape and type of buildings and structures to be used for leak testing of components need to be investigated. Catastrophes resulting in large loss of life and heavy property damage often are due to inadequate planning stage considerations. High hazard leak testing operations should be located in small isolated buildings of limited occupancy. Buildings can be designed so that internal explosions will produce minimum damage and minimum broken glass. Lower hazard operations can justify large units.

Pressure Vessel Code Requirements for Safety Procedures

The degree of safety precautions necessary during leak testing varies greatly with the type of system being tested. In the case of hydrostatic and pneumatic tests of pressure vessels, the *ASME Boiler and Pressure Vessel Code* outlines the minimum safety procedures to be followed during pressure testing. The *ASME Boiler and Pressure Vessel Code* and other applicable specifications should be followed with care to ensure safety in all operations to which they apply. However, often it is the rather subtle hazard that may be disastrous. Potential hazards should be taken into account both when preparing for or performing leak testing. These include tracer gas safety aspects such as flammability, asphyxiation or specific physiological effects as well as the possibility of pressure vessel explosions.

Protecting Test Personnel during Pressure Testing

Greater respect for high pressure testing has led to increased emphasis on safety, with the result that overall safety experience has been very good. This respect is well justified when one realizes that a valve stem operating at 200 MPa (3×10^4 $lb_f \cdot in.^{-2}$) that fails and is blown out is propelled under conditions similar to those of a bullet fired from a high powered rifle. The energy released from a completely liquid system should not be underestimated either. Compressed liquid, although smaller volumetrically than compressed gas, is very much to be reckoned with in considering potential forces to be handled when pressure is released. For example, a gasket 0.4 mm (0.016 in.) thick, blown between split flanges under a pressure of more than 10 MPa (more than 2×10^3 $lb_f \cdot in.^{-2}$), will release a thin sheet of water like a knife edge that could cause injury, eye damage and loss of sight.

Successful personnel protection during pressure testing involves not only mechanical devices to guard against

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injury should failure occur, but thorough training of people, establishment and enforcement of rigid safety rules and necessary disciplinary action when justified. Without the proper attitude and respect for what is being handled, trouble is sure to occur.

Safety with Scaffolds

A scaffold is an elevated working platform, usually temporary, for supporting both men and materials. For safety's safe, scaffolds should be designed to support at least four times the anticipated weight of men and materials to be placed on them and all elevated working platform areas should be guarded (as by railings) on all exposed sides. Working scaffolds should not be used as a platform for jacking or leverage purposes without proper allowance for the added loads and stresses.

Barricades, Protective Walls and Distance for Safety during Leak Testing

Based on safety experience accumulated during laboratory operations and on sound design principles, a custom vessel can be built with reasonable assurance that it may be leak tested or pressure tested safely. While complete isolation usually is not required, certain pieces of equipment may need barricade protection. Access to the test area during testing should be restricted to minimize exposure of personnel to hazards.

Remote control and observation may be used where possible during leak testing. Periscope techniques, shatterproof glass windows and industrial television offer opportunities to check on operating equipment without exposure. Instrument data can be transmitted electrically or by low pressure pneumatic systems to a separate control room. Valves that are not controlled automatically can be operated by rods or shafts extended through a barricade gage board combination with proper seals.

Pressure Vessel Design and Causes of Failures

Fired and unfired pressure vessels of many types are in common use in industrial, commercial and public buildings for space and process heating and heat exchange; for processing food, chemicals, petroleum and other industrial products; and for processes involving nuclear energy. These vessels hold gases, vapors, liquids and solids at various temperatures and at various pressures, ranging from absolute pressures of nanopascals or lower pressures to tens of megapascals (10^{-9} to 10^{7} Pa).

Some common causes of failure in pressure vessels are the following: (1) errors in design, construction and nondestructive testing; (2) improper education of testing personnel; (3) mechanical breakdown such as failure, blocking or lack of safety devices; (4) poor visual inspection before pressurization; (5) improper test procedure; (6) improper application of test equipment; (7) blocked or dysfunctional gages; (8) test flanges or valves of wrong material; (9) improperly designed test flanges; and (10) test pressure too high. These causes of potential failures should be anticipated and avoided insofar as possible. Before a pressure vessel is tested, three questions should be answered about its design.

1. Can the filled vessel carry the weight of its contents in addition to the internal pressure without undue strain?
2. Can the support structure and building floor carry the weight of the filled vessel?
3. Can the vessel withstand any vacuum and not collapse under external atmospheric pressure that may be created either accidentally or intentionally?

It is imperative that any safety enclosures be designed to withstand the worst possible conditions of failure; otherwise, protective walls may break and lethal fragments of metal or concrete can be blown outward. Vented roofs or pressure testing below ground level should be considered when pressure testing with compressed air or gases.

Precautions for Protection against Equipment Failure from Overpressure

Safety precautions to protect personnel and equipment from failures during pressure testing include the following.

1. Ensure that the test equipment and vessel under test are properly designed and constructed in the first place.
2. Before pressure testing, ensure that equipment is properly assembled to avoid overstressing. This includes proper bracing and shoring under pressure vessels to support critical points. Otherwise it is possible that failure may actually be started before or while equipment is being set up for the test.
3. Be sure that careful visual and other inspections are done during

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construction and testing to guarantee compliance with design, proper manufacturing procedures, material choices and workmanship standards.
4. Watch out for areas where stress concentrations or nonuniform loading in enclosures, pump and compressor cylinders, valves etc. may cause sudden or gradual failures.
5. Install preliminary warning devices that alert the leak testing technician when test pressures are increasing too rapidly or when pressurization is approaching an excessive level. These devices can call attention to an abnormal situation before a pressure relieving device is set off. Prompt correction of trends toward excessive pressure can often forestall the actuation of emergency pressure relief valves. This is most valuable in extreme pressure work.
6. Check temperature of test water or other test medium for compliance to test procedure.
7. Assure the availability on test site of approved written test and safety procedures for all test personnel.

Pressure Relieving Devices in Pressure Leak Testing

Spring loaded relief valves are used up to 100 MPa (1.5×10^4 $lb_f \cdot in.^{-2}$) as pressure relieving devices. They are quite reliable for nonpulsating operations at 15 to 20 percent above working pressure, but cannot be completely relied on to reseat without leakage. Shear rupture disks, made of bronze, stainless steel or other metals, depending on service conditions, are suitable for nonpulsating operations at test pressures up to 20 to 30 percent above working pressure. Formed heads failing in tension have been applied to appreciable pressures but do not possess the accuracy required at higher pressure up to 70 MPa (1×10^4 $lb_f \cdot in.^{-2}$).

Sometimes relief valves and rupture disks are used in parallel. In this case, the relief valve is set to open at a lower pressure. This warns test technicians that prompt corrections may be necessary to avoid rupture disk failure, with resulting lost time. Rupture disks and relief valves are also used in series, with one or the other in the upstream position. In this series case, unless a small vent hole is used between the two to prevent seepage, the back pressure caused by seepage can force the failure pressure on the upstream unit to rise to a dangerous value equal to the relieving pressure.

Hydraulically loaded plugs using O-ring seals are dependable and will relieve at test pressures closer to the working pressure than other devices. O-ring seals are typically flexible ring shaped inserts placed in circular grooves and compressed to form tight seals between mating parts of pressure or vacuum systems. They can have any cross sectional area required for protection and can be designed for any relieving pressure. The O-ring seals should be made of material that will not fail or deteriorate from the test medium used.

Pressure Gage Calibration and Safety Applications

One of the best means of protection from overpressure is to use an accurate gage. To ensure accuracy of a pressure gage, it must be periodically checked against some known standard pressure. Dead weight testers are used for calibration and checking of the elastic gages for pressures exceeding approximately 100 kPa (15 $lb_f \cdot in.^{-2}$) and extending to 70 MPa (10^5 $lb_f \cdot in.^{-2}$) or even higher. Dead weight gages are used for the precise determination of essentially constant pressures maintained in a vessel by some pressure generating mechanism. The dead weight tester and the pressure gages should both be calibrated over their full scale. Pressure gages should be calibrated both before and after testing on critical high pressure tests. Gage calibration should follow approved written procedures.

Care, Handling and Storage of Pressure Gages

Handling and storage should be done with the knowledge that a gage suitable for accurate pressure measurement is as delicate as a watch. Its removal and replacement for calibration purposes (and, of course, its installation and use) should be entrusted only to persons who can be depended on to avoid dropping or jarring the gage or subjecting it to rough treatment. The gage should always be attached by using a wrench on the flats provided on the connection. A gage must never be screwed or unscrewed by using the gage casing. If the gage has the proper tolerances and is handled correctly, the gage corrections determined before and after the pressure test for each test for which the gage was used should agree within specified calibration accuracies. If the gage is not handled properly, there is a chance that the calibration and corrections determined before and after the test will differ appreciably. In such events there is no sure way to know which correction to use and the result of the test will be in doubt.

Any accident to a gage requires that the gage be given a complete calibration

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and correction test before further use. This would apply also if a gage shows obvious evidence of prior damage. In the event that a gage is mishandled by dropping it, exceeding its pressure range or exposing it to vacuum (unless it is an absolute pressure gage), the gage must be repaired and recalibrated by its manufacturer, a qualified laboratory or equipment manufacturer with proper calibration facilities.

Hazards of Pressurized Test Systems

The necessary safety precautions vary greatly with the type of system being leak tested. Some general types are listed below in ascending order of the potential danger involved.

1. With small hydraulic systems of moderate pressure, the major hazard is from a jet of the liquid either from a leak or failure. Occasionally, the necessity to include a brittle material such as a sight glass or glass flow meter in the system adds the hazard of flying particles.
2. Low pressure systems involving nonreactive gases or liquids above their boiling point involve little hazard if correctly handled. However, it is important to have the proper relief valves, rupture disks and pressure regulators to maintain safety in low pressure systems. The hazard of low pressure systems can be higher if large volumes of gases are involved.
3. Systems involving flammable gases or liquids (such as kerosene) as the pressure testing fluid involve major hazards, including those of fires or explosions resulting from leakage or failure of some component.
4. The hazards of high pressure hydraulic and inert gas systems increase with the increase in pressure, the compressibility of the testing media and the volume of the system. There is an increasing probability that equipment in the higher pressure ranges will not permanently resist the effect of pressure.

Explosion of Systems or Vessels Pressurized for Leak Testing

If a system to be leak tested is pressurized with tracer gas or gas mixtures, rupture of its containment walls or pressure boundaries could produce considerable damage. If the system being pressurized is small, it might seem as if few precautions would be necessary during pressurizing. However, the damage from rupture of a gas filled volume results from the total amount of gas it contains. Therefore, either a small system under high pressure can be as dangerous a large system under lower pressure. The energy stored in a pressurized gas volume is equal to the product of its pressure and its volume. The pressure in pascal or newton per square meter multiplied by the volume in cubic meter results in energy in joule, $(N \cdot m^{-2}) \times m^3 = Nm = J$. By comparison, 1 kg of gasoline contains about 44 MJ, enough to blow up a tank.

When pressurizing a system, a pressure regulator fitted with a safety overpressure release device should be installed so that a pressure in excess of the design pressure can never be applied to a vessel or system under test.

Rupture Hazards in Pressure Testing

Although the prevention of clogged leaks dictates that leak testing with gaseous tracers should be done before contact of the system with liquid, the need for safety might overrule this procedure. Pressurizing a system with a liquid does not create the explosion hazard involved with gases under high pressure. Therefore, safety requirements may dictate pressure testing of a system with a liquid before gases are introduced for leak testing. An alternate preliminary leak test might be a low pressure, high sensitivity mass spectrometer leak test using helium as the tracer gas.

The amount of energy stored in a tank pressurized with gas is a function of the quantity and type of gas contained in the vessel. Because of this, a high volume, low pressure vessel can contain the same stored energy as a low volume, high pressure vessel. Therefore, each presents a hazard of similar magnitude.

The exact rupture hazard involved in pressure testing is difficult to define, although the structural burst limit is a reasonably predictable design factor. Any damage incurred during fabrication, erection, testing or service by a vessel under pressure, such as weld undercut or a deep nick or gouge, may cause explosive failure if the damage is severe. Small flaws can be progressive depending on metal strain and the type of load. Surface stress concentrations caused by vessel damage may not result in immediate failure, but may progress and cause failure later. When a skin puncture takes place, it results in a tearing action that tends to enlarge the hole. An inspection of a failed vessel will show tears extending across the entire face of the skin.

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Effects of Leak Size and Shape of the Opening on Failure Mechanisms

A smaller leak will dissipate the same energy as a larger leak, but over a long period without the explosive effect. The critical leak size is related to the tensile strength of the enclosing skin. The critical point of explosive pressure release is reached when the force of the gas escaping from the hole exceeds the force that can be withstood by the skin.

Another factor influencing failures is the shape of the leak opening. An irregularly shaped opening, with many microscopic irregularities, each providing a stress point, offer an ideal starting point for a tearing and shredding action. As the pressure in a tank increases, a critical pressure is reached where the stress exerted by the confined gas exceeds the strength of the metal surrounding the failure. This causes an explosive disintegration. Variation of the critical point of explosive pressure release can occur with conditions of service, vessel shape, size and wall thickness and material, fabrication methods and type of failure.

Energy Contained in Pressurized Vessels

The work done to compress gas in a vessel is stored in that gas. It is normally returned by propelling the gas to places where it is needed. However, a rupture in the tank may suddenly release all the energy at once as an explosion. An explosion is so fearsome because of the short duration of the energy release that can be calculated by means of equations for isentropic processes:

$$\textbf{(3)} \qquad PV^{k} = \text{constant}$$

and for work ${}_1W_2$:

$$\textbf{(4)} \qquad {}_1W_2 = \frac{P_2V_2 - P_1V_1}{1 - k}$$

Equations 3 and 4 take into account any sudden temperature change during the explosion. In this equation, $k = 1.4$, a constant. The work done (energy released) is that resulting from a change from conditions identified by subscript *1* to those identified by subscript *2*. As an example, compute the energy released when a small pressurized tank is ruptured and compressed gas escapes to atmospheric pressure and temperature. If the internal pressure within the tank is $P_1 = 15$ MPa (150 atm), the volume of the tank is V_1 equals 0.04 m^3, and the compressed gas escaping to atmospheric pressure P_2 equals 100 kPa (one atmosphere), the energy released can be computed as follows. Because PV^k is a constant,

$$\textbf{(5)} \qquad V_2 = V_1\left(\frac{P_1}{P_2}\right)^{\frac{1}{k}}$$

$$= 0.04\left(\frac{15\,000}{100}\right)^{\frac{1}{1.4}}$$

$$= 1.43 \text{ m}^3$$

Substituting in the work equation:

$${}_1W_2 = \frac{\left(100\right)\left(1.43\right) - \left(15\,000\right)\left(0.04\right)}{1 - 1.4}$$

$$= 1.142 \text{ MJ}$$

This energy (somewhat more than 1 MJ) could be evaluated in comparison with the 44 MJ of energy available by combustion of 1 kg of gasoline, of 38 MJ from 1 m^3 of natural gas or of 32 MJ from 1 kg of coal.

Evaluating Hazards of Explosive Pressure Release

The critical point of explosive pressure release is a very important factor in determining the hazard magnitude of high pressure leak tests. However, calculation of available stored gas energy is necessary for a thorough analysis of the potential hazard. This calculation includes two important considerations: (1) the amount of energy stored in the compressed gas and (2) the rapidity with which this energy is released.

The amount of energy stored in a noncombustible compressed gas can be approximated by Eq. 6:

$$\textbf{(6)} \qquad E = \frac{P_1V_1}{1 - K}\left[\left(\frac{P_2}{P_1}\right)^{\frac{K-1}{K}} - 1\right]$$

where K is the ratio of specific heat C_p at a constant pressure to that of a constant volume C_v, where P_1 is initial absolute pressure, V_1 is initial volume and P_2 is final pressure (100 kPa or 1 atm). This equation is based on the ideal gas law and isentropic expansion. At high pressure (e.g., above 20 MPa) where the deviation from an ideal gas may be appreciable, the equation is still valid provided one divides the right hand side by 2, the so-called compressibility factor found in gas handbooks.

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Applying Eq. 3 to helium, for example, one obtains a stored energy of 1.1, 12 and 106 $MJ \cdot m^{-3}$ for initial pressures of 1, 10 and 100 MPa, respectively. The stored energy can be converted to 2,4,6-trinitrotoluene (TNT) equivalents by using the conversion factor of 2.38×10^{-10} tons of TNT per joule. Also evidenced in Eq. 3 is the fact that a high volume, low pressure vessel can contain the same stored energy as a low volume high pressure vessel. Therefore, it can present a hazard of similar magnitude.

Of critical importance is the rapidity with which the energy release occurs. For the purpose of hazard definition, the extreme case of total and instant removal of gas confinement is used. The sudden release of energy is transmitted through the air in the form of a shock wave, generated by the sudden displacement of air surrounding the vessel. The shock wave carries with it measurable overpressure, varying with the intensity of the initial displacement. This shock wave, however, is never greater than the pressure that caused the displacement. The shock wave diminishes as a factor of distance.

Human Injury from Shock Wave Overpressures

It has been established that no damage will occur to a human body when it is subjected to shock overpressure of not more than 17 kPa (2.5 $lb_f \cdot in.^{-2}$). Body displacement can occur with shock wave overpressures of 20 to 35 kPa (3 to 5 $lb_f \cdot in.^{-2}$). However, a human body can be subjected to shock overpressures as high as 35 kPa (5 $lb_f \cdot in.^{-2}$) without injury to the internal organs. Above 35 kPa (5 $lb_f \cdot in.^{-2}$), eardrum rupture can occur. Permanent lung damage will be experienced with shock wave overpressures of 100 kPa (15 $lb_f \cdot in.^{-2}$ or 1 atm). Fatalities will occur with increasing probability with shock wave overpressures above 250 kPa (35 $lb_f \cdot in.^{-2}$).

The distance from the source of a shock wave at which personnel will be subjected to 35 kPa (5 $lb_f \cdot in.^{-2}$) overpressure is selected as the minimum safe distance. Because injury can occur from body displacement against the ground or nearby structures, personnel must be protected from direct exposure to a 35 kPa (5 $lb_f \cdot in.^{-2}$) shock. No one except the minimum crew necessary to conduct leak tests should be allowed inside the area when the vessels are being pressurized.

Hazards of Vacuum Testing

Evacuated systems, while not generally considered hazardous, involve the dangers of implosion or the possibility of personnel entering a vessel which, even though it has been vented to the atmosphere, does not contain enough breathable air to sustain life. Most vacuum testing involves gases such as helium, nitrogen and hydrogen, which will not support life. The same general precautions of handling pumping equipment, compressed gases, sight glasses etc. apply to vacuum testing as well as pressure testing.

Hazard of Implosion of Systems of Vessels Evacuated for Leak Testing

Implosion is the collapse of a pressure boundary or the walls of a containment vessel or structure when evacuated and subjected to atmospheric or higher external pressures. Many vessels and chambers are made for use under vacuum to simulate high altitude or outer space conditions where the maximum pressure differential that will ever be applied across their boundaries is 100 kPa (1 atm) of external pressure. Systems fabricated of thin wall materials, glass or foils cannot withstand high external or internal pressures.

For example, although they are not internally pressurized, glass bell jars that are evacuated can become a dangerous source of flying glass as a result of implosions. Pieces of flying glass, propelled by a pressure difference of about 100 kPa (1 atm), will travel great distances unless they should happen to collide with a safety shield or glass pieces coming from the opposite direction. The hazard of personnel injury by flying glass becomes particularly serious when the capacity of the glass vessel exceeds about 30 L (1 ft^3). For this reason, all evacuated bell jars should be enclosed in some type of safety shield.

Safety shields should be used on small thin wall vessels and glass bell jars under all vacuum conditions if an implosion hazard exists. The pressure differential between atmospheric pressure (101 kPa or 1.01 atm) and an absolute pressure of a typical vacuum (100 Pa or 0.001 atm) is essentially equal to atmospheric pressure (100 kPa or 1 atm). Any additional increase in pressure differential is negligible as the contained vacuum is further evacuated from 100 Pa to 1 Pa. Most of the atmospheric pressure is thus exerted on the bell jar or thin wall system when rough evacuation takes place. The

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increase in pressure difference resulting from further pumping to obtain a high vacuum is very small. Thus, it is a mistake not to use bell jar safety shields for any but the most moderate vacuum.

Vacuum Vessel Design

Vacuum vessel design may be divided roughly into two parts: (1) physical design, which is chiefly concerned with design for strength and satisfactory mechanical operations and (2) functional design, which is in the realm of vacuum engineering. Unless a thorough understanding of all the vacuum process variables is obtained, the finest mechanical design will not ensure satisfactory results when the equipment is placed in operation. The final design of a vessel, as in all engineering work, represents a number of compromises between conflicting conditions. The designer must consider all factors involved, both physical and functional, and then endeavor to reach the optimum solution. Where vacuum vessels do not come under *ASME Boiler and Pressure Vessel Code* requirements, it is recommended that the *ASME Boiler and Pressure Vessel Code* be used whenever applicable.[14]

Pressure Proof Testing of Systems before Leak Testing

Before undertaking leakage measurements, large systems may require proof testing to determine their capability to withstand leak test pressurization. For example, the *ASME Boiler and Pressure Vessel Code* (Section I, "Power Boilers"; Section III, "Nuclear Vessels"; and Section VIII, "Unfired Pressure Vessels")[14] specifies that all vessels should be hydrostatic proof tested to 1.5 times the maximum allowable working pressure.

The alternative to hydrostatic proof testing with water is to perform a pneumatic proof test to 1.25 times the maximum allowable working pressure. The pneumatic proof test may be performed by pressurizing with gas to a high pressure while all personnel are removed from the test area. The disadvantage of the proof test made with gas or air pressure is that if the system bursts during testing, considerable damage can result.

The alternative to proof testing with pressurized gas is to make a hydrostatic pressure proof test in which the system is pressurized with water.) Because water is relatively incompressible under pressure (as compared with gases), the energy released when a system bursts under water pressure is far less than when the system bursts under an equal gas pressure. On the other hand, if hydrostatic testing is performed before leak testing with gaseous tracers, any small leaks in the test system will become clogged with water. Therefore, if at all possible, hydrostatic testing should not be performed on test vessels or systems where the allowable leakage rate is less than 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}).

Codes and Requirements for Testing of Pressure and Vacuum Vessels

The most valuable source of information for the guidance of the engineer in the matter of physical design is the *ASME Code*, which is issued by the American Society of Mechanical Engineers and governs the design of unfired pressure vessels.[14] This *ASME Code* is the result of the contributions of many authorities representing designers, builders and users of vessels. The *ASME Code* rules and procedures have safe operation as their fundamental objective.

The mandatory pressure or vacuum vessel requirements of the states, municipalities and insurance companies involved should be studied, as it may become necessary to have the vessel *ASME Code*-stamped. Under these conditions, the *ASME Code* must be adhered to, Code calculations and design submitted to the proper authorities for approval and the vessel fabricated by those companies holding an *ASME Code* Certification of Authorization for the manufacturing involved. Chemical analysis and mechanical properties of all material that is under *ASME Code* rules are required and vessel manufacturers must have verified material certifications from the supplier. The *ASME Code* vessel or component inspection and stamping verification must be done by an authorized inspector holding a valid and current National Board commission (from the National Board of Boiler and Pressure Vessel Inspectors, Columbus, Ohio) in the area involved and who is employed by an authorized inspection agency. In addition, the *ASME Code* pressure vessels must be fabricated and manufactured under a controlled manufacturing system and quality assurance program as outlined in the manufacturer's quality assurance manual.

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PART 8. Preparation of Pressurized Systems for Safe Leak Testing

Methods for Leak Testing of Pressurized Systems (without Tracer Gases)

Pressure vessels and pressurized systems and components are designed to contain fluids at atmospheric or higher than atmospheric pressures. Pressure systems are commonly subjected to hydrostatic, hydropneumatic or pneumatic pressure proof tests during their manufacture, erection or periodic inservice maintenance inspections. Proof tests are made with pressurized liquids, with liquids and gases or with gases under pressures adequate to stress the containment structures to ensure their integrity. These tests often provide evidence of locations of leaks or indicate the presence of leakage by changes in pressure or fluid flow rates. Similar tests are also made on joints or sections of transmission line pipe following welding of longitudinal seams in pipe mills and on completed sections of pipelines following girth welding. Proof testing by pressurizing is used to ensure structural integrity and may indicate leak locations or leak tightness.

The most sensitive leakage rate testing is done by pressurizing the pressure vessels, components or systems with gases (or gaseous mixtures containing tracer gases) to establish a pressure differential across the containment boundary. The rate of leakage can often be increased by pressuring up, a technique in which internal pressure is raised to increase the rate of flow of gas through leaks and thus permit faster or more sensitive leak testing. The presence of leakage can then be detected (1) by measurement of pressure changes within the pressurized system or in an enclosure containing the pressurized components under test, (2) by input flow rates required to maintain pressure at constant levels or (3) by sensitive detection of specific tracer gases passing through the leaks.

Personnel for Pressure Testing and Leak Testing of Pressurized Systems

The best equipment that can be devised and assembled for pressure tests and leak testing of pressure vessels and systems is useless without properly trained and competent leak testing personnel. Training, although extremely necessary, cannot take the place of intelligence and clear thinking that is often referred to as horse sense, ingenuity, resourcefulness, imagination or innate ability. In addition, special training and caution are essential to prevent accidents or possible disastrous pressure vessel explosions, to avoid exposure of personnel to toxic tracer gases and to avoid asphyxiation where atmospheric oxygen has been displaced by accumulations of tracer gases and mixtures that do not support life. Where flammable or toxic pressurizing gases or liquids are used, full precautions must be taken to prevent fires, explosions or contamination of the atmosphere with toxic gases or gases that are flammable in air. Leak testing of pressurized systems requires that test personnel be trained, be intelligent and have considerable experience in operations performed under adverse conditions and with temporary equipment arrangements used only during leak testing.

Development of Techniques for Testing of Pressure Vessels

Until recent years, leak testing of most pressure vessels was performed in a relatively crude manner. Hydrostatic and pneumatic pressure tests were performed primarily to ensure the structural integrity of pressure vessels. Many pressure vessels are fabricated in accordance with the recommendations of the *ASME Boiler and Pressure Vessel Code*.[14] This code was prepared by the Boiler and Pressure Vessel Committee, established in 1911 by the American Society of Mechanical Engineers (ASME). The purpose of the Committee is to formulate standard rules for the construction of steam boilers and other pressure vessels. The Committee

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establishes safety rules governing the design, fabrication and inspection during construction of boilers and unfired pressure vessels and interprets these rules when questions arise regarding their intent. The *ASME Boiler and Pressure Vessel Code* provides a *Standard Recommended Guide for the Selection of a Leak Testing Method* (SE 432).[15]

Leakage has become a serious concern in the fabrication of nuclear reactors and components, as well as for vessels to contain lethal substances. Leak testing is also required on vessels used in the processing of materials that are affected by the presence of contaminants that react with the product they contain. Similar guides have been developed for inspection of pressure equipment in other industries. For example, the American Petroleum Institute (API) provides quidelines and recommended practices with information on pressure vessels and components of chemical plants and petroleum refineries. Inspectors are required to have complete knowledge of the requirements and recommended practices applicable in the specific industry in which the pressure vessels will be used. These inspectors often have responsibility for both leak testing and nondestructive testing of new construction and of plant facilities in use or during maintenance shutdown periods.

Mechanisms of Material Failures at High Pressure

Many people do not realize the hazards associated with hydrostatic testing in the higher pressure ranges. Materials that ordinarily are ductile can fail in a brittle manner at low temperatures. Small defects inherent in the grain structure, poor quality workmanship in fabrication or faulty design may, when the material is stressed, start a local crack that can no longer be arrested by the ductility and toughness characteristics of the material. Brittle fracture usually occurs at high stress levels and is more likely to occur in thick plate than in thin plate. This thickness effect is due in part to the increased restraint to plastic flow provided by the component thickness and in part to the coarse grain structure. When hot thick plate passes through the hot working rolls, the interior region receives less hot working and grain refinement than the near surface layers. As a result the center of the thick plate has a structure more nearly similar to that of the cast ingot from which the plate was wrought.

Effects of Pressure Vessel Wall Thickness and Temperature

There is not exact thickness or accompanying pressure above which brittle fracture will occur and below which ductile fracture will occur in hydrostatic testing of a vessel. However, caution should be exercised when the wall thickness is above 40 mm (1.5 in.) and when the pressure is above 10 MPa (1.5×10^3 $lb_f \cdot in.^{-2}$). When testing vessels that fall into this category, it is good, safe practice to ensure that water used for hydrostatic testing be at a temperature of at least 38 °C (100 °F). In addition, no pressure should be exerted on the vessel until the wall temperature both inside and outside is about the same as that of the pressurizing liquid, usually water. This precaution has a twofold effect: (1) there is less chance for the metal to fail in a brittle manner when the temperature of the wall of the vessel is close to the temperature of the contained liquid and (2) there is less air entrained in the water at a temperature of at least 38 °C (100 °F).

Minimum Temperature Limit for Leak Tests of Thick Walled Steel Vessels

When testing vessels with wall thicknesses above 40 mm (1.5 in.) and pressures less than 10 MPa (1.5×10^3 $lb_f \cdot in.^{-2}$) and where vessels are constructed of steels whose resistance to brittle fracture at low temperature has not been enhanced, test temperatures above 18 °C (65 °F) should be used to minimize the risk of brittle fracture during the test. Again, the test pressure should not be applied until the vessel structure (inside and out) and its contents are at about the same temperature.

Procedure for Heading Up Vessels for Pressure Tests

Before application of pressure within vessels or systems to be subjected to pressure tests, it is essential to close and seal all openings in the vessel pressure boundary so that pressurizing fluids do not escape or leak. The operation of assembly (also known as heading up) of a vessel for pressure testing must be done with adequate care to ensure safety. There are small details, many of which seem insignificant but could be potential hazards, that must be given careful attention. The small details are items that usually cause most of the problems because they are the most easily overlooked. A checklist type of test procedure is recommended to ensure that details are not overlooked and that safe practices are followed.

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Precautions during Installation of Blind Flanges and Covers

The first item to consider when planning the installation of blind flanges and covers for opening and open connections in pressure vessels before pressure testing or pressure leakage testing is the material to be used for these closures when heading up the vessel. Blind flanges and covers, as well as the bolts or studs with which they will be attached, must be of the proper material, thickness and size. If the cover is burned or sawed from plate, the outside diameter should have no notched areas that could serve as points of stress concentration or nuclei for crack propagation.

Precautions in Selection and Installation of Flange Bolts for Pressure Tests

Bolting or studding is a vital area for careful safety conditions. Carbon steel bolts, studs and nuts are generally recommended for attaching flange covers to pressure vessels for working pressures below 1.7 MPa (250 $lb_f \cdot in.^{-2}$) for tests at temperatures below 200 °C (400 °F). For temperatures exceeding 200 °C (400 °F), alloy steel bolts, studs and nuts are recommended regardless of the test pressure. For pressures exceeding 1.7 MPa (250 $lb_f \cdot in.^{-2}$), only alloy steel studs or bolting should be used. When alloy steel studs, bolts and nuts are necessary, their thread pitch should be not less than 3 mm (eight threads per inch). Loading to be applied to studs or bolts is recommended by suppliers of the various types and sizes of flanges and gaskets. Bolt or stud loading is generally expressed in terms of the torque required to tighten a nut or bolt to give a specific longitudinal stress (megapascal) in the stud or bolt for the specific stud or bolt material and cross section. Also specified by suppliers are the proper gasket pressures in megapascal or pound force per square inch. One important precaution is to determine whether or not the stud or bolt torque recommended applies for a lubricated or a dry bolt or stud. Lubrication of bolt or stud threads results in a great (and variable) increase in the actual stud or bolt stress and in the applied gasket pressure, for any specified level of stud or bolt torquing.

Precautions in Tightening Studs or Bolts on Gasketed Flanges or Covers

It is important that a flange be tightened evenly so that equal pressure is applied to the gasket. Having the flange cocked to one side by tightening one side more than another will almost always result in a gasket blowout.

When bolting up, it is necessary that every thread on the nut be engaged by a thread on the bolt or stud or, in machinist's terms, one must *have a full nut*. Be cautious in situations where the opening consists of a pad on the vessel with bottom tapped holes for studs. Assume that a pad is headed up for testing and that the studs are of different lengths. Or perhaps the studs are the same length and some of them are not threaded far enough into the tapped holes. A rule of thumb is that a stud must be threaded in to a depth equal to or greater than its diameter. There is only one way to be sure the threaded attachment is safe. Remove the bolts or studs from the threaded hole to see how many threads were engaged. Many times the person doing the bolting will not be the one standing beside the tank watching the gage pressure increase during the test. This is unfortunate, for the installer might be much more careful if he or she expected to be present for the test. Under no circumstances should a stud not driven to the proper engagement depth in blind hole be cut off at the nut end. Each stud or bolt should be turned into the threaded hole to provide a uniform length of threaded connection to the proper depth. Failure to ensure adequate depth of engaged threading results in an unacceptable and highly dangerous condition that could result in catastrophic failure when test pressures are applied.

Precautions in Selection and Installation of Gaskets for Pressure Tests

The choice of gasketing is important and vital for safety in pressure testing of vessels and systems with gasketed attachments, flanges and instrument connections. In most cases, a soft rubber gasket may be sufficient for low pressure testing. However, as the test pressure increases the gasket strength must be increased to prevent gasket failure where a portion of gasket or a small stream of high pressure liquid may be expelled with considerable force. For low pressure testing, a flat elastomer gasket of 60 to 70 durometer will make a safe seal. For

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higher pressures, one can use the same type of gasket, reinforced with some type of fiber. Beyond the range of application of the elastomeric gaskets, an asbestos or some other fiber gasket can be used. For very high pressures, a metal-to-metal seal such as the ring type joint or other patented seals must be used.

Proper gasket width and thickness are important, particularly with high pressures. With a fixed bolt load, a gasket that is too wide will result in low gasket pressure and consequent saturation and blowout. When a gasket is too narrow, a high gasket pressure will result and either the gasket will be crushed to uselessness or the flange may become grooved or warped. There are many flange designs on the market and it is important that correctly proportioned gaskets of suitable material and thickness be used to produce the correct ratio between effective bolt areas and the gasket contact surface areas. The suppliers of gasket materials generally have published data for proper application of their products. One should never exceed the recommended operating conditions for a gasket. A gasket failure may be a major eye hazard because pieces of gasket or a jet stream of liquid of gas under high pressure could cause a serious eye injury. One should always avoid being in direct eye line with a gasket while a vessel is being pressurized. Safety glasses and face shields should be worn while inspecting any vessel under pressure. A good means of protection when approaching the maximum design conditions of a gasket would be to wrap the outside diameter of the flange and cover with rope and surround the flange with a secured metal shield of at least 2 mm (0.08 in.) thickness.

Installation and Care of Sight Glasses for Pressure Tests

Certain precautions must be observed in installing and using sight glasses. Sight glass ends should be cut square and free of chips, scratches and rough edges. Care must be taken to protect the glass from scratches or severe deformation that might cause failure by explosion. A small scratch on the surface can greatly weaken the glass. Deformations caused by objects bearing against the glass or by improper tightening of the flange bolts can cause serious difficulties. It is important that the temperatures at testing be held constant or allowed to vary slowly enough to keep all parts of the sight glass assembly at approximately the same temperature to avoid localized stresses in the glass. Properly designed, applied and installed safety valves, maintained in good operating condition, are essential to the safety of personnel and the protection of equipment during pressure leak testing and during abnormal operating conditions in service. Inspections should be made of safety valves and overpressure relief devices to make sure that their performance meets the requirements of a given test operation and those for a given installation in operating equipment.

Functions and Types of Safety Valves and Pressure Relieving Devices

Pressure relieving safety devices can be divided into five basic classifications: (1) spring loaded devices, (2) weight loaded devices, (3) pressure loaded devices, (4) pilot operated devices and (5) rupture disks. All of these types of devices are designed to function automatically at a predetermined set pressure to prevent excessive overpressures in the equipment on which they are installed. The term safety valve is often used loosely to indicate any or all of these types of pressure relieving devices. Normally, safety valves and their discharge systems are used for pressure vessels and equipment designed for a maximum allowable working pressure in excess of 100 kPa (1 atm or 14.7 $lb_f \cdot in.^{-2}$ absolute). Table 9 lists specifications and codes applicable to pressure relief devices.

The following terminology and definitions identify the devices in each of the preceding categories.

1. Safety valves are automatic spring loaded pressure relieving devices actuated by the static pressure upstream of the valve and are characterized by rapid full opening or pop action. Safety valves are used on steam boilers, drums and super heaters. They may also be used for general air, steam and pressurized gases during service or in leak testing.
2. Relief valves are automatic spring loaded pressure relieving devices actuated by the static pressure upstream of a valve that lifts in proportion to the increase in pressure over the operating pressure. Relief valves are used primarily in systems filled with liquids.
3. Safety relief valves are automatic spring loaded pressure relieving devices actuated by the static pressure upstream of the valve. They are characterized by rapid full opening or pop action on gas or vapors and are suitable for use either as a safety valve or as a relief valve, depending on the application. There are two types of

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safety relief valves. Conventional safety relief valves are constructed in such a manner that the back pressure on the downstream side of the valve affects the action of the valve. Balanced safety relief valves have been balanced by the addition of a pressure balancing mechanism (bellows, piston or both) to decrease the valve's sensitivity to change in back pressure.

4. Pilot operated safety relief valves are pressure relief valves in which the major relieving device is combined with and is controlled by a self-actuated pilot relief valve. Pilot operated safety release valves consist of two basic units: a pilot or control unit and the main valve. These two basic units are mounted either on the same or on separate connections, depending on their design. The pilot is a spring loaded valve that senses the pressure differential and causes the main valve to open and close.
5. Pressure and vacuum vents are automatic pressure or vacuum relieving devices actuated by the pressure or vacuum in the protected vessel or tank. These pressure vacuums vents fall into two main categories: weight loaded pallet vents and pilot operated vents.
6. Rupture disks are thin diaphragms usually held between special flanges. They are designed to rupture at a predetermined pressure so as to relieve pressure from a vessel or system being protected.

Terms Related to Applications of Pressure Relief Devices

The following terms are related to the design and application of safety valves and the pressure systems on which such valves may be applied.

Maximum allowable working pressure is defined in the construction codes for pressure vessels. The maximum allowable working pressure depends on the type of material, its thickness and the service conditions set as the basis of design. The vessel may not be operated above this pressure or its equivalent at any metal temperature other than that used in specifying its design. Consequently, for that metal temperature, it is the highest pressure at which the primary safety valve is set to open.

The *operating pressure* of a vessel is the gage pressure to which the vessel is usually subjected in service. A processing vessel is usually designed for a maximum allowable working pressure that will provide a suitable margin above the

TABLE 9. Typical specifications and standards for pressure relief devices, including those applicable in petroleum refineries.

Issuer	Specification or Standard
API	Bulletin 2521, *Use of Pressure-Vacuum Vent Valves for Atmospheric Pressure Tanks to Reduce Evaporation Loss*
	Guide for Inspection of Refinery Equipment: Chapter 5, *Preparation of Equipment for Safe Entry*
	RP 520, *Recommended Practice for the Sizing, Selection and Installation of Pressure-Relieving Systems in Refineries*
	RP 521, *Guide for Pressure-Relieving and Depressuring Systems*
	RP 576, *Inspection of Pressure-Relieving Devices*
	Standard 526, *Flanged Steel Pressure Relief Valves*
	Standard 527, *Seat Tightness of Pressure Relief Valves*
	Standard 620, *Design and Construction of Large, Welded, Low-Pressure Storage Tanks*
	Standard 2000, *Venting Atmospheric and Low-Pressure Storage Tanks Nonrefrigerated and Refrigerated*
ASME	*ASME Boiler and Pressure Vessel Code:*
	Section I, *Power Boilers*
	Section IV, *Heating Boilers*
	Section VI, *Recommended Rules for Care and Operation of Heating Boilers*
	Section VII, *Recommended Rules for Care of Power Boilers*
	Section VIII, *Pressure Vessels*
ASTM	A 216, *Standard Specification for Steel Castings, Carbon, Suitable for Fusion Welding, for High-Temperature Service*
	A 217, *Standard Specification for Steel Castings, Martensitic Stainless and Alloy, for Pressure-Containing Parts, Suitable for High-Temperature Service*
	A 351, *Standard Specification for Castings, Austenitic, Austenitic-Ferritic (Duplex), for Pressure-Containing Parts*
	F 1508, *Standard Specification for Angle Style, Pressure Relief Valves for Steam, Gas, and Liquid Services*
NBBPVI	NB 23, *National Board Inspection Code*
	NB 27, *National Board Rules and Recommendations for the Design and Construction of Boiler Blowoff Equipment*

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operating pressure to prevent an undesirable operation of the safety valve.

Set pressure is the inlet gage pressure at which the safety valve is adjusted to open under service conditions. In a liquid service, the set pressure is the inlet gage pressure at which the valve starts to discharge under the service conditions. In a gas or vapor service, the set pressure is the inlet gage pressure at which the valve pops under service conditions.

Cold differential test pressure is the gage pressure at which the valve is adjusted to open on the valve or leak test stands. This cold differential pressure includes the corrections for service conditions of back pressure, temperature or both.

Accumulation is the pressure increase over the maximum allowable working pressure of the vessel during discharge through the safety valve. It is expressed in kPa or $lb_f \cdot in.^{-2}$, or as a percentage of the maximum allowable working pressure (MAWP). Maximum allowable accumulations are established by the applicable ASME Codes for operating and fire contingencies.

Overpressure is the pressure increase over the set pressure of the safety valve. It is the same as the accumulation when the safety valve is set at the maximum allowable working pressure on the vessel. The overpressure may be greater than the allowable accumulation if the valve is set lower than the vessel maximum allowable working pressure. Likewise, if multiple safety valves are installed, some with staggered set pressures above the maximum allowable working pressure, the overpressure for the staggered valves will be less than the allowable accumulation.

Blowdown is the difference between the set pressure and the reseating pressure of the safety valve, expressed in kilopascal or as a percentage of the set pressure.

Lift is the rise of the disk in a safety valve.

Back pressure is the pressure on the discharge side of a safety valve.

Superimposed back pressure is the pressure in the discharge header before the safety valve opens.

Built up back pressure is the pressure in the discharge header that develops as a result of flow after the safety valve opens.

Causes of Improper Performance of Safety Valves

Corrosion is one of the basic causes of difficulties observed in operation of safety valves. Corrosion may be apparent in pitting of valve parts, in breaking of various parts of a valve, in deposits of corrosive residues that interfere with operation of moving parts and in general deterioration of the material in a safety valve. Leaking valves can allow circulation of corrosive fluids into the upper parts of a valve so as to contribute to corrosion of the movable parts of the valve.

Damaged seating surfaces on safety valves can contribute to improper safety valve action during service. API Standard 527-78, *Commercial Seat Tightness of Safety Relief Valves with Metal-to-Metal Seats,* gives acceptable leakage rates.[16] Seating surfaces on safety valve must be maintained to optical precision. Any imperfection of these seating surfaces will contribute to improper valve action in service, as during leak testing. Foreign particles such as mill scale, welding spatter, coke or dirt that get into the valve inlet and pass through the valve when it opens may destroy the precision seat contact required for leak tightness in most safety valves.

Valve chatter causes hammering that sometimes damages safety valve seating surfaces severely. Careful handling of the valve during all phases of maintenance, installation and disassembly is important. Bumping or dropping the valve during installation should be carefully avoided. all valve parts, particularly guiding surfaces, should be checked thoroughly for any type of fouling. Lubrication of all sliding surfaces with molybdenum disulfide compounds or graphite and grease is recommended for safety valve used in refinery service where valves and piping can sometimes become plugged by process solids such as coke and solidified products.

Causes of Leakage in Safety Valves

Leakage past the seating surfaces of a valve after it has been leak tested, installed and placed in service may be caused by inadequate maintenance or installation procedures such as misalignment of the parts. Leakage could also result by piping strains resulting from improper support or by complete lack of support of discharge piping. This leakage contributes to seat damage because it causes erosion or corrosion of the seating surface and thus progressively aggravates the leakage problem. Valves subject to vibration, pulsating loads, low differential between set and operating pressures and other circumstances leading to valve leakage should be inspected and tested more frequently than valves not operating under such conditions.

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Testing for Leakage in Safety Valves

A properly designed test block is important to facilitate setting and adjustment of each safety valve during its inspection and repair. Valve settings are generally set in the maintenance shop by using water, air or an inert gas such as bottled nitrogen as the leak testing medium. Care should be taken and some overpressure should be applied to the valve to be certain that the valve is opening at the proper set pressure. An audible leak can otherwise be misinterpreted as the set pressure of the valve. In most types of safety valves, a distinct pop occurs at the set pressure, making misinterpretation impossible. Incorrect calibration or lack of calibration of pressure gages is another frequent cause of improper valve setting. The pressure range of the gage used to set valves should be chosen so that the required set pressure of the safety valve falls within the middle third of the range of the pressure gage.

Safety Valve Inspection Standards

Because of the difficulty in obtaining absolute leakage tightness in most safety valves, valve manufacturers use a commercial leak tightness standard according to which they manufacture valves. Subsequent rough handling of the valve can destroy the commercial tightness and produce excessive leakage in the valve after it is placed in service. Rough handling can occur during shipment, maintenance or installation of the valve. Occasionally, safety valve manufacturers are in a position to assist the user in establishing inspection and test intervals for safety valve. Each manufacturer is familiar with the nature of the loading, the stress levels and the operating limitations of their particular designs, thus enabling them to suggest inspection intervals appropriate for their valve equipment. In some instances, the frequency of inspecting and testing safety valves used in service is established by regulatory bodies. This should be investigated for each locality to avoid any possible conflict between such regulations and the frequencies of valve inspection considered to be satisfactory on some other basis.

Advantages of Testing Safety Valves with Air or Nitrogen

Air or inert gas is generally used to test safety valves, relief valves and safety relief valves for both set pressure and for leakage tightness. In general, some means is required to blind the valve discharge. Leakage may be detected qualitatively by placing a thin membrane (such as a wet paper towel) over the outlet and noting any bulging of the membrane. A quantitative measurement can be made by trapping the leakage and conducting it through a tube submerged in water, so that bubble emissions can be observed. Leaking valves can also often be detected with ultrasonic leak detectors.

Limitations of Testing Safety Valves with Water

Testing of safety valves with water is usually limited to measuring the set pressure because very small leaks cannot be readily detected when using water as the test medium. Water tends to clog small leaks and prevent detection of leakage. For this reason, leakage rate and leak tightness tests of relief valves are usually made with air as the pressurizing medium.

Inspection of Safety Valves on Steam Boilers

Inspection of safety valves on steam boilers should be carried out in accordance with local regulatory requirements as well as in conformity with manufacturer's recommendations and operating company practice. Because Section I of the *ASME Code*[14] does not permit block valves between boilers and boiler safety valves, testing on the equipment must be done periodically by raising the steam pressure to pop the valves while the boiler is in operation. Precision calibrated pressure gages should be used during the test procedure. The accumulation and blowdown should also be noted. The *ASME Code* also requires that the boiler safety valves have a substantial lifting device by which the valve disk may be lifted from its seat when there is at least 75 percent of full working pressure on the boiler. This permits checking to be sure that the moving parts are free to operate.

Frequency of Inspection of Safety Valves

The inspection of safety valves provides data that can be evaluated to determine a safe and economical frequency for scheduled inspections. This frequency can be expected to vary greatly because of the different operating conditions and environments to which safety valves are frequently subjected. Usually the intervals between inspections are increased as a

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result of satisfactory operating experiences and are decreased where corrosion, fouling and leakage problems exist. Historical records reflecting periodic test results and service experiences for each safety valve are valuable for establishing safe and economical inspection frequencies.

A definite time interval between inspections should be established for every safety valve on operating equipment to ensure proper performance. The time interval should be sufficiently firm to ensure that the inspection is accomplished but it should be sufficiently flexible to permit revision and temporary waiving where justified by circumstances. The interval between inspections is normally determined by operating experience. Obviously, the interval between inspections of a valve in corrosive and fouling service conditions would be shorter than for the same valve in a clean and nonfouling service.

Where corrosion, fouling and other service conditions are not known and cannot be predicted with any degree of accuracy (as in a new type of process or in occasional use during leak testing), the initial inspection should be accomplished as soon as practical after operations begin to establish a safe and suitable testing interval. Safety valves in service should carry an identifying tag or plate. This identification is needed to minimize errors in testing and handling of safety valves. Identification of safety valves is essential in keeping accurate historical records on each valve.

Routine Checking of Safety Valve Set Pressure and Leak tightness

An important phase of the safety valve maintenance routine is to determine set pressure and leak tightness of the valve both in the as-received condition and after overhauling. A visual inspection of safety valves should be made as the valves are removed from the system or from a leak testing setup. Many types of deposits or corrosion products may be loose and drop out of the safety valve while it is being transported to the shop for inspection and repair, if needed. Any obstruction in the valve should be noted and corrected. Inspection of the piping or flange connections at the location of the safety valve should be done to detect evidence of corrosion, indications of thinning and deposits that may interfere with valve operation.

Determining Safety Valve Pop Pressure before Dismantling

Before the safety valve is dismantled, it is generally considered important to determine the pop pressure of the valve when removed from service. If the valve opens at the set pressure, it need not be tested further to determine the as-received relieving pressure. If the initial pop is higher than the set pressure, it is advisable to make a second test for pop pressure. If the valve then pops at about the set pressure, this indicates that the valve was probably stuck because of deposits. If the valve does not pop near the set pressure, this indicates that the valve setting was higher in error originally or that it may have been changed during operation. The as-is test pressure should be recorded for review and facilitation of any necessary corrective action.

Maintenance Procedures for Safety Relief Valves

When safety relief valves are to be given maintenance servicing, each valve should be carefully dismantled in accordance with its manufacturer's instruction manuals and recommendations. Proper facilities should be available for segregating valve parts as the valve is dismantled. At each stage in the dismantling process, the valve, stem, guide, disk, nozzle and other parts require visual testing. The bellows in balanced type valves should be checked for cracks or other failures that might permit leakage or affect valve performance.

Cleaning, Repair and Replacement of Safety Valve components

The valve parts that most often require cleaning are the nozzle, springs and seats. Deposits that are difficult to remove should be cleaned off with solvents or wire brushing or should be carefully scraped. The dismantled parts should be checked carefully at this time for wear and corrosion. Checking of valve components is important. It should be done carefully with the proper equipment calibrated for measuring valve dimensions and with frequent reference to the proper valve drawings and literature. Parts that are worn or damaged should be replaced or reconditioned. Parts such as damaged springs or bellows should be replaced without attempting repairs. The valve body and bonnet may be reconditioned by means considered suitable for repairs to other pressure containing parts of similar materials. After the valve has been inspected and reconditioned, it should be assembled in accordance with the manufacturer's instructions as to the order of assembly and the procedure for adjustment of the various parts.

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Setting Repaired Relief Valves to Required Pop Pressure

After a used relief valve has been reconditioned and reassembled, it is ready for the final spring adjustment to the required set pressure. The manufacturer's recommendations should be used as a guide in adjusting the spring to the correct setting. If a new pressure setting is required, the manufacturer's limits for adjustment of the spring must not be exceeded and applicable *ASME Code* requirements must be observed. It may be necessary to provide a different spring. After the final adjustment is made, the valve should be popped at least once to prove the accuracy of the setting. The final pop should be within the manufacturer's listed accuracy for the cold set pressure before the valve is approved for service. Allowance for hot setting should be made in accordance with the manufacturer's data.

Checking Reassembled Safety Valve for Leak Tightness

After a reconditioned relief or safety valve has been satisfactorily checked for conformance to the set pressure, it is then desirable to check the valve for leakage. Excessive leakage could lead to fouled or inoperable valves, hazard to personnel and equipment and possible loss of leak testing fluid or product from processing systems (see discussion of safety valve leakage). All necessary records for inspection, repair, assembly and resetting should be completed before the valve goes back into service. These records are important for effective future use of the valve. They will provide guidelines for replacement of valves and components as well as providing the historical record of the conditions and services under which the valve operated.

Need for Keeping Permanent Records for Safety Valves

A complete permanent record file should be kept for each safety valve in service. The record should provide specification data for the valve and a history of inspection and test results. The specification record is needed to provide basic information needed to evaluate the adequacy of the valve for a given leak testing operation or permanent installation. It also provides correct dimensional and material information to minimize shop errors and expedite repairs. Historical records showing dates and results of inspections on safety valves are necessary for a followup on the control phase of the program. One of the foremost reasons for keeping service records is that they provide a practical and realistic basis for maintaining safe and economical inspection intervals that provide safety to all operators using the valves.

Precautions with Venting Devices on Atmospheric Storage Tanks

Atmospheric storage tanks are widely used in petrochemical industries. Venting devices are usually mounted on top of these tanks to protect the tank from damage due to excessive internal pressure or from excessive vacuum. Venting devices are all to often taken for granted and forgotten once they have been installed. They must be considered when leak testing atmospheric tanks by pressure change or flow measurements. These relatively simple venting devices will normally work properly for long periods with little attention, but if one fails, it can result in catastrophic failure of the tank and loss of its product.

The two main types of venting devices are breather vents and conservation vents. Breather vents or open vents usually take the form of an open pipe of predetermined size. They permit the equalization of pressure inside a tank with the varying external atmospheric pressure. In general, breather vents are used when the product stored has a flash point about 40 °C (100 °F) and evaporation losses are not a concern. The vent should be equipped with a return bend or weather head to exclude rainfall, both being equipped with screens to prohibit any entry of animals or any other foreign matter. Vents should be designed so that any condensate will drain back into the tank without creating a trap or pocket. The vent should be located so there is the least chance of encountering an ignition source when flammable materials are stored within the tank. Additionally, the vent should in no case be smaller than the discharge or withdrawal connection. It is bad practice to manifold vents. Each tank should have its own vent.

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Conservation Vents or Breather Valves

Conservation vents or breather valves isolate a tank until specific pressure or vacuum levels are reached (relative to atmospheric pressure). The standard conservation vent usually relieves at pressure or vacuum gage levels of 215 Pa gage (0.5 oz·in.$^{-2}$ or 0.865 in. of water). The pressure setting is determined by the weighting of pallets. The heavier these weights are, the greater the pressure difference must be to open them. Conservation vents can reduce evaporation losses by 50 percent over breather vents and each additional increase of 400 Pa (1 oz·in.$^{-1}$) in the valve setting will further reduce the breathing losses by about 7 percent. These conservation vents are used where evaporation loss is a concern and/or when the product being stored has a flash point equal to or less than 40 °C (100 °F).

Functions of Vents

With modern welded metal tanks and roofs, storage tanks have become airtight vessels. Because of this, it is important to ensure that the tank has some means of equalizing the external and internal pressure. Normal venting devices do not eliminate evaporation losses but they do reduce these losses. The majority of evaporation losses are due to either the normal tank breathing or to the filling of the tank. The breathing of the tank refers to the action caused by increasing atmospheric temperature or decreasing atmospheric pressure. The increasing temperature causes the vapor pressure of the tank to increase until it is greater than the atmospheric pressure and the vapors of the tank are driven out until the pressure is equalized. The normal breathing cycle involves exhaling during the late morning and early afternoon and inhaling during the evening when the temperature decreases. Likewise, as atmospheric pressure decreases, the vapor pressure inside the tank becomes greater than the surrounding atmosphere and the vapors of the tank are driven out until the pressure is equalized. When the tank is being filled, the liquid coming in acts to displace the vapors in the tank, causing these vapors to be driven out. Both actions would cause a differential pressure far in excess of the normal design pressure of atmospheric tanks if the movement of vapors were prohibited and the tank acted as a closed system.

The vent reduces the evaporation losses by adding another resistance to the normal vapor movement; it does not prohibit the movement. The effect is that a pressure slightly below the design pressure is maintained on the tank. It makes it harder for the vapors to escape. The resistance is caused by an orifice effect in breather vents and by the pressure setting (the pressure required to open the pallets) on conservation vents.

Beside reducing evaporation losses, the vent is also a safety device. The safety aspect has priority over loss reduction. Safety is the first concern when selecting the proper vent. Other considerations necessary when determining the proper vent are filling and emptying rates for the tank, the size of the tank, the product being stored, the strength of the tank and the normal daily ambient temperature change rates.

Effects of Flame Arrestors in Vents

Flame arrestors consist of a group of tightly spaced metal plates placed at the entrance to a vent. They are intended to prevent a flashback of flame through a vent, which could cause an explosion of flammable products in a tank. For a flashback to occur, an ignition source must be present and the tank must be expelling flammable vapors. The theory of the flame arrestors is that they should dissipate enough of the heat energy to prevent a flame front from passing through them. However, many users now believe that a conservation vent will prohibit flashback just as well as a flame arrestor without the maintenance problems caused by a flame arrestor.

Therefore, a tight steel roof and a conservation vent may provide all the protection that is required. The negligible additional protection offered by a flame arrestor may not warrant assuming the maintenance problems and risk of tank damage as a result of a flame arrestor clogging up or prohibiting flow. This topic is discussed in Petroleum Safety Data Publication PSD 2210, *Flame Arrestors for Vents of Tanks Storing Petroleum Products,*[17] compiled by the Committee on Safety and Fire Protection of the American Petroleum Institute.

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PART 9. Exposure to Toxic Substances

The following discussion pertains to measurement and reporting of recommended limits of exposure to toxic substances by United States government agencies.

Threshold Limit Value and Time Weighted Average

The threshold limit value (TLV) is a recommended upper limit (ceiling) or time weighted average (TWA) concentration of a substance to which most workers can be exposed without adverse effect. This concentration may be designated as a ceiling (C_1) or time weighted average (TWA) concentration. The notation (SKIN) indicates that even though the air concentration may be below the limit value, significant additional exposure to the skin may be dangerous. Threshold limit values are quantified in *TLVs: Threshold Limit Values for Chemical Substances and Physical Agents in the Work Environment,* (third edition, 1971), its supplement or from documentation in the annual reports of the America Conference of Governmental Industrial Hygienists (ACGIH).[18]

NIOSH Water Quality Toxicity Ratings

The National Institute for Occupational Safety and Health Aquatic Toxicity ratings are published in *Water Quality Characteristics of Hazardous Materials.*[19] The format for this line is AQUATIC TOXICITY RATING: Tlm96 $\mu L \cdot L^{-1}$ where TLm96 is defined as the 96 h static or continuous flow standard protocol. Because of the lack of standardization and the wide variety of species investigated, ratings are used to give an indication of the toxicity of substances to aquatic life.

Hazardous Substances

Except as provided for certain export and import shipments, no person may offer or accept a hazardous material, as defined by the *Code of Federal Regulations [CFR],*[1] Title 49, for transportation in commerce within the United States unless that material is properly classed, described, packaged, marked, labeled, and in the condition for shipment as specified by 49 *CFR,* Parts 100 to 189. For transportation purposes, a hazardous material means a substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce and which has been so designated.

Basic hazard classes include compressed gases, flammables, oxidizers, corrosives, explosives, radioactive materials, and poisons. Although a material may be designated by only one hazard class, additional hazards may be indicated by adding labels or by other means.

It is essential, therefore, that all required labels(s) as well as the hazard class be known. Generally, poison must always be labeled as a poison regardless of the other labeling requirements in order that adherence to the prohibition against shipping poisons with foodstuffs can be assured.

Specific shipping names are designated for hazardous materials in regulations because of the presence of many nontechnical names or the use of archaic names for some materials.

Determination of the correct classification for transportation of materials is the responsibility of the shipper.

National Institute for Occupational Safety and Health criteria documents recommending environmental (occupational) exposures are currently available for various toxic substances encountered in leak testing. The reference citation (NTIS) is the National Technical Information Service, United States Department of Commerce, from which these publications are available.

Occupational Diseases

The National Institute for Occupational Safety and Health publication *Occupational Diseases — A Guide to Their Recognition*[20] (revised periodically) describes both biological hazards and chemical hazards and the harmful health effects of many substances used in industry. Most of the known occupational disease producing chemicals are listed by

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chemical groups, e.g., aliphatic hydrocarbons, alcohols, glycols. Listed also are occupations in which workers are potentially exposed to each toxic agent. Whether the exposure to the toxic agent constitutes a hazard depends on such factors as the concentration of the agent, how the agent is handled and used, duration of exposure, susceptibility of the worker to the agent and health protection practices adopted by management. Thus, all hazardous situations imply an exposure but not all exposures are hazardous.

Topics covered for each substance or group of toxic chemicals include the following: (1) description and chemical formula, (2) synonyms and common names for material, (3) potential mechanisms of occupational exposures, industries in which exposures can occur and worker occupations which may lead to exposures, (4) permissible exposure limits (if established), (5) routes of entry of toxic chemical into human body, (6) harmful effects of toxic substance, (7) symptoms and systemic effects of exposure, (8) medical surveillance recommendations, (9) special tests used or recommended to detect worker ingestion or response to toxic chemicals, (10) personal protective methods and (11) bibliography of pertinent references.

General warnings are given in other sections of this book, where experience indicates that possible hazards may exist. However, this volume is devoted to leak testing; its users are referred to qualified authorities on industrial safety, toxic substances, exposure limits, biological effects, and legal requirements and responsibilities. For advice, the user should refer specifically to plant safety rules and procedures; local, municipal, county, state and national laws and regulations; and qualified safety and health organizations and agencies.

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CHAPTER 5

Pressure Change and Flow Rate Techniques for Determining Leakage Rates

Charles N. Sherlock, Willis, Texas

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PART 1. Introduction to Pressure Instrumentation, Measurements and Analysis

Functions of Pressurizing Gases in Leak Testing

Atmospheric air and nitrogen are often used as pressurizing fluids in leak testing and leakage measurements. Their fluid pressure serves to create pressure differentials across pressure barriers or walls. This pressure differential, in turn, causes the pressurizing gas to flow, by various mechanisms, through leaks in the containment walls.

Leaks are the physical holes or passageways that may exist in wall materials, welds, mechanical seals or joints. The fluid that flows through the leak passageways constitutes leakage. The rate of leakage in turn is taken as a measure of the size of the leak.

In general, the higher the differential pressure, the greater the rate of leakage. With higher rates of leakage, the sensitivity of leak detection and leakage measurement is typically increased. Closed systems with air or other gas pressures above atmospheric pressure (101.325 kPa) respond to leakage by pressure changes (within closed systems) or require inflow of gas to maintain constant pressure conditions. These pressure changes or rates of fluid flow can be used to determine (1) the presence of leaks or (2) the rates of leakage, when internal volumes, fluid temperatures and other variables are known or can be measured accurately. The physical properties and characteristics of the pressurizing fluids must be known and the effects of fluid reactions to various test conditions must be calculated to make quantitative measurements of leakage rates. Pressurizing gases should obey the ideal gas laws. In some cases, the effects of water vapor and other gaseous materials that do not obey the general gas laws must be determined and their effects subtracted from the pressure measurements.

Compressibility of Gaseous and Liquid Fluids

Gases are frequently regarded as compressible and liquids as incompressible. Strictly speaking, all fluids are compressible to some extent. Although air is usually treated as a compressible fluid, there are some cases of flow in which the pressure and density changes are so small that the air may be assumed to be incompressible. Examples include the flow of air in ventilating systems and the flow of air around aircraft at low speeds. Liquids like oil and water

TABLE 1. Typical operating ranges and probable accuracy limits of pressure gaging systems.

Pressure Measuring Instruments	Ranges of Pressures		Accuracy Limits	
	SI Unit (kPa)	English Units	SI Units	English Units
Deadweight testing machines	2 to 350	(0.3 to 50 $lb_f \cdot in.^{-2}$)	typically about 0.003 percent[a]	
with various operating ranges	350 to 3500	(50 to 500 $lb_f \cdot in.^{-2}$)	typically about 0.003 percent[a]	
	3500 to 16 000	(500 to 2400 $lb_f \cdot in.^{-2}$)	typically about 0.003 percent[a]	
	16 to 80 000	(2400 to 12 000 $lb_f \cdot in.^{-2}$)	typically about 0.003 percent[a]	
Mechanical dial pressure gages	0 to 700 000	(0 to 100 000 $lb_f \cdot in.^{-2}$)	±0.066 to ±2 percent of full scale	
Quartz Bourdon tube gages	0 to 20 000	(0 to 3000 $lb_f \cdot in.^{-2}$)	±0.01 to ±0.02 percent of full scale	
Metal Bourdon tube gages	7000 to 140 000	(1×10^3 to 2×10^4 $lb_f \cdot in.^{-2}$)	see manufacturer's specifications	
Water U-tube manometer	0 to 7.5	(0 to 30 in. H_2O)	±1 Pa	(±0.03 torr)
Direct-reading mercury manometer	0 to 350	(0 to 100 in. Hg)	±80 Pa	(±2.5 torr)
Digital U-tube mercury manometer	0 to 285	(0 to 84 in. Hg)	±3 Pa	(±0.1 torr)
Digital aneroid capsule	35 to 3500	(5 to 500 $lb_f \cdot in.^{-2}$)	±0.05 percent of full scale	
Ion mass detector sensor	50 to 800	(7 to 120 $lb_f \cdot in.^{-2}$ gage)	10^{-5} $Pa \cdot m^3 \cdot s^{-1}$ leakage rates	(10^{-6} std $cm^3 \cdot s^{-1}$)

a. Traceable to US National Institute of Standards and Technology.

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may be considered as incompressible in many cases; in other cases, the compressibility of such liquids is important. For instance, common experience shows that sound waves travel through water and other liquids; such pressure waves depend on the compressibility or elasticity of the liquid.

Instrument Systems for Precise Pressure Measurements during Leak Tests

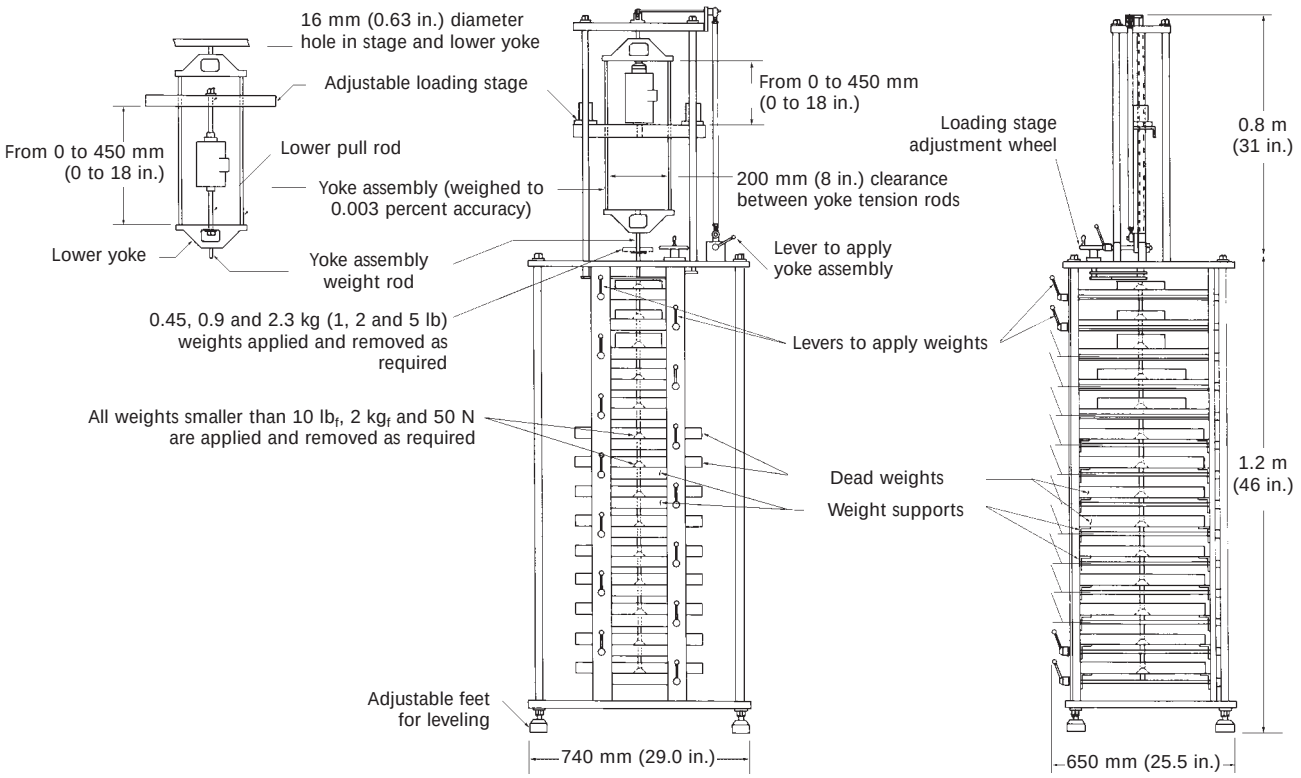
Quantitative and reproducible leakage rate testing by pressure change measurements depends critically on the control and measurement of test pressures applied to systems under test. The most precise pressure measuring instruments are deadweight testers. These are used most commonly only for calibrations of other pressure measuring instruments. Water or mercury manometers (U-tubes partially filled with liquid) are also used for calibration of other pressure gages and instruments. Other pressure measuring instruments include Bourdon gages; rapid response electrical output signal sensors used in potentiometric, capacitance, reluctance and piezoelectric pressure gages; spiral wound quartz crystal and wire resistance strain gages; and specialized electronic gages with digital output signals of pressure. Table 1 lists typical pressure gages used in leak testing of pressurized systems and indicates their typical pressure range and accuracies.

Deadweight Piston Calibration Standards for Pressure Measurements

The deadweight piston gage is a calibration standard for measuring pressures. Pressure or force per unit area is provided by known weights acting on the known area of the cylinder. Fluid pressure to be measured is applied against the bottom of the piston, developing enough force to lift the weights. Thus, the two factors of primary importance are the weights used and the effective area of the piston-and-cylinder combination. Figures 1 and 2 show a deadweight calibration machine.

Three types of deadweight piston gage are available: (1) simple piston pressure gage, (2) controlled clearance piston pressure gage and (3) reentrant piston pressure gage. The first is simple and most commonly used. The controlled clearance

FIGURE 1. Schematic of dead weight machine for calibrating force measurement devices.



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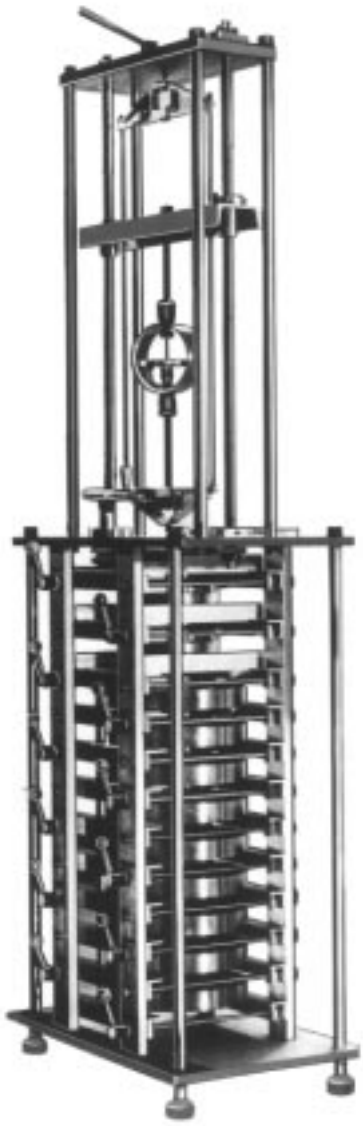
gage reduces errors caused by deformation of the cylinder because of the pressure in the cylinder. The reentrant gage is a compromise between the first two types of gages.

Factors Influencing Piston Gage Pressure Measurements

Temperature affects the dimensions of gage piston and cylinder. Gravitational force depends on the location of the instrument on the earth's surface and on its altitude above sea level. Air is a fluid and has a buoyant effect on objects (weights) submerged in it. Compressibility affects fluid density, which can affect calibrations if the pressure is measured at a level different from that of the piston face. All these effects are predictable and correction factors can be obtained from the various pressure gage manufacturers, the National Institute of Standards and Technology and the local weather bureau.

FIGURE 2. Dead weight machine for calibrating force measurement devices.



Measuring Fluid Pressure with Manometers

The manometer balances hydrostatic pressures with the weight of a column of liquid. Thus, the accuracy with which a pressure can be measured by a manometer depends on (1) the several factors that affect the weight of the fluid columns and (2) the accuracy with which the column heights can be observed. For the basic U manometer configuration, if both ends of the U-tube are open to the atmosphere, the same pressure acts on each side. Then the column of liquid on one side of the U-tube will exactly balance the column of liquid on the other side. The top surfaces of the two columns will be at the same level. However, if one leg of the manometer is subjected to a pressure greater than that applied to the other leg, the heights of the two liquid columns will differ. The difference in column heights will be proportional to, and a true measure of, the differences in pressure applied to the tops of the liquid columns in the two legs of the manometer.

The difference in the height of the liquid in the two legs is exactly the same whether (1) the diameter of the glass tube is the same in both legs or (2) the legs have different diameters, provided that the diameter of the smaller tube does not approach capillary diameters where surface tension effects have an influence on the height of the liquid columns. The mercury barometer is an example of a well type absolute manometer, where atmospheric pressure operates on the liquid in the open dish of the well whereas vacuum pressure acts on the top of the liquid column in the closed barometer tube.

Effect of Fluid Density in Manometers

When a manometer measures a pressure, the difference in the U-tube liquid column heights depends not only on the external pressures applied to the two sides of the U-tube but also varies with the density (mass per unit volume) of the liquid within the U-tube. To illustrate, suppose that three U-tube manometers contain oil, water and mercury, respectively, as their fluids. The difference in fluid column heights will differ in these manometers when subjected to the same differential pressure. The largest difference in column heights is observed with the

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low density oil, slightly less with water and considerably less with the high density mercury. The differential heights vary in a ratio of approximately 17 (oil), 14 (water) and 1 (mercury).

Silicon Based Pressure Sensors

Because of its role in the production of electronic integrated circuits, enormous research effort has been committed to understand, control and commercialize the electronic performance of silicon as a semiconductor. A byproduct of research has been an increased use of silicon as the sensing member for many types of electromechanical sensors.

Silicon's unique mechanical and electrical properties make it well suited for sensing various phenomena. Some of these properties are the following.

1. It has a strength-to-weight ratio five times greater than stainless steel.
2. It is as hard as quartz.
3. Its thermal conductivity is close to that of aluminum.
4. It has almost perfect elasticity, exhibiting no mechanical hysteresis.
5. It is readily machined both mechanically and chemically to achieve a required shape or profile.

Furthermore, silicon responds to light, magnetic fields, stress and temperature and is impervious to most media. Just as in its use as a semiconductor, it can be produced as pure single crystal silicon or it can be doped with various impurities to provide specific effects. When a micromachined silicon chip is used as a sensor it is a practical matter to include signal enhancing circuitry directly on the sensing element just as in integrated circuits.

Precision Pressure Measurements with Silicon Pressure Transducers

Some of the advantages of using a silicon chip as a strain gage or sensor for precision pressure measurements are the following.

1. It can be very small, which reduces package size.
2. It is highly stable for long term reliability.
3. It is inherently rugged, so it is practically immune to the effects of tilt and vibration.

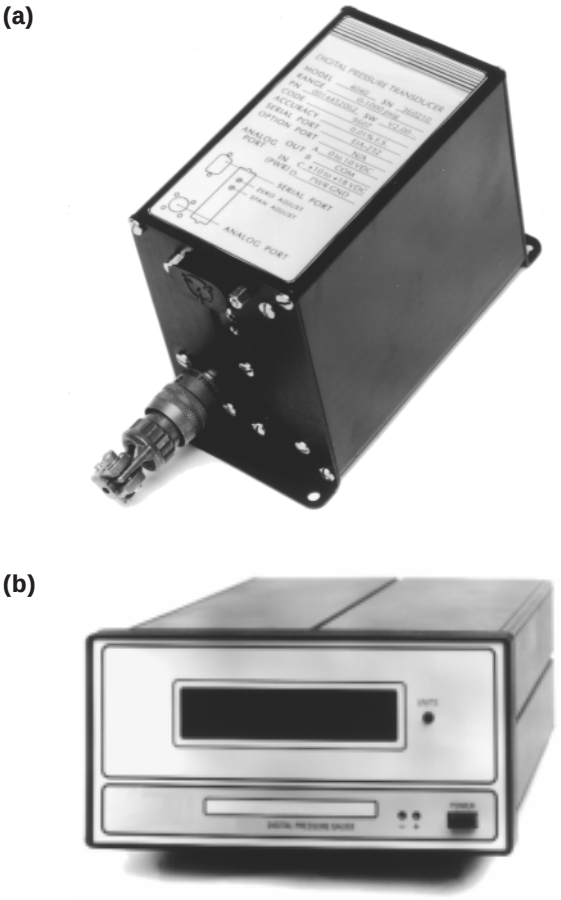
When a silicon pressure sensor incorporates temperature sensing the device can be characterized for pressure response over a range of temperatures. Using microprocessors, the unique pressure/temperature characterization for an individual silicon sensor can be temperature compensated to produce a pressure reading with a high degree of precision over an extended temperature range.

A digital pressure transducer (Fig. 3a) uses a silicon pressure transducer to provide pressure measurements with an accuracy of 0.01 percent of full scale over a temperature range of 15 to 45 °C (59 to 113 °F). The gage is available as absolute, bidirectional, compound, gage and vacuum types in full scale ranges as low as 0 to 2.5 kPa (0 to 10 in. H_2O) and up to 0 to 41 MPa (0 to 6×10^3 $lb_f \cdot in.^{-2}$). Several of these digital pressure transducers with different ranges can be connected in series for multiple test pressure ranges.

Another pressure instrument (Fig. 3b) uses a silicon pressure transducer and handles pressure ranges up to 0 to 70 MPa (0 to 1×10^4 $lb_f \cdot in.^{-2}$). Standard accuracy is typically 0.025 percent of full scale, with temperature compensation of 15 to 45 °C (59 to 113 °F).

A specialized variation of the digital pressure transducer is the precision barometer (Fig. 4a). This absolute device

FIGURE 3. Digital pressure gage system components: (a) digital pressure transducer; (b) console.



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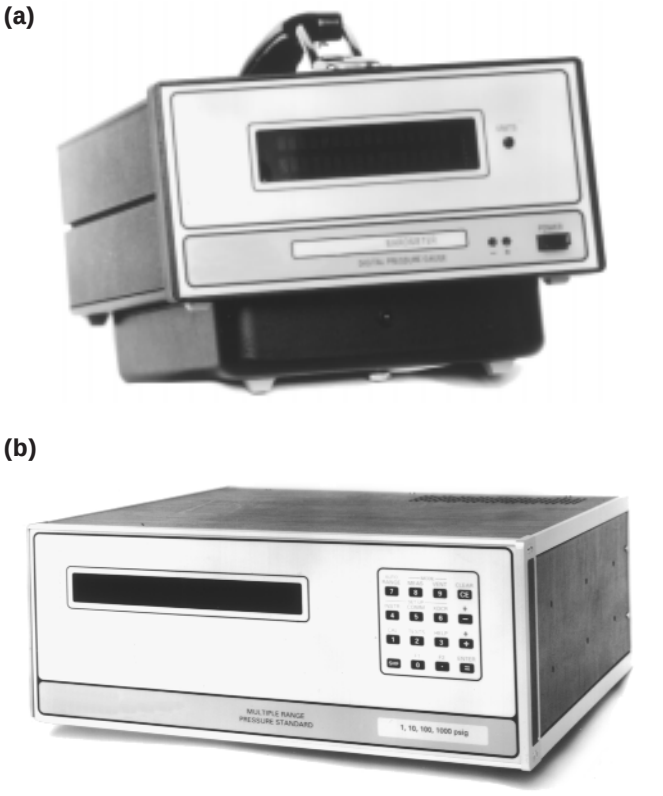
has a fixed pressure range of 75 kPa to 115 kPa (22 to 34 in. Hg) with a resolution of 0.34 Pa (1×10^{-4} in. Hg).

A practical instrument for testing or calibrating pressure devices of many different ranges is the multiple range pressure standard (Fig. 4b). This instrument incorporates from four to seven precision silicon pressure transducers inside a single chassis with a common central processing unit and user interface. Each transducer can be custom made to operate in any range from 0 to 2.5 kPa (0 to 10 in. H_2O) up to 6.9 MPa (0 to 1×10^3 $lb_f \cdot in.^{-2}$), each with an accuracy of 0.01 percent of full scale over the temperature range of 15 to 45 °C (59 to 113 °F). Each transducer is individually protected from overpressure by relief and shutoff valves. This system can be switched between *range hold* and *autorange*. In the *range hold* mode all tests are performed using a single range transducer, whereas in *autorange* mode the applied pressure is automatically directed to the internal transducer that will provide the highest level of accuracy for that pressure. In this mode operator or programmed switching between tests of different pressure ranges is eliminated.

FIGURE 4. Pressure measurement instrumentation: (a) barometric pressure gage; (b) multiple range pressure standard for calibrating pressure transducers.

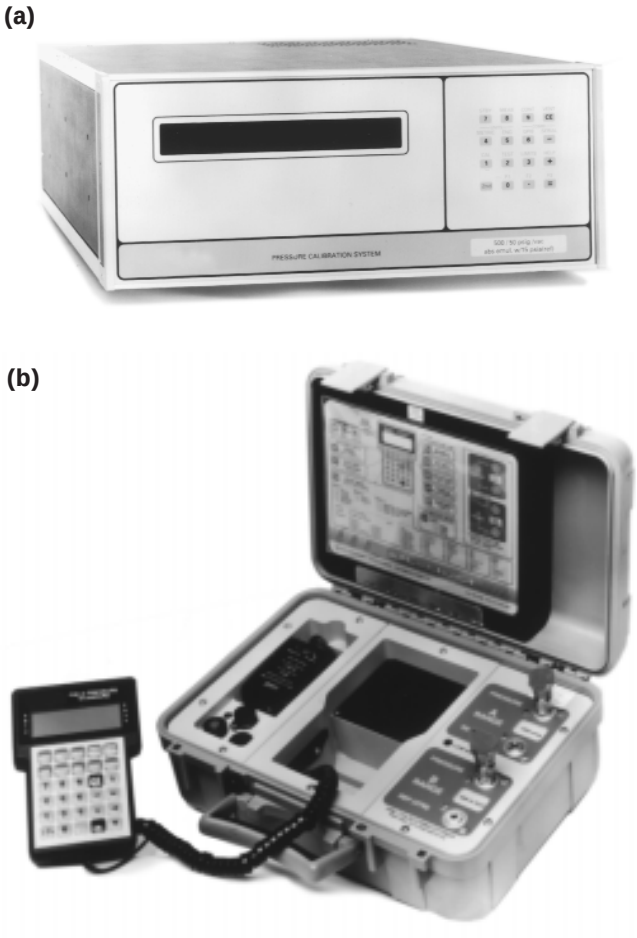


Precision Regulated Pressure Output

A pressure calibration system (Fig. 5a) finds application when precise pressure output in the range of 0 to 10.35 MPa (0 to 1.5×10^3 $lb_f \cdot in.^{-2}$) is required. The pressure calibration system has a measurement accuracy of up to 0.01 percent of full scale and a 0.002 percent of full scale control stability over the compensated temperature range of 15 to 45 °C (59 to 113 °F). The pneumatics module consists of from one to three internal silicon pressure transducers, the reed valve regulator, the valves and plumbing. The system's macro capability lets the user program up to 64 different test routines with up to 256 steps in each routine.

A high pressure control unit can extend the range of the pressure calibration system for precision regulated pressure up to 40 MPa (6×10^3 $lb_f \cdot in.^{-2}$). Both units are operated from the front panel of the pressure calibration system or

FIGURE 5. Pressure measurement instrumentation: (a) pressure calibration unit; (b) portable pressure standard.



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its communication ports. The high pressure control unit also uses a fully temperature compensated silicon pressure transducer with an accuracy of up to 0.01 percent of full scale. Control stability of the high pressure control unit is better than 0.01 percent of full scale.

Dual Range Precision Pressure Measurement in the Field

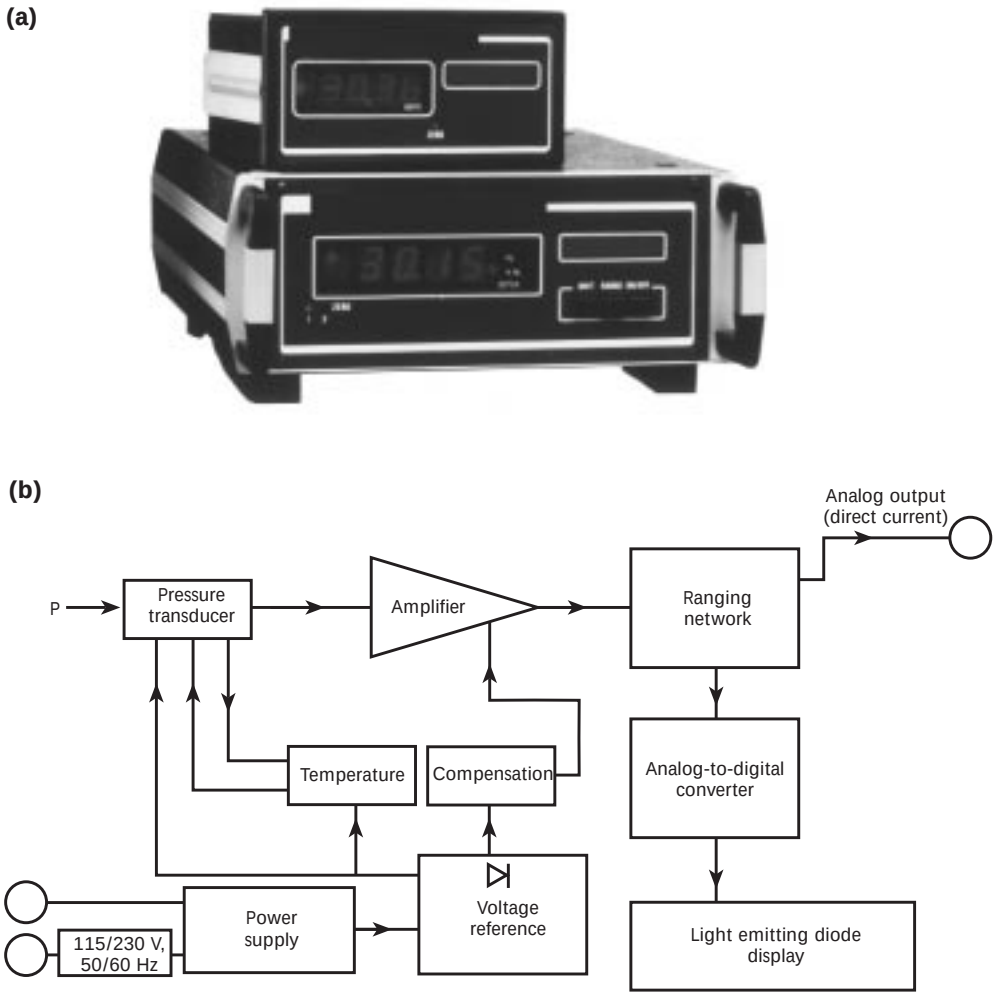
A field pressure standard (Fig. 5b) is suited for high accuracy pressure measurement requiring two different pressure standards or pressure types. Both pressure ranges use temperature compensated silicon pressure transducers, available for pressures from 0 to 2.5 kPa (0 to 10 in. H_2O), up to 40 MPa (6×10^3 $lb_f \cdot in.^{-2}$), and up to 0.01 percent of full scale accuracy. Either pressure range is available with an absolute, gage, compound, vacuum or bidirectional pressure transducer.

Digital Pressure Gages

A representative digital pressure gage (Fig. 6) has as its pressure sensing element a piezoresistive, strain gage transducer coupled to solid state circuitry. The transducer's integrated strain gage bridge is diffused on one side of a single-crystal silicon diaphragm. Application of the pressure to be measured activates the silicon diaphragm only slightly. Minimum movement causes the strain gage bridge fused to the diaphragm to produce an electrical signal proportional to the pressure.

Because there is no mechanical load on the sensing element, there are no friction errors. The transducer's direct current output is proportional to pressure and is electronically linearized and compensated for temperature and line voltage effects. It is then scaled, stabilized and converted for high resolution display. The analog

FIGURE 6. Digital pressure gage: (a) photograph; (b) schematic.



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voltage output can be used for remote readout or as a process control input.

For pressures up to 1 MPa (150 $lb_f \cdot in.^{-2}$), differential and gage measurements are handled by the same instrument. Gages are available for pressure ranges between 0 to 0.035 and 0 to 6.2 MPa (0 to 5 and 0 to 900 $lb_f \cdot in.^{-2}$).

Digital U-Tube Mercury Manometer Pressure Measurement System

A high precision digital mercury U-tube manometer system can be used for measurement and transmission of pressure readings as binary coded digital signals to computers, digital display systems or electronic data processing equipment. This instrument uses the principles of ultrasonic pulse reflection for measurement of transit time of pulses reflected off the mercury meniscus in each leg of the manometer. Its sensitivity is better than 0.3 Pa (2.5 mtorr). Accuracy is about 3 Pa (25 mtorr) and the direct reading electronic display is readable to this accuracy. The pressure ranges of this instrument extend from 0 to 280 kPa (0 to 2.1 ktorr).

Precision Calibrated Absolute Pressure Dial Gages

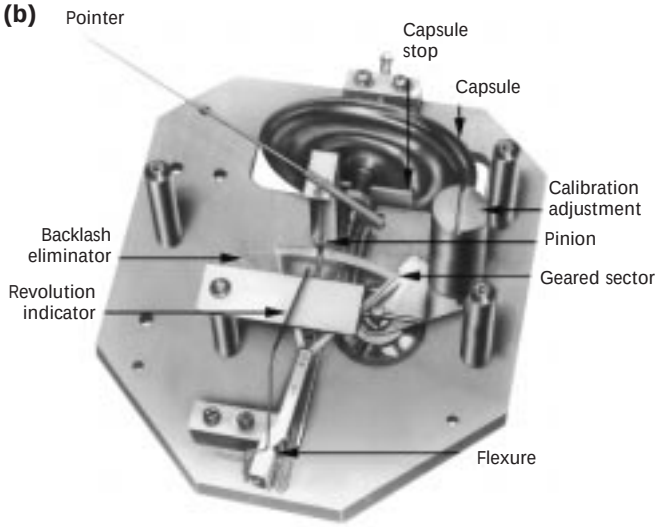
A series of precision dial gages are available for measurement of absolute

FIGURE 7. Example of two-revolution extended scale precision dial gage for measuring absolute pressure, calibrated by methods traceable to National Institute of Standards and Technology.

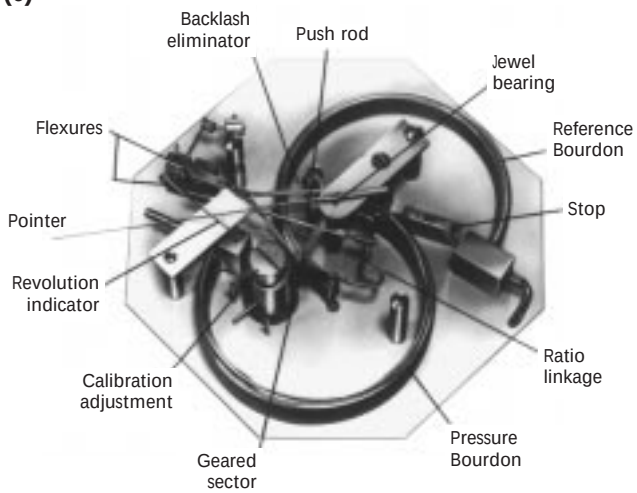
FIGURE 8. Absolute pressure dial gage: (a) with 150 mm (6.0 in.) diameter dial, scale length of 0.75 m (30 in.), accuracy of 0.1 percent of full scale and full scale ranges from 7 to 3 400 kPa (1 to 500 $lb_f \cdot in.^{-2}$); (b) typical aneroid capsule pointer operating mechanism; (c) typical Bourdon tube pointer operating mechanism.

(a)

(b)



(c)



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pressures with accuracies of 0.066 percent of full scale readings. Aneroid capsules are used for the lower pressure range dial gages; Bourdon tubes are used for the higher pressure range dial gages. In the capsule types, pressure is applied to the case of the gage, which is rated for gage pressure of 240 kPa (35 $lb_f \cdot in.^{-2}$). The gage case is also provided with a tempered glass dial cover and an overpressure blowout plug on the back of the case. In other models with a double revolution scale, accuracy is 0.1 percent of full scale. Sensitivity is 0.01 percent of full scale and repeatability is 0.03 percent of full scale. Gages are aneroid capsule types in absolute pressure ranges up to 350 kPa (50 $lb_f \cdot in.^{-2}$). Above 350 kPa, gages incorporate Bourdon tubes. Bourdon tube gages have a high strength plastic dial cover and a blowout plug in the back of the case. These dial gages are calibrated with precision mercury manometers or primary standard pneumatic piston gages, to provide calibrations traceable to the National Institute of Standards and Technology. The gage of Fig. 7 has a dial diameter of 220 mm (8.7 in.) and a scale length of 1.15 m (45 in.) and can be configured for pressure ranges up to 3.5 MPa (500 $lb_f \cdot in.^{-2}$ absolute).

Practical Visual Pressure Indicators for Leak Testing in the Shop or Field

Types of pressure gages that provide visible indications during leak testing include absolute pressure dial gages (Fig. 8), aneroid barometers, calibration instruments (Fig. 9) and ordinary dial gages indicating pressure relative to ambient atmospheric pressure (gage pressure) (Fig. 10), as well as water manometers, U-tube mercury manometers and mercury column barometers. The ordinary calibrated pressure dial gage is the type used for short duration pressure hold tests of test channel zones, double gasket flange interspaces and airlocks. For short duration pressure hold test, barometric pressure variations are ignored

FIGURE 9. Electropneumatic calibrator for field applications.

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and ordinary pressure gages showing gage pressures are used. One reason for this procedure is economic; absolute pressure gages cost five or more times as much as ordinary pressure gages. The ordinary dial pressure gage has typical accuracies in the range of ±0.25 to ±0.33 percent of full scale indication when recently calibrated. A mirror reflector behind the pointer of quality dial gages permits the observer to reduce the parallax error in the readings.

When reading a pressure dial gage, manometer column or quartz manometer, observers should position their heads so that their eyes are at the same level as the indicator on the gage or the top of the fluid column in the manometer. If the height of the gage or manometer is other than normal eye level, the observer should position the line of sight directly in front of the gage or manometer. These pressure readings should not be taken while viewing at an angle other than perpendicular to the face of the instrument. Following these procedures will help to reduce the variable parallax error in reading which results when different observers read test data from the same instruments. With dial gages equipped with a mirror reflector, the reading is taken by aligning the pointer directly over its own reflection in the mirror. The same techniques are used when reading an aneroid barometer because the barometer is itself an absolute pressure dial gage.

FIGURE 10. Ordinary dial pressure gage that measures gage pressure (the difference between actual pressure and atmospheric pressure), calibrated in inches of water for low pressure differentials.

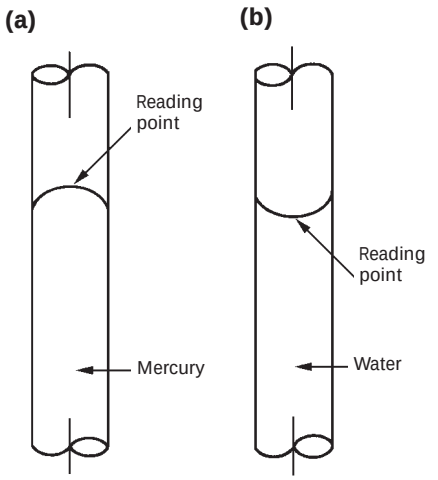
Technique for Precision Reading of Height of Manometer Columns

The reading point for mercury manometers is the top of the meniscus, as shown in Fig. 11a. The readings point for water manometers is the bottom of the meniscus, as shown in Fig. 11b. When a manometer is equipped with a mirror reflector, the reading is taken by aligning the reading point on the meniscus directly over the reflection of the reading point in the mirror.

Techniques for Reading Pressure Test Instruments Consistently and Accurately

When reading pressure gages, manometers or temperature instruments during absolute pressure leak tests, the operator should estimate pointer position or meter indications to at least one half of the smallest scale division on the instrument. It is important that the leak testing operator be consistent in the reading of instruments. Consistency in reading is as important as the assurance that the instrument is properly calibrated. This consideration results from the cancellation of calibration errors during successive instrument readings. For example, assume that a pressure gage reads high by 5 kPa at the test pressure. In this case the initial pressure reading may be shown as 340 kPa instead of the true value of 335 kPa. The final reading would appear as 334 kPa instead of its true value of 329 kPa. Assuming that the test system

FIGURE 11. Reading points for liquid column pressure gages, manometers and barometers: (a) mercury manometer meniscus reading point; (b) water manometer meniscus reading point.



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remains at uniform temperature, the pressure loss is found to be 6 kPa (0.9 $lb_f \cdot in.^{-2}$) in the operator's gage readings, as well as in the true pressure readings.

Variations in Atmospheric Pressure at Earth's Surface

All pressure measurements made within the earth's atmosphere are influenced by the fact that the earth's atmosphere imposes a pressure on any object in it. This atmospheric pressure varies not only with elevation and altitude but also with time and temperature. Although the atmospheric pressure at any one location is not constant, a standard atmosphere is now specified to be a pressure of 101.325 kPa (14.696 $lb_f \cdot in.^{-2}$ or 760.000 torr). Note that a pressure gage that indicates 50 kPa gage pressure is indicating 50 plus 101 or a total of 151 kPa of absolute pressure. Therefore, an absolute pressure gage would indicate the absolute pressure of 151 kPa when it is connected to a source of 50 kPa gage pressure above atmospheric pressure.

Effect of Ambient Barometric Pressure on Gage Pressure Readings

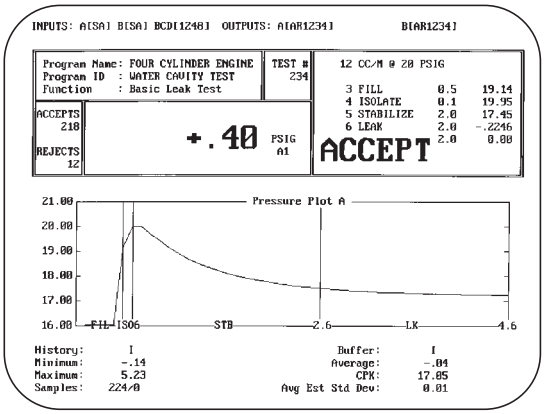
If temperature remained constant and uniform and no significant leakage occurred during a pressure change leak test period, the absolute pressure would remain unchanged. Yet even when the absolute pressure remains constant, the gage pressure decreases as barometric pressure increases, in accordance with definitions of absolute and gage pressures in this chapter. Conversely, if the barometer rises during the test period, the gage pressure would decrease by the same pressure increment. These changes in indicated gage pressure of the test volume that result from variations in ambient barometric pressure (and that are not caused by leakage) are factored out of the test data when a barometer or absolute pressure gage is used to measure the pressure used in computing the actual leakage rate.

Electronic Memory Pressure Decay Leak Testing System

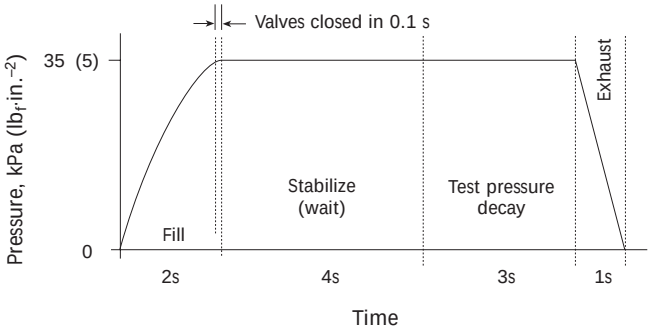
Two types of pressure change leak testers used for pressure decay leak testing are the electronic memory type and the differential pressure type. The electronic memory type leak tester shown in Fig. 12 is widely used in pressure decay leak testing. This system uses a (gage) pressure sensing transducer, available in pressure ranges varying from 10 kPa to as high as 140 MPa (2×10^4 $lb_f \cdot in.^{-2}$). For most applications, the electronic memory type of pressure decay leak testing system permits detection of smaller changes in pressure and faster testing times can be obtained. It also eliminates the problems associated with use of a reference pressure

FIGURE 12. Pressure decay leak tester with pressure sensitivity of 0.05 percent of full scale, pressure transducers ranging from vacuum to 140 MPa gage (2×10^4 $lb_f \cdot in.^{-2}$) and full scale ratings with electronic memory and automatic control of pressure sensitivity range, delay time, test time and set points: (a) automatic control display; (b) typical test plot; (c) diagram of pneumatic test system.

(a)



(b)



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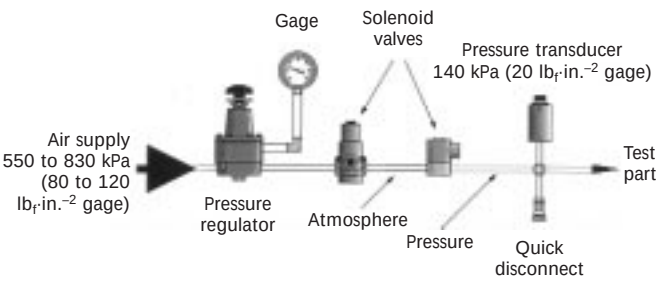

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chamber and the effects of adiabatic heating during pressurization.

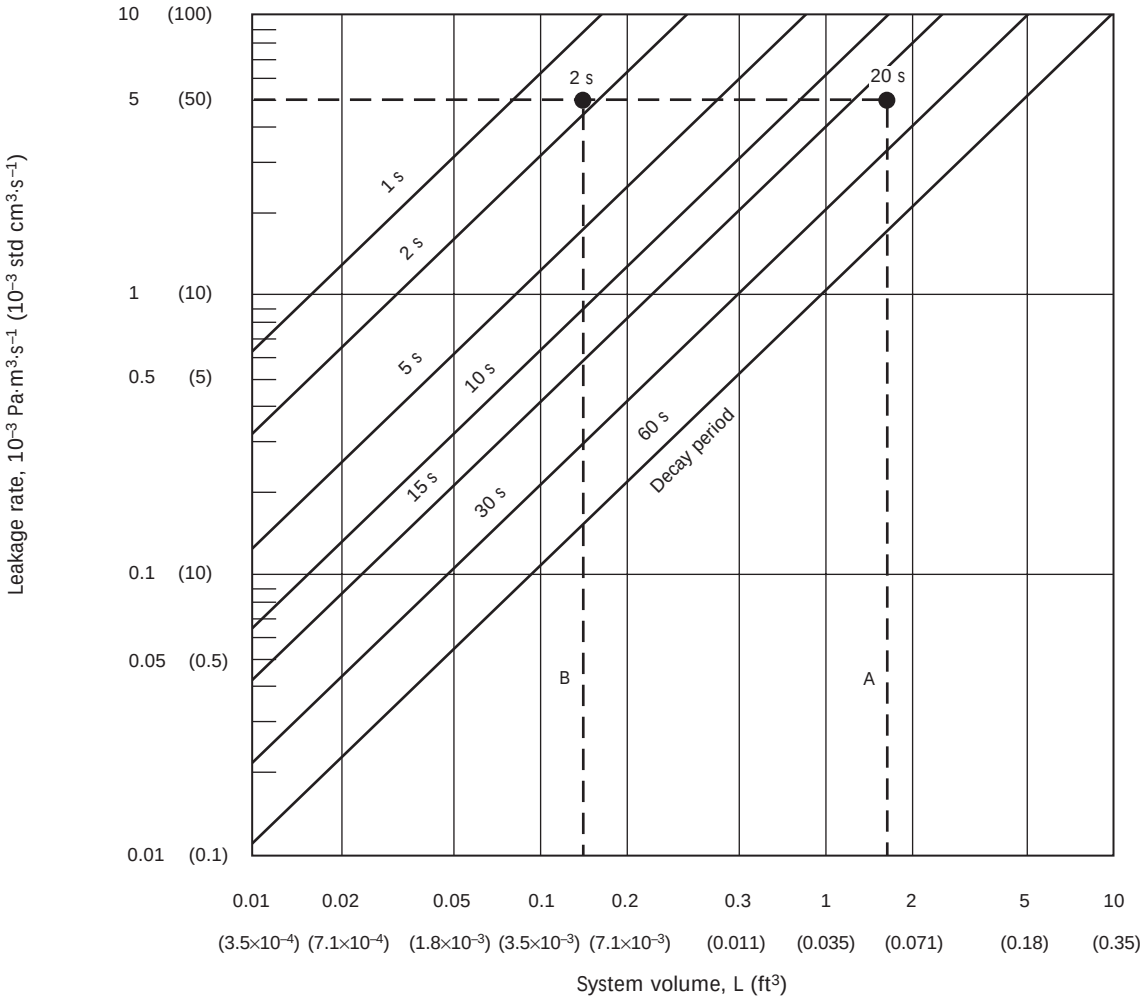
As an example, assume that leakage rate tests are to be conducted on a test system volume of 1.6 L (100 $in.^3$) pressurized to 140 kPa gage (20 $lb_f \cdot in.^{-2}$ gage). The leakage rate sensitivity chart (Fig. 13) for a 140 kPa (20 $lb_f \cdot in.^{-2}$ gage) transducer indicate that a pressure decay period of about 20 s would be required to achieve a leakage rate sensitivity of 5×10^{-3} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-4} std $cm^3 \cdot s^{-1}$). See dashed lines A on Fig. 13.

Pressure Decay Leak Testing

Figure 14 shows a variable capacitance differential pressure test setup. For low pressure leak testing in the range from 0 to 70 kPa (0 to 10 $lb_f \cdot in.^{-2}$) gage, this equipment is used with the reference pressure port of the differential sensor open to the ambient atmospheric pressure. For pressure decay leak testing, a reference chamber is used to equalize the pressures acting on the differential pressure sensor diaphragm at the start of each leak test. This initial pressure value can be stored in an electronic memory before the pressure decay leak test.

Mass flow leak testing instruments are used for applications where quantitative flow measurements are required (Fig. 15). Pressure decay instruments offer precision of ± 0.02 Pa (3×10^{-6} $lb_f \cdot in.^{-2}$).

FIGURE 13. Graphical relationship between leakage rate sensitivity and test system volume for instrument shown in Fig. 12.



Legend
A = 140 kPa (21 $lb_f \cdot in.^{-2}$) transducer
B = 14 kPa (2 $lb_f \cdot in.^{-2}$) transducer

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Ambient to Absolute Temperature Measurement

Absolute zero temperature corresponds to zero kelvin (0 K) and is equal to –273.15 °C (–459.67 °F). Absolute kelvin temperatures can be derived from temperatures in other units by Eqs. 1, 2 or 4:

$$\text{(1)} \quad K = 273.15 + {}^{\circ}C \cong 273 + {}^{\circ}C$$

and from fahrenheit (°F) temperatures by Eq. 2:

$$\text{(2)} \quad K = \frac{459.7 + {}^{\circ}F}{1.8} \cong \frac{460 + {}^{\circ}F}{1.8}$$

Absolute temperatures in degrees rankine (°R) are derived form temperatures in fahrenheit degrees by Eq. 3:

$$\text{(3)} \quad {}^{\circ}R = 459.67 + {}^{\circ}F \cong 460 + {}^{\circ}F$$

Finally, absolute temperatures in degrees kelvin (K) can be determined from rankine temperature values by Eq. 4:

$$\text{(4)} \quad K = \frac{{}^{\circ}R}{1.8}$$

FIGURE 14. Schematic diagram of differential decay test setup.

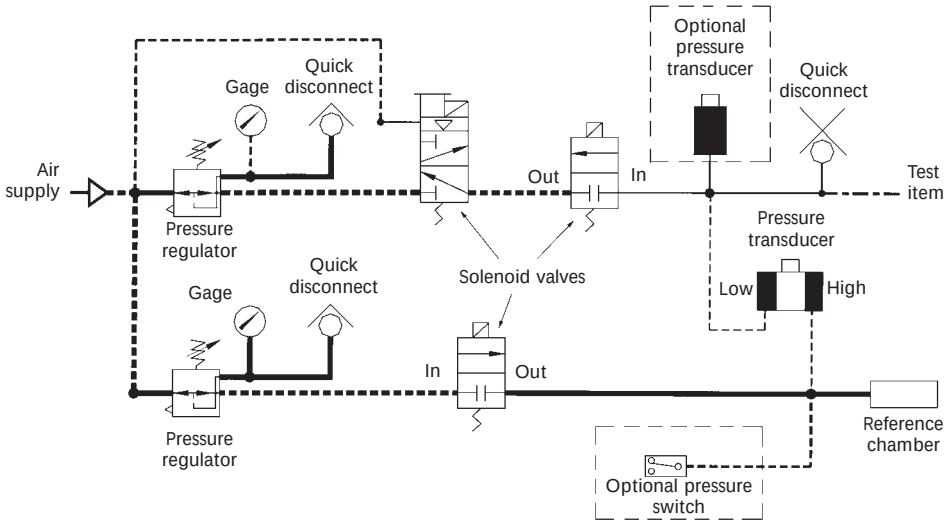
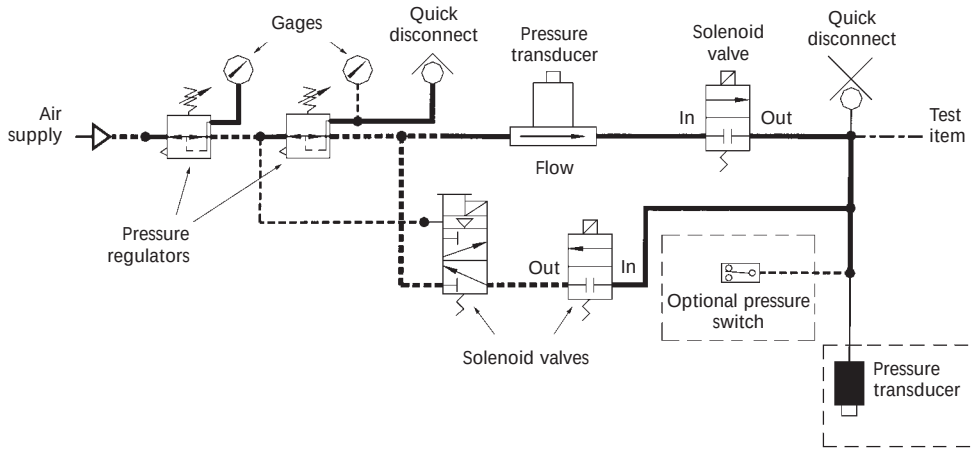


FIGURE 15. Schematic diagram of mass flow test setup.



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Techniques for Surface Thermometers in Pressure Change Leakage Tests

Surface thermometers, such as those shown in Fig. 16, may be used for small volume systems during leak testing, where it would be impractical to attempt to measure the internal air temperature. Temperature measurements must be made during pressure change leak testing in any case where temperature change can affect the results of the pressure testing due to the magnitude of the allowable pressure change or the duration of the pressure test. Surface thermometers must be held tightly against the surface whose temperature is to be measured. Any suitable techniques such as tape, magnets, couplant or clamps may be used to ensure this firm and intimate contact between the thermometer sensing surface and the surface of the test object whose temperature is to be measured. Procedures and test reports for pressure hold tests should specify the number and locations of surface thermometers used during each test.

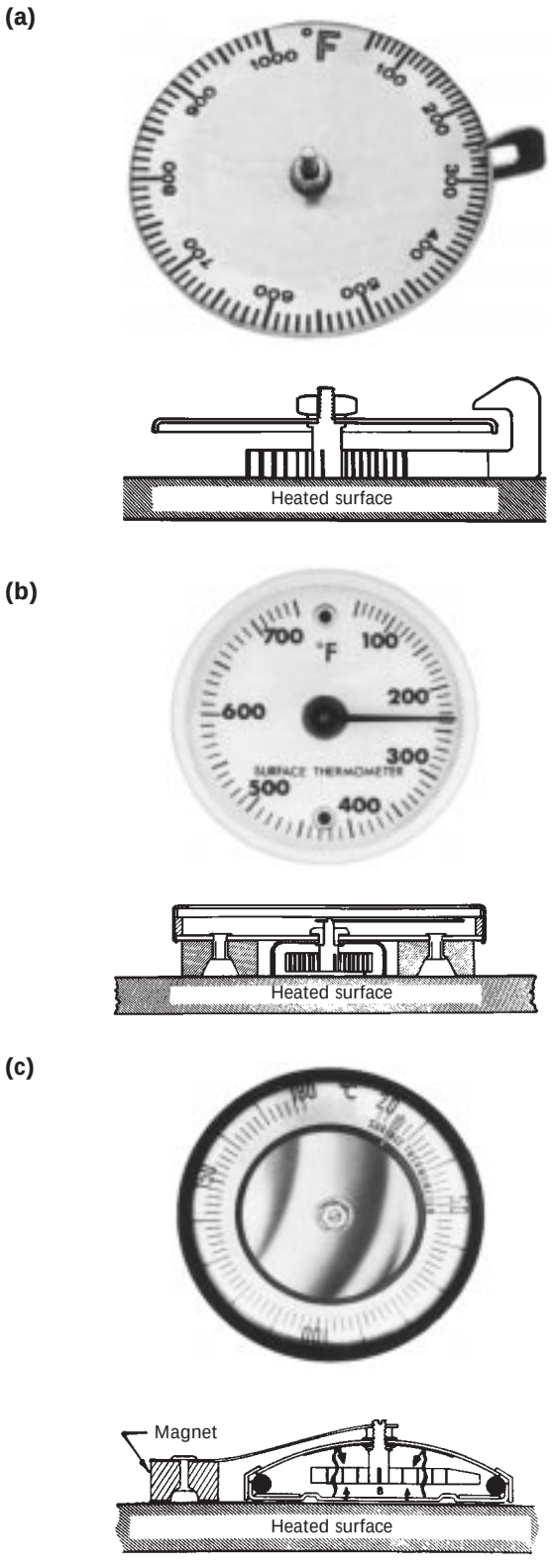
The double nut in the center of surface thermometers such as the types shown in Fig. 16 should not be loosened or tampered with by test operators or other personnel because it is locked in position to preserve the calibration setting of the thermometer. Thermometers used for testing should be calibrated periodically by a qualified instrument laboratory to provide assurance of their accuracy.

Surface Thermometer Designs and Mounting Techniques

Small, lightweight temperature indicating surface thermometers are available in various designs to cover several temperature ranges from 0 to 300 °C (0 to 500 °F) or from 300 to 550 °C (550 to 1000 °F), for example. Typical accuracy is ±2 percent of full scale range. The basic thermometer of Fig. 16a is designed for horizontal or slightly curved surfaces. The bimetallic coil in these instruments rests directly on the surface whose temperature is to be measured. The bimetallic spiral coil of the sensor expands or contracts in response to changes in temperature, thus causing the dial itself to rotate. The temperature of the surface is indicated by the hooklike pointer outside the periphery of the dial.

Figure 16b shows a type of surface thermometer with three main parts: a cover glass, a calibrated dial and indicator and a magnetic base containing a bimetallic thermal sensing element in an inverted cup. The sensor is a bimetal alloy designated by the applicable standard of

FIGURE 16. Surface thermometers on metal surfaces indicate adjacent air temperature during leakage rate testing: (a) basic surface thermometer; (b) surface thermometer with dual permanent magnets in base for mounting on ferromagnetic materials; (c) surface thermometer using both radiated and conducted heat input.



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the American Society for Testing and Materials and remains in permanent calibration. In use, the sensing element comes into virtual contact with the surface whose temperature is to be measured and provides a relatively fast response, reaching full temperature indications in about 3 min. The thermal response time constant (time to achieve one third of the temperature change) varies from 0.06 s to 1.04 min depending on the temperature range. The thermometer is mounted by merely laying it on any horizontal surface. On ferromagnetic material surfaces, two magnets in the base permit mounting in any orientation. An ancillary, hand adjusted pointer can be added to this surface thermometer to remember specific settings such as starting temperature or final values.

Figure 16c shows a type of surface thermometer that senses surface temperature by conduction and radiation effects. The base is applied to the surface to be measured. Heat is transferred to the base of the unit, which contains a bimetallic element. Radiation from the base inward causes the sensing element to react, producing a resulting dial readout. The bimetallic sensor is a specially processed alloy that is preconditioned and pretested for permanent calibration. The instrument contains a highly reflective, evaporated mirror that acts to protect the sensor from the effects of external radiation. This protective feature helps to provide more accurate temperature readings. The instrument is sealed against entry of corrosive atmospheres. The accuracy is ±2 percent of the full scale range.

Dry Bulb Temperature Measurements by Resistance Thermometers

In pressure change leak tests of larger structures, the temperature sensors in general use are 100 Ω copper thermohm detectors using a temperature sensitive element of extremely pure copper wire, wound into a helix and annealed to minimize mechanical strain. This type of construction provides a definite resistance value for each temperature within the range of the temperature detector. This stability and accuracy ensures the repeatability of measurements — important in leakage rate calculations because data to be analyzed are based primarily on measuring changes in temperatures and not on measuring the actual temperature. Response time of the copper wire temperature detectors for 90 percent of a temperature change is about 40 s. The limit of error of the detector is about ±0.03 °C (±0.05 °F) over the temperature range from 0 to 120 °C (32 to 250 °F).

Generally, suitable numbers of resistance thermometers are located throughout the volume of the structure during leakage rate testing to provide an adequate representation of internal temperatures in each significant volume. The number of detectors selected is a function of the contained free air volume, the configuration of the system under test and the redundancy desired to ensure representative contained air temperature sampling if one or more temperature sensors malfunctions. Each temperature sensor is then assigned a volume fraction based on the fraction of the total volume under test. This volume fraction is a temperature zone that may be determined by prior temperature surveys and represents the portion of the contained gas or air that the individual sensor is monitoring. The values of temperature indicated for each temperature sensor are recorded together with readings of pressure sensors, at each interval during the pressure change leakage test. These temperature data are multiplied by the fractional volumes they represent and the weighted average contained air temperature for the test volume is computed and recorded for use in correcting pressure indications for the effects of temperature changes.

Sensors for Dew Point Temperature Measurements

The dew point temperature is a direct indication of the amount of water vapor present in the air contained within a test volume subject to pressure change leakage rate tests. If the temperature was reduced to the dew point temperature, moisture would condense on solid surfaces and thus be temporarily removed from the contained air. Vapor pressure due to moisture evaporated into the contained air adds to the total pressure measured by most pressure detecting instruments used in leakage rate testing. Two types of dew point sensors used in leak testing are aluminum oxide capacitance sensors and resistance dependent sensors mounted on thermoelectric cooling elements.

Capacitive Dew Point Sensors

Capacitance type dew point gages (also known as aluminum oxide dew point detectors) consist of a strip of metallic aluminum anodized by a special process to provide a porous oxide layer. A very thin coating of gold is then evaporated

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over this oxide structure to provide a conducting electrode. The aluminum base metal and the gold layer electrode thus form two electrodes with the dielectric oxide layer between them, which serves as an electrical capacitor. The concentration of water vapor in the ambient air changes the dielectric constant and so varies the electrical capacitance of the sensing element. Used as an impedance element in an electronic circuit, this variable capacitor produces output signals that measure the dew point temperature in the atmosphere contained within the system under pressure change leakage test. The system accuracy is typically ±1 °C (±1.8 °F) over a dew/frost point temperature range from –80 to +20 °C (–110 to + 68 °F). The repeatability of output signal readings is reportedly ±0.5 °C (±0.9 °F) in the dew point range commonly encountered in leak testing.

Resistive Dew Point Sensors

Resistance dew point sensors are formed on the surface of an insulating disk consisting of epoxy filled fiberglass cloth. A pair of intermeshing gold conductive fingers provides electrodes for the surface resistive element (the uncoated fiberglass insulator). This surface resistance is affected by moisture condensed on the fiberglass insulator between the two electrodes. The resistance sensing disk is mounted on a two stage thermoelectric cooler. Current supplied to the thermoelectric cooler is controlled by comparing the sensor resistance to that of a fixed resistor. The dew point determination is based on this surface conductivity (which increases when liquid water is formed by condensation on the cooled surface of the sensor). The dew point temperature range of this detector system extends from –29 to + 57 °C (–20 to +135 °F). The repeatability of signals is in the range of ±0.3 C (±0.5 °F).

Correcting Pressure Change Leak Test Data for Changes in Vapor Pressure

The partial pressure of water vapor adds to the true pressure of gases to produce the total pressure of contained fluid measured by the pressure sensors used in pressure change leakage rate testing. If the partial pressure of water vapor remained constant throughout the duration of a leakage rate test and constant throughout the test volume, the value of this constant partial pressure could be subtracted from the total pressure measured to obtain the pressure due to contained gases that generally obey the ideal gas laws. However, if the temperature changed, condensing water from the air or evaporating more water into the air, within a constant volume system under test, the vapor pressure of the water would change significantly. If no correction were made for these nonideal variations in vapor pressure, leakage measurements by pressure change could have considerable error. This error can be avoided if all total pressure measurements are corrected by subtraction of the known water vapor pressure, so that leakage rate calculations are based only on the changes in the partial pressure of air (or nitrogen or other pressurizing gas that obeys the laws for ideal gases). Numerous physical tables relate the partial pressure of water vapor to dew point temperatures, to temperatures of air in equilibrium over water of the same temperature or to other data such as relative humidity, temperature and barometric pressure. For example, the *CRC Handbook of Chemistry and Physics* lists tables relating the pressure of aqueous vapor over water (torr) to temperature (°C).[1] Steam tables based on American Society of Mechanical Engineers (ASME) data also relate the partial pressure of water vapor ($lb_f \cdot in.^{-2}$ absolute) to temperature (°F) as well as in SI units of pressure (kPa) and temperatures (°C and K).[1]

At the dew point temperature, equilibrium exists between the partial pressure of water vapor in air above a surface on which water is condensing or from which water is evaporating. Thus, the dew point temperature measured during leak testing can be related immediately to the partial pressure of water vapor at the location and temperature of the dew point sensor (Table 2).

Effect of Pressurization on Dew Point Temperature and Water Vapor Pressure

During pressurization of systems to be tested for leakage by pressure change or flow rate leakage tests, the partial pressure of water vapor is increased in proportion with the total pressure of contained air. Thus, the dew point and probably the relative humidity will also increase during pressurization. Therefore, use of an air dryer on the supply air during pressurization is recommended. If a large volume system (such as a nuclear power reactor containment structure) is to be tested and is provided with cooling coils for the ventilation system, these cooling systems should be used to minimize any increases in dew point temperature during pressurization. During the leakage rate test, the dew point temperature should be

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monitored for any changes in trends. A sudden change in the rate of variation of dew point temperature with time could indicate water leakage.

TABLE 2. Water vapor pressures as a function of dewpoint temperature in degree Celsius, in pascal and in pound per square inch absolute.

Dewpoint Temperature		Vapor Pressure, Absolute	
°C	(°F)	Pa	($lb_f \cdot in.^{-2}$)
–18	(–0.4)	124.8	0.0181
–17	(1.4)	137.2	0.0199
–16	(3.2)	152.4	0.0221
–15	(5.0)	166.2	0.0241
–14	(6.8)	181.3	0.0263
–13	(8.6)	197.9	0.0287
–12	(10.4)	216.5	0.0314
–11	(12.2)	235.8	0.0342
–10	(14.0)	257.9	0.0374
–9	(15.8)	281.3	0.0408
–8	(17.6)	307.5	0.0446
–7	(19.4)	335.8	0.0487
–6	(21.2)	370.3	0.0537
–5	(23.0)	402.0	0.0583
–4	(24.8)	436.4	0.0633
–3	(26.6)	473.7	0.0687
–2	(28.4)	514.4	0.0746
–1	(30.2)	558.5	0.0810
0	(32.0)	610.2	0.0885
1	(33.8)	657.1	0.0953
2	(35.6)	706.0	0.1024
3	(37.4)	757.7	0.1099
4	(39.2)	813.6	0.1180
5	(41.0)	872.2	0.1265
6	(42.8)	934.9	0.1356
7	(44.6)	1001.8	0.1453
8	(46.4)	1072.8	0.1556
9	(48.2)	1148.0	0.1665
10	(50.0)	1228.0	0.1781
11	(51.8)	1312.8	0.1904
12	(53.6)	1402.4	0.2034
13	(55.4)	1497.6	0.2172
14	(57.2)	1598.2	0.2318
15	(59.0)	1705.1	0.2473
16	(60.8)	1818.2	0.2637
17	(62.6)	1937.4	0.2810
18	(64.4)	2063.6	0.2993
19	(66.2)	2196.7	0.3186
20	(68.0)	2337.3	0.3390
21	(69.8)	2486.3	0.3606
22	(71.6)	2643.5	0.3834
23	(73.4)	2808.3	0.4073
24	(75.2)	2982.7	0.4326
25	(77.0)	3166.8	0.4593
26	(78.8)	3360.5	0.4874
27	(80.6)	3564.6	0.5170
28	(82.4)	3779.0	0.5481
29	(84.2)	4004.5	0.5808
30	(86.0)	4241.7	0.6152
31	(87.8)	4491.3	0.6514
32	(89.6)	4754.0	0.6895
33	(91.4)	5029.1	0.7294
34	(93.2)	5318.0	0.7713
35	(95.0)	5621.3	0.8153
36	(96.8)	5939.9	0.8615
37	(98.6)	6273.6	0.9099
38	(100.4)	6623.8	0.9607

Determining Gas Pressure from Total Pressure and Water Vapor Pressure

The air, nitrogen or other typical pressurizing gas used in pressure change leakage tests is selected so that it obeys the ideal gas laws relating pressure, temperature and volume. The water vapor contained in the pressurizing gas fails to obey these ideal gas laws, yet it contributes a partial pressure which adds to the ideal gas pressure to equal the total gas pressure measured by pressure sensing instruments during the leakage tests. To permit valid estimations of true gas leakage rates, the partial pressure P_v of water vapor must be subtracted from the total absolute pressure P to obtain the true gas pressure P_g as shown in Eq. 5 for net ideal gas pressure:

$$P_g = P - P_v \quad (5)$$

Equation 5 applies to absolute pressure only, in any single system of pressure units. Water vapor pressure varies in air as a function of dew point temperature, in SI units (see also Table 2).

Calculation of Leakage Rate by Pressure Change Test (Constant Temperature)

If the test is of short duration and it is known that temperature has not changed during a pressure hold test (or if temperature conditions remain constant), the test requires only measurement of gage pressure. In this case, the time rate pressure change can be calculated from Eq. 6:

$$\frac{\Delta P}{\Delta t} = \frac{P_1 - P_2}{\Delta t} \quad (6)$$

As an example of a calculation using Eq. 6, suppose that a pressure hold test is conducted on a system with an allowable pressure loss rate of 7 kPa (1 $lb_f \cdot in.^{-2}$) in 30 min. If the initial gage pressure was 400 kPa (56.0 $lb_f \cdot in.^{-2}$) at time 13:00 and the final gage pressure was 396 kPa (55.4 $lb_f \cdot in.^{-2}$) at time 13:30, Eq. 6 indicates that the time rate of pressure loss is

$$\frac{\Delta P}{\Delta t} = \frac{P_1 - P_2}{\Delta t} = \frac{400 - 396}{30} = \frac{4 \text{ kPa}}{30 \text{ min}} = 130 \text{ Pa} \cdot \text{min}^{-1} = 2.2 \text{ Pa} \cdot \text{s}^{-1}$$

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In English units, the same test calculation would appear as:

$$\frac{\Delta P}{\Delta t} = \frac{P_1 - P_2}{\Delta t} = \frac{56.0 - 55.4}{30}$$

$$= \frac{0.6 \text{ lb}_f \cdot \text{in.}^{-2}}{30 \text{ min}}$$

$$= 1.2 \text{ lb}_f \cdot \text{in.}^{-2} \cdot \text{h}^{-1}$$

$$= 3.33 \times 10^{-3} \text{ lb}_f \cdot \text{in.}^{-2} \cdot \text{s}^{-1}$$

The measured rate of pressure loss is less than the allowable pressure loss rate of 7 kPa (1 $\text{lb}_f \cdot \text{in.}^{-2}$) in 30 min, indicating that the system under test is acceptable because its leakage rate is below the specified maximum allowable leakage rate.

Calculation of Leakage Rate by Pressure Change Test (Constant Volume)

During a pressure change leakage test of a system with fixed volume, the initial volume V_1 and the final volume V_2 remain essentially identical. Thus, for the special case of constant volume systems under test, $V_1 = V_2$ and Eq. 7 applies to the pressure change leak test period:

$$\frac{P_1}{P_2} = \frac{T_1}{T_2} \quad (7)$$

or

$$P_1 = \frac{T_1}{T_2} P_2$$

As can be seen from the first form of Eq. 7, absolute pressure varies in direct proportion with absolute temperature. In the absence of significant leakage, the absolute pressure increases in proportion with an increase in contained absolute gas temperature. Conversely, lowering the gas temperature lowers the absolute internal gas pressure proportionately.

Effect of Ambient Barometric Pressure on Absolute Pressure Gage Readings

If temperatures remained constant and uniform and no significant leakage occurred during a pressure change leak test period, the absolute pressure would remain unchanged. This is in contrast to the gage pressure, which increases as barometric pressure decreases by the same pressure increment when no significant leakage occurs.

These changes in indicated gage pressure of the test volume which result from variations in ambient barometric pressure (and which are not caused by leakage) are factored out of the test data when a barometer or absolute pressure gage is used to measure the absolute pressure.

Correcting Pressure Change Leak Test Data for Changes in Temperature

When a short duration pressure hold test is conducted under varying temperature conditions and requires measurement of both gage pressure and temperature but does not require measurement of barometric pressure, the barometric pressure is assumed to be one standard atmosphere (101.3 kPa or 14.7 $\text{lb}_f \cdot \text{in.}^{-2}$). The pressure loss per unit of time is then determined from the initial gage pressure P_1 and temperature T_1 and the final gage pressure P_2 and the final temperature T_2, by means of Eq. 8. The temperatures must be absolute temperatures and the absolute pressures may be taken as the gage pressures plus an assumed standard barometric pressure.

For gage pressures in kilopascal and temperatures in degree celsius, using SI units and measuring time in seconds, the pressure change rate is given by Eq. 8:

$$\frac{\Delta P}{\Delta t} = \left\{\left[\left(P_1 + 101\right) - \left(P_2 + 101\right)\left(T_1 + 273\right)\right] \div \left(T_2 + 273\right)\right\} \div \Delta t \quad (8)$$

For gage pressures in pound per square inch and temperatures in degree fahrenheit, using English units and measuring time in minutes:

$$\frac{\Delta P}{\Delta t} = \left\{\left[\left(P_1 + 14.7\right) - \left(P_2 + 14.7\right)\left(T_1 + 460\right)\right] \div \left(T_2 + 460\right)\right\} \div \Delta t \quad (9)$$

For absolute pressures in kilopascal and temperatures in degree celsius, using SI units and measuring time in second:

$$\frac{\Delta P}{\Delta t} = \frac{P_1 - P_2 \dfrac{T_1 + 273}{T_2 + 273}}{\Delta t} \quad (10)$$

For absolute pressures in pound per square inch and temperatures in degree fahrenheit, using English units and measuring time in minute:

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$$\frac{\Delta P}{\Delta t} = \frac{\left(P_1 - P_2 \frac{T_1 + 460}{T_2 + 460}\right)}{\Delta T} \quad (11)$$

For absolute pressures and absolute temperatures, the correction takes on the simpler form of Eq. 12:

$$\frac{\Delta P}{\Delta t} = \frac{\left(P_1 - P_2 \frac{T_1}{T_2}\right)}{\Delta T} \quad (12)$$

where all terms are expressed in SI units or where all terms are expressed in English units.

Determining Mass of Contained Gas for Pressure Change Leakage Tests of Large Volume Systems

The time rates of leakage are determined by the changes in the total mass of air, nitrogen or other ideal pressurizing gas contained within the test volume V, after corrections for temperature T and water vapor pressure P_v. In the absolute test technique, the ideal gas law can be expressed in the form of Eq. 13, for the case in which the test volume remains constant:

$$W = K_1 \frac{V}{R} \frac{P' - P_v}{T} \quad (13)$$

where W is measured mass of contained (ideal) gas or air, kilogram (or pound); V is internal free volume of system under test, cubic meter (or cubic foot), constant; R is individual gas constant. (For air, R = 287 $J \cdot kg^{-1} \cdot K^{-1}$ or 53.35 $ft\text{-}lb_f \cdot lb_m^{-1} \cdot °R^{-1}$) P is total absolute pressure in test volume, pascal (or $lb_f \cdot in.^{-2}$ absolute); P_v is partial pressure of water vapor in contained air, *pascal* (or $lb_f \cdot in.^{-2}$ absolute); T is mean absolute temperature of air contained in test volume kelvin (or degree rankine); K_1 is 1 (for SI units). K_1 = 144 (for English units for conversion from pressure in $lb_f \cdot in.^{-2}$ to $lb_f \cdot ft^{-2}$). Typically, the leakage rate can be determined from the change in contained air mass through a succession of test point data readings or by subtracting the final mass (at the end of a test period) from the initial contained mass (at the beginning of the test period). The mass change must be divided by the time interval between successive readings or between initial and final readings, to provide the time rate of leakage. The mass leakage rate would then be given by Eq. 14:

$$Q_t = \frac{\Delta W}{\Delta t} \quad (14)$$

where ΔW is $W_{start} - W_{end}$ = change in contained mass during test interval; ΔT is $t_{end} - t_{start}$ = time interval between start and end of test interval.

Determining Mass Loss of Contained Gas for Pressure Decay Tests of Large Volume Systems

When the test volume is constant, the mass of contained air or gas at the beginning of the test period is given by Eq. 15:

$$W_1 = P_1 \frac{V}{R T_1} \quad (15)$$

The mass of contained air at the end of the test period is given by Eq. 16:

$$W_2 = P_2 \frac{V}{R T_2} \quad (16)$$

The mass loss due to leakage during the test period is then given by Eq. 17:

$$W_1 - W_2 = \left(\frac{P_1}{T_1} - \frac{P_2}{T_2}\right) \frac{V}{R} \quad (17)$$

In Eq. 15 through 17, W_1 is initial mass of contained air (kilogram); W_2 is final mass of contained air (kilogram); P_1 is initial absolute test pressure (pascal); P_2 is final absolute test pressure (pascal); T_1 is initial contained air temperature, kelvin (= °C + 273.15); T_2 is final contained air temperature (kelvin); V is test volume (cubic meter); and R is gas constant for air (287 $J \cdot kg^{-1} \cdot K^{-1}$).

Determining Leakage Rate in Volume Units at Standard Temperature and Pressure

The standard conditions for volume loss leakage rates are as follows: P_s is standard pressure, 101.325 kPa (14.696 $lb_f \cdot in.^{-2}$ absolute); T_s is standard temperature, 20 °C or 293.15 K (68 °F or 527.67 °R); V_s is volume of air at standard conditions corresponding to a particular mass W. The mass of air at standard conditions is related to the standard volume V_s by Eq. 18:

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$$\textbf{(18)}\quad W_s = \frac{P_s V_s}{R T_s}$$

The volume of air at standard conditions is given in terms of mass W by Eq. 19:

$$\textbf{(19)}\quad V_s = \frac{W R T_s}{P_s}$$

The leakage rate Q_s in standard volume units is given by Eq. 20:

$$\textbf{(20)}\quad Q_s = \frac{V_{s1} - V_{s2}}{\Delta t} = \frac{\frac{W_1 R T_s}{P_s} - \frac{W_2 R T_s}{P_s}}{\Delta t} = \frac{R T_s}{P_s \Delta t}\left(W_1 - W_2\right)$$

When the actual pressure change leakage test data are used (as measured at test temperatures T_1 and T_2 and with corresponding test pressures P_1 and P_2), the standard leakage rate Q_s in standard volume units is given by Eq. 21:

$$\textbf{(21)}\quad Q_s = \frac{V}{\Delta t}\frac{T_s}{P_s}\left(\frac{P_1}{T_1} - \frac{P_2}{T_2}\right)$$

When SI units are used in Eq. 20 or 21, test volume V is given in cubic meter; the time interval Δ_t, in second; the pressure P, in pascal; and the temperature T, in kelvin (K = °C + 273.15). P_s is simply dropped; the leakage rate is then given in pascal cubic meter per second.

When English units are used, the test volume is measured in cubic foot; the time, in hour; the pressure, in pound per square inch; and the temperature, degree rankine (= °F + 459.7). The leakage rate Q_s is then given in standard cubic foot per hour.

Determining Leakage Rate in SI Units at Standard Temperature and Pressure

It should be noted that the leakage rate Q in SI units has been expressed in this book in units of $Pa{\cdot}m^3{\cdot}s^{-1}$, which is the product of volume and pressure, divided by time. In this case, the leakage rate Q_s in SI units is given by Eq. 22:

$$\textbf{(22)}\quad Q_s = \frac{R T_s}{t}\left(W_1 - W_2\right)$$

When actual pressure change leakage rate test data are used, the leakage rate Q_s in SI units is given by Eq. 23:

$$\textbf{(23)}\quad Q_s = \frac{\frac{V}{t} T_s\left(\frac{P_1}{T_1} - \frac{P_2}{T_2}\right)}{P_s}$$

where Q is leakage rate ($Pa{\cdot}m^3{\cdot}s^{-1}$); t is test duration (second); R is individual gas constant, $J{\cdot}kg^{-1}{\cdot}K^{-1}$ (for air, R = 287 $J{\cdot}kg^{-1}{\cdot}K^{-1}$); V is test volume (cubic meter); T_s is standard absolute temperature, K (i.e., 293 K); W_1 is mass of contained air or gas at beginning of test (kilogram); P_1 is pressure at beginning of test (pascal); W_2 is mass of contained air or gas at end of test (kilogram); P_2 is pressure at end of test (pascal); T_1 is absolute temperature at beginning of test, kelvin (K = 273 + $°C_1$); T_2 is absolute temperature at end of test, kelvin (K = 273 + $°C_2$); the subscript s denotes standard. P_s is standard pressure of 101.3 kPa.

Determining Mass of Contained Air after Correction of Water Vapor Content

The actual pressure of ideal pressurizing gas (air, nitrogen or other gases obeying the ideal gas law) can be determined by subtracting the pressure of contained water vapor from the total pressure, in accordance with Eq. 5. The mass W_g of ideal gas is given in terms of total pressure P minus the pressure of water vapor P_v:

$$\textbf{(24)}\quad W_g = \frac{V}{RT}\left(P - P_v\right)$$

Equation 24 would apply to quantities expressed in SI units as listed for Eq. 17.

In practical (mixed) units (used in shop or field leak tests in industry before 1981), Eq. 25 gives the mass of contained air (or ideal gas) after correction for water vapor content:

$$\textbf{(25)}\quad W_g = 144\frac{V}{RT}\left(P - P_v\right)$$

where W_g is mass of contained air (pound); V is internal free volume of containment (cubic foot); R is gas constant for air, 53.35 $ft\text{-}lb_f{\cdot}ft\text{-}lb_m^{-1}{\cdot}°R^{-1}$; T is mean absolute temperature (dry bulb) of contained air (degree rankine); P is total absolute pressure in containment ($lb_f{\cdot}in.^{-2}$ absolute); and P_v is partial pressure of water vapor in containment ($lb_f{\cdot}in.^{-2}$ absolute).

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Leakage Rate Test Data Obtained by Absolute Test Technique in English Units

The analysis used with absolute pressure leakage rate tests consists of determining the mass of air in the containment, using the ideal gas law, at each time point during the test and using a straight line least squares analysis to estimate the leakage rate. Errors in the determined masses are assumed to be equally variable (i.e., the slope and intercept of the line are estimated by ordinary as opposed to weighted least squares) and uncorrelated. An upper one-sided confidence limit for the leakage rate is based on normal regression theory (i.e., the masses are related by a straight line and deviations from that line are normally distributed) and a technique due to Fieller for finding confidence limits for ratios of means of normally distributed random variables. For each time point t_i, the corresponding mass of contained air W_i is determined directly from the application of the ideal gas law as given in Eq. 26:

$$W_i = \frac{144\,V}{R} \times \frac{P_i - P_{v_i}}{T_i} \quad (26)$$

A linear least squares fit of the data is then made according to the relation $(W_i)_a = At_i + B$.

The estimate of the leakage rate is a function of both the slope and the intercept of the regression line (percent per day):

$$Q_{am} = -2400\,\frac{A}{B} \quad (27)$$

In Eq. 27, the term A represents the slope of the least squares straight line. The term B indicates the intercept of this straight line with a vertical line drawn through the time scale point for $t = 0$. The numerical constant 2400 is the product of the number of hours in a day (24) and the multiplier (100) for a percentage calculated from a ratio. The negative sign (–) indicates that, for a pressure decay test, the regression line slopes downward from the initial point at $t_i = 0$ to later points at $t_i = t_n$.

Effects of Time Duration of Pressure Change Leakage Rate Tests

For short duration absolute pressure change leak tests, such as a 2 h pressure hold test, the change in atmospheric pressure is usually insignificant and standard barometric pressure can be assumed to exist. (Care is needed to avoid this assumption during passage of a cold front or low pressure storm system, because rapid changes in barometric pressure can accompany such storm periods.) If the allowable pressure loss per unit of time is large enough, it may also be possible to eliminate measurement of temperature and to measure only pressure and time. For very short duration pressure tests (such as 15 to 120 min), the leak testing procedure may require only measurement of gage pressure and time (in constant volume systems).

For longer duration tests such as 24 h pressure hold or leakage rate tests, it is most likely that specifications for test procedures will require measurement of both temperature and barometric pressure (or absolute pressure) because of the larger atmospheric changes that could occur in these two test variables. It may also be necessary to measure dew point temperature to account for variations in water vapor pressure with temperature.

Analysis Techniques for Pressure Change Leakage Test Data[2,3]

Three techniques for analysis of data obtained during pressure change leakage rate testing of pressurized test systems are (1) the mass point analysis technique, (2) the leakage rate point analysis technique based on total time from start of test and (3) the leakage rate point analysis technique based on test interval data.

Mass Point Technique of Analysis of Pressure Change Leak Test Data

In mass point data analysis, data from an absolute technique leak testing system are reduced to a value for the mass W of air within a pressurized test volume, by application of the ideal gas law. The test data consist of a time sequence of independent values for the contained air mass. Figure 17a is a graphical illustration of a short sequence of mass point test data, plotted vertically as a function of elapsed test time, shown horizontally. The successive sets of test data are identified by subscripts $n = 0, 1, 2, 3, 4 \ldots k$. The term W_n is the value of the air mass inside the test volume at the time t_n. In practice, W_n often is represented in percentages of the initial air mass at the start of the leakage test at time $t = 0$ and the elapsed test time is often recorded in hours. (Later, the leakage rates may be stated in percentages of initial mass change per

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day.) In Fig. 17a where $k = 5$, t_0 is the time when leak testing begins (zero hours) and W_0 is the mass of air within the test volume when leak testing begins. W_5 is the mass of contained air after an elapsed test time of t_5 in hours.

Point-to-Point Analysis of Pressure Change Leakage Rate Test Data

Figure 17 illustrates a simple example of the leakage rate point-to-point technique of analysis of leakage rate test data. Individual leakage rates Q_n are calculated from the mass differences between successive adjacent test points:

$$\textbf{(28)} \quad Q_n = \frac{W_n - W_{n-1}}{t_n - t_{n-1}}$$

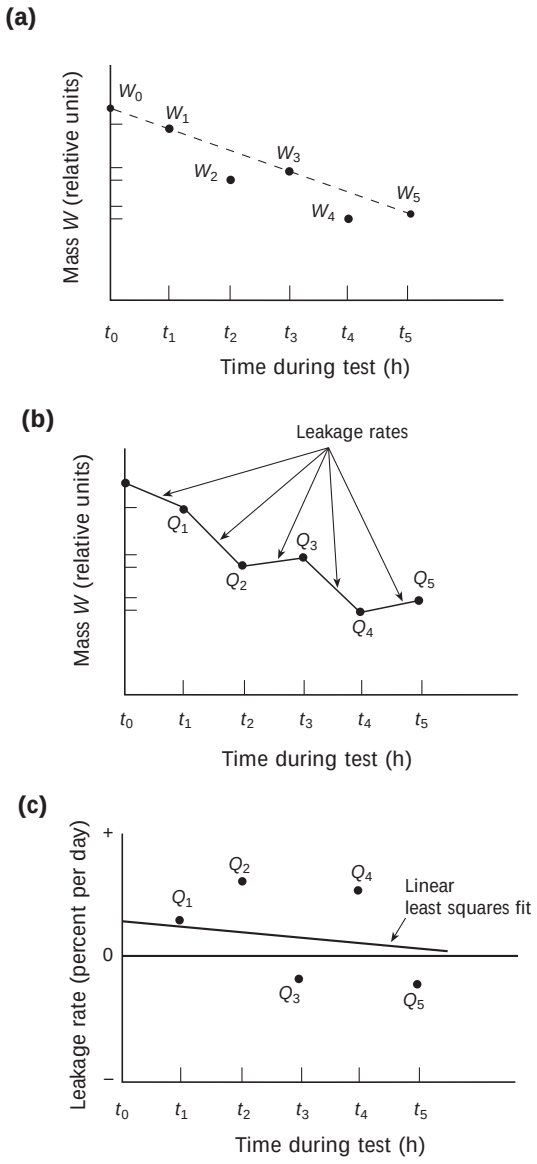
The values of this point-to-point leakage rate are equivalent to the slopes of the lines labeled as Q_1, Q_2 ... Q_5 in Fig. 17b. If these point-to-point leakage rates are then plotted on a new graph as a function of elapsed test time, the result is similar to Fig. 17c. Here, the computed leakage rate in percentage change per day of the initial contained mass W_0 is shown on the vertical scale and the elapsed test time on the horizontal scale. Positive values for leakage are shown in Fig. 17c when the slope of the line Q_n in Fig. 17b is downward.

Negative values of leakage are shown in Fig. 17c when the slope of the corresponding line in Fig. 17b is upward. The sloping line in Fig. 17c indicates the leakage rate trend with elapsed time. When this trend line flattens, it indicates establishment of the leakage rate with additional test time serving only to increase the reliability of the data. When test data are taken at regular time intervals, there is no implicit weighting of data. The effective leakage rate is simply the arithmetic mean of all the individual leakage rates when these data are taken at roughly equal time intervals. This greatly simplifies online data analysis during pressure change leakage rate testing.

Leakage Rate Total Time Technique of Analysis of Pressure Change Leak Test Data[2,3]

Various statistical techniques may be used for analyzing leakage rates from identical point-by-point leak test data during pressure change leakage rate test, where slopes of lines equal leakage rates.[2,3] Figure 18a illustrates the total time technique of calculating leakage rates based on the mass difference between the initial mass W_0 at the start of the test and the mass W_n for the most recent data point, as slopes of individual lines, $Q_n = (W_n - W_0)/(t_n - t_0)$. Each successive leakage calculation is therefore based on a longer period of time, $t_n - t_0$. A different leakage rate may thus be computed for

FIGURE 17. Various statistical techniques for analyzing leakage rate from identical point-by-point test data during pressure change leakage rate test, after Fleshood[2] and Lau[3]: (a) leak testing data with computed air mass plotted as function of elapsed test time t_n for the mass point analysis technique, where slope of dashed line from W_0 to W_5 indicates overall leakage rate $Q = (W_0 - W_5)/(t_5 - t_0)$; (b) leakage rates calculated from mass differences between adjacent test points, where slopes of short lines indicate incremental leakage rates, $Q_n = (W_{n-1} - W_n)/(t_n - t_{n-1})$, valid for n greater than zero; (c) leakage rate trend line calculated by linear least squares analysis of incremental leakage rates Q_n shown in Fig. 17b.



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each test point following the initial point, by Eq. 29 for total time leakage rate:

$$(29)\quad Q_n = \frac{W_n - W_0}{t_n - t_0}$$

With this analysis technique, it is not proper to assume that the effective leakage rate for the total test period is a simple average of all the individual leakage rates calculated by Eq. 29. Each successive leakage rate is calculated from the contained air mass change over a longer elapsed time period. The result of averaging Q_n leakage rates is heavily weighted toward the larger values of n (longer total times, $t_n - t_0$).

Caution must be taken to time weight each datum appropriately. Also, the instrumentation errors for small n values will show up as relatively large deviations in the analysis. See also Fig. 18d for linear least squares fit evaluation of total time leakage test data.

Figures 18a shows the test volume air mass vertically, as a function of elapsed test time shown horizontally, by the individual test point dots. The several lines connecting the initial W_0 mass point (at upper left) to the successive W_n mass points at different elapsed test times have slopes corresponding to the leakage rates computed for each of the successively longer elapsed testing times. Figure 18b shows the leakage rates plotted vertically in percentages of W_0 change, $\pm(W_{n-0}/W_0)$ per day, as a function of elapsed testing time (shown horizontally) from start of test to most recent mass measurement, in percent of initial mass change per day. In this case, all values of leakage rate shown in Fig. 18b are positive, because all line slopes in Fig. 18a are downward.

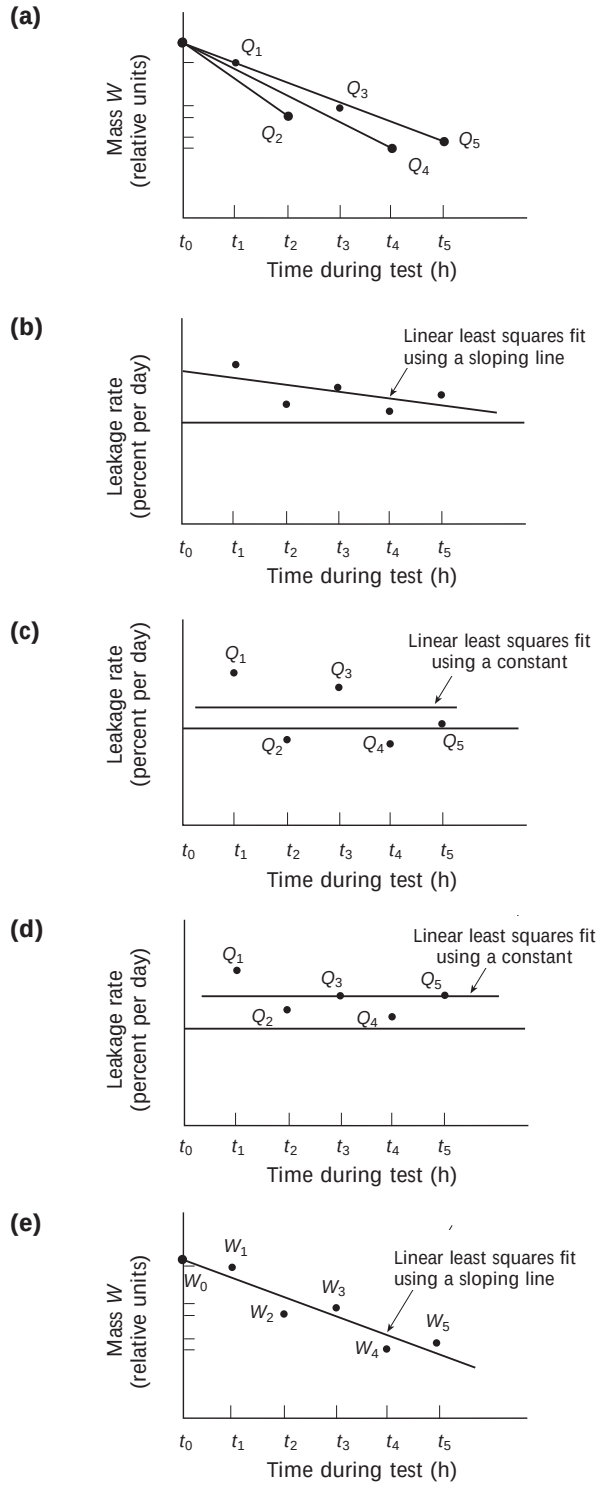
Limitations of Time Dependent Test Data from Pressure Change Leak Tests

From the example illustrated by Fig. 17, it is self evident that numerous different values for leakage rate could be derived from the same initial test data from an absolute technique test. For example, in many leak tests, it is considered appropriate to determine the leakage rates simply from the initial mass or pressure within an enclosure and the final mass or pressure at the end of some arbitrary testing time. In this case, Eq. 30 gives the endpoint leakage rate:

$$(30)\quad Q''_n = \frac{W_0 - W_n}{t_n - t_0}$$

This leakage rate corresponds to that indicated as Q_5 in Fig. 18a. Had the arbitrary test period been different in

FIGURE 18. Statistical techniques for pressure change leakage rate test: (a) leakage rates calculated from mass difference between starting mass and mass at test time; (b) leakage rates plotted as function of elapsed test time in percent of initial mass change per day; (c) average leakage rate; (d) total time test data with least squares fit to eliminate time dependency; (e) linear least squares fit drawn through mass point leakage test data shown in Fig. 17a.



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length, the leakage rate might have been equally well determined as Q_1, Q_2 or any other Q_n shown on a graph similar to that of Fig. 18a. For this reason, techniques of statistical analysis are often used to lend credence to leak testing data, where measured leakage rates are significantly influenced by test conditions, some of which may be chosen arbitrarily. Several different statistical techniques are described next, to illustrate their possibilities.

Estimating Constant Leakage Rates from Average or Least Squares Fit of Data

It might be reasonable to assume that leakage rates are essentially constant throughout the pressure change tests when the absolute pressure is essentially constant throughout the test and the size of any leakage path should not change. For this case, it would be necessary to fit a constant to the test data, as shown by the horizontal lines of Fig. 18c for the point-to-point analysis (same as average leakage rate) and of Fig. 18d to eliminate time dependency in total time analysis of the leak testing data of Fig. 17. The least squares relationship requires that the sum of the squares of the deviation $(Q_n - Q)$ should be a minimum. This is equivalent to requiring that the derivative $(d \cdot dQ^{-1})$ of this sum of mean squares with respect to Q should be equal to zero, as shown in Eq. 31 for the condition for minimum:

$$\textbf{(31)} \quad 0 = \frac{d}{d_Q}\left[\left(Q - Q_1\right)^2 + \left(Q - Q_2\right)^2 + \ldots + \left(Q - Q_k\right)^2\right]$$

Therefore, in the case in which point-to-point leakage rates are taken at roughly equal time intervals during the pressure change leak testing period, the linear least squares fit is equal to the simple arithmetic means value of all of the individual values for leakage rates. This greatly simplifies online data analysis during the leakage test, where the best linear least squares fit to the test data can be computed continuously during testing operations by the simple average of Eq. 32:

$$\textbf{(32)} \quad Q_a = \frac{Q_1 + Q_2 + \ldots + Q_k}{k}$$

Note that in Eq. 32, k is the total number of leakage measurements made at equally space time intervals, after $t = 0$.

Applying Least Squares Fit to a Line of Mass Point Leak Test Data

Figure 17a shows an example illustrating data collected in the mass point analysis technique before any data analysis has taken place. In Fig. 18e, these data have been fitted to a sloping line by a least squares technique. The slope of this line is drawn through mass point leakage test data shown in Fig. 17a. Leakage rate, percent per day = $100\,[(W_0 - W_n)/W_0]\,[24/(t_n - t_0)]$, where t is time (hour).

If it can be assumed that the leakage rate is constant with respect to elapsed time during the leakage test, the data are appropriate for analysis by the technique of least squares because of the independent nature of this type of analysis, an error during testing will result in only one bad datum and will not materially affect the leak testing results. If mass point analysis and fitting by least mean squares is carried out continuously as each set of data is taken during the mass point analysis, results are consistent, although not identical. When two hourly sets of data are combined to make a third set, the results always average as expected. With techniques using real time data analysis and graphical plotting in real time during tests, the approach to uniform rates of leakage can be seen and tests extended or terminated as appropriate to the quality and consistency of data.

The mass point of 95 percent confidence ranges from 0.05 to 0.2 times the measured leakage rate. By comparison, the 95 percent confidence interval may range from one half to twice the measured leakage rate with the total time technique and from two to 20 times the measured leakage rate with the point-to-point technique. For these reasons, many organizations prefer the mass point technique with continuous data analysis to the alternative techniques of analysis.

Formulas for Computing Least Squares Line Fitting Mass Point Leak Test Data

The theoretical basis for using least squares techniques to compute a leakage rate lies in the so-called Gauss-Markoff theorem. As applied to the measurement of leakage rates, the theorem states that, if the linear relationship between W and t is appropriate and if the W values are independent and equally variable, the *best estimators* of the slope and intercept of the line are given by least squares analysis. Here, *best* means two things: (1) the estimators are not biased and (2) the estimators have the smallest variances of any other unbiased estimators that might be derived from arbitrary linear

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combinations of the W values. The least squares line is given by Eq. 33:

$$\textbf{(33)} \quad W_a = At + B$$

where the slope A and intercept B are given, respectively, by Eq. 34 and 35:

$$\textbf{(34)} \quad A = \frac{n\sum t_i W_i - \sum W_i \sum t_i}{n\sum t_i^2 - \left(\sum t_i\right)^2}$$

$$\textbf{(35)} \quad B = \frac{\sum W_i \sum t_i^2 - \sum t_i W_i \sum t_i}{n\sum t_i^2 - \left(\sum t_i\right)^2}$$

Each t_i is the elapsed time between the clock time at which the initial reading is taken and the clock time at which the ith reading is taken. Thus, $t_1 = 0$ in all test situations, t_2 is the length of the time elapsed before the next reading and so on. In most test situations, the time intervals between tests will be constant but the formulas for A and B do not require constancy.

The leakage rate is expressed as the ratio of the rate of change of mass to the mass in the containment at time $t_1 = 0$. Because values of t_i have units of hours and percentage daily leakage rates are desired, the mass point leakage rate is expressed as a positive number of Eq. 36:

$$\textbf{(36)} \quad Q_{am} = -2400\ \frac{A}{B}$$

Note that B — not the mass W_0 measured at the initial time — is used as the denominator of Q_{am}. B is the better measure of the contained mass because W_0 has the same error structure as the W_i values.

The uncertainty in the estimated value Q_{am} is assessed in terms of the standard deviations of A and B and their covariance, followed by the computation of an upper limit of the 95 percent confidence level for Q_{am}. In what follows, the full details are spelled out. Conditions are stated that result in considerable simplification applicable to most leakage test situations.

Formulas for Computing Standard Deviation in Mass Point Leak Test Data

The estimate of the common standard deviation (following from the equally variable assumption) of the masses with respect to the line is given by Eq. 37:

$$\textbf{(37)} \quad S = \sigma = \sqrt{\frac{\sum_{n=1}^{n=K}\left(W_1 - W_i\right)^2}{n-2}}$$

where S is the standard deviation, W_i is the computed mass at time t_i (from Eqs. 33 or 35) and n is the number of the leak test measurements. Now, let the quantity K be defined by Eq. 38:

$$\textbf{(38)} \quad K = \frac{S}{\sqrt{n\sum t_i^2 - \left(\sum t_i^2\right)}}$$

Then, the standard deviation of the slope is given by Eq. 39:

$$\textbf{(39)} \quad S_A = K\sqrt{n}$$

The standard deviation of the intercept is given by Eq. 40:

$$\textbf{(40)} \quad S_B = K\sqrt{\sum t_i^2}$$

And the covariance of the slope and intercept is given by Eq. 41:

$$\textbf{(41)} \quad S_{AB} = K^2\left(-\sum t_i\right)$$

Confidence Limits for Mass Point Leak Test Data[2,3]

The confidence limit is a measure of the statistical consistency in test data. Figures 19 and 20 illustrate the meaning of the confidence limit in terms of the normal Gaussian distribution of data with random errors. The shaded area of the curve in Fig. 19 is equal to 95 percent of the area within the total Gaussian distribution curve when the latter is integrated from –X to +X. A 95 percent confidence limit means that 95 percent of the measurements will fall within the shaded range of leakage rates. It can also mean that, if another identical test was run, then statistically there is a 95 percent chance that the calculated leakage rate will be within the shaded range of Fig. 19.

In Fig. 20 the confidence limit is plotted vertically as a function of the dispersion index (plotted horizontally). The units of the dispersion index scale are the standard deviation of Eq. 37. The 95 percent confidence limit corresponds to the dispersion index value equal to three standard deviations. With a dispersion index equal to only one standard

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FIGURE 19. Normal Gaussian distribution curve. Shaded area includes 95 percent of the measurements in a normal distribution. After Fleshood[2] and Lau.[3]

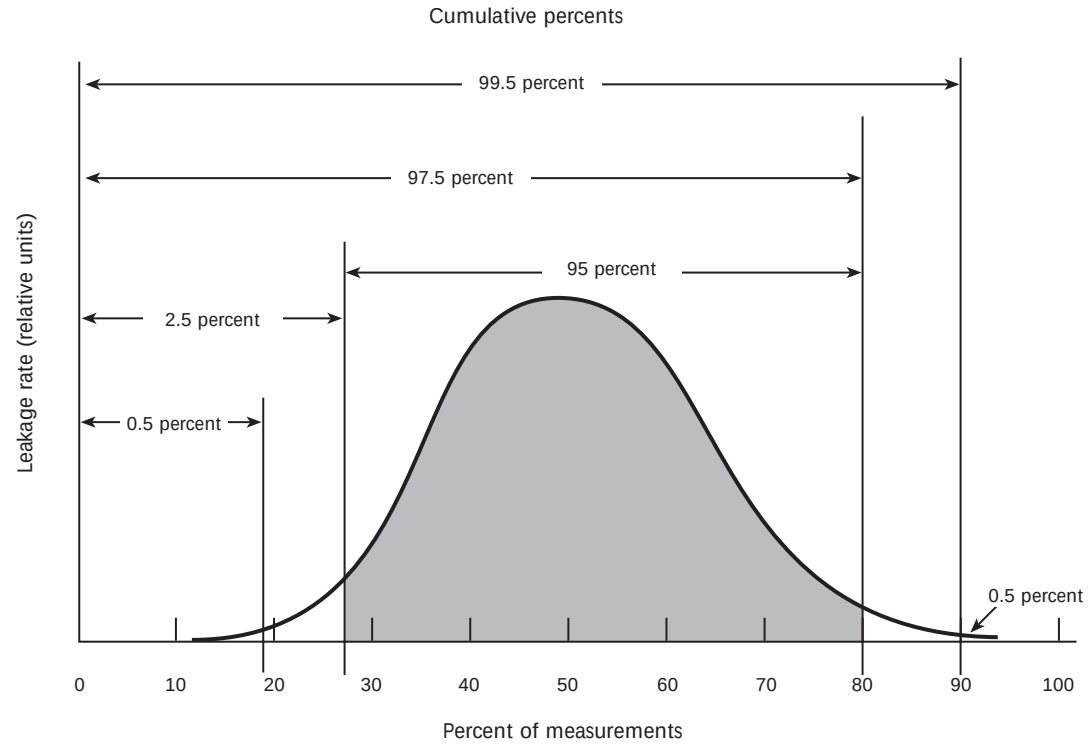
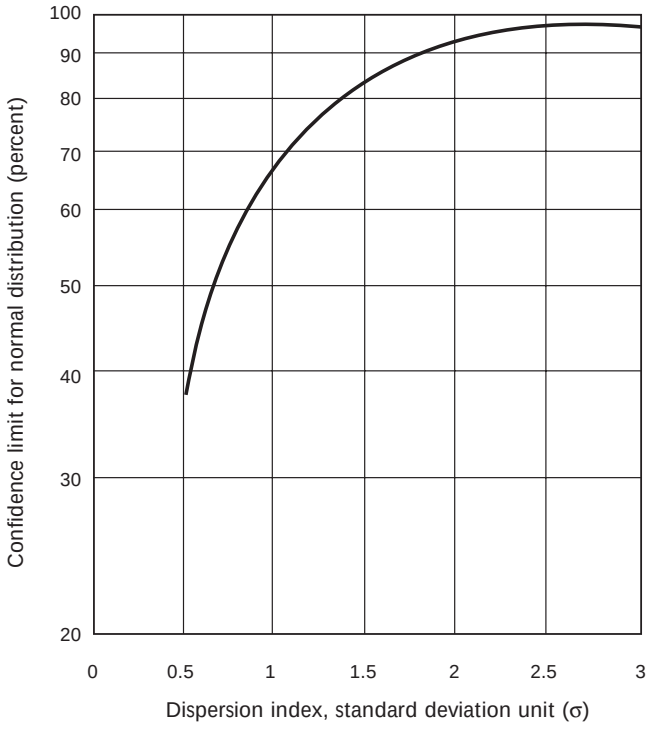


FIGURE 20. Percentage confidence limit plotted as a function of dispersion index, measured in standard deviation units (σ). Ordinate or curve shows what confidence level applies for each value of dispersion index shown on horizontal scale. After Fleshood[2] and Lau.[3]



deviation, the confidence limit taken from the curve of Fig. 20 would be reduced to about 70 percent. The universal standard deviation σ is defined by Eq. 42:

$$S = \sigma = \sqrt{\frac{\sum_{n=1}^{n=k} (Q - Q_n)^2}{k}} \quad (42)$$

Because the standard deviation and the confidence limit can be calculated easily with the aid of programmable hand calculators, microprocessors or minicomputers as the leak test progresses, the test operator can readily determine what percentage confidence level is attained. Decisions can then be made as to whether the test should be extended to attain the required degree of statistical confidence or discontinue until repairs are made to the test system or unreliable instrumentation is replaced.

Formulas for Calculating Approximate and Exact Limits of Confidence Level

The data of Table 3 relate the 95th percentile $t_{0.95}$ of the test data distribution

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to selected values for the number of degrees of freedom, $d_F = n - 2$, where n is equal to the number of leak test measurements of the mass W of contained air at the corresponding elapsed test time t, following the initial measurement at time $t = 0$.

The standard deviation of the slope S_A was defined by Eq. 39 and the standard deviation of the intercept S_B was defined by Eq. 40 for the least squares line defined by Eq. 33, namely $W = At + B$. In most leakage testing situations, the ratio $S_B \cdot B^{-1}$ is very small compared with the ratio $S_A \cdot A^{-1}$. Thus, an approximate upper limit (UCL) for the 95 percent confidence level of the percentage leakage rate of Eq. 36 is given by Eq. 43:

$$\sim \text{UCL} = Q_{am} + 2.4 \times 10^3 \; t_{0.95} \; \frac{S_A}{B} \tag{43}$$

Values for $t_{0.95}$ are selected from the data of Table 3, with $d_F = (n - 2)$.

For the case of $n = 20$ or more test points, following the initial data at time $t = 0$, the values of $t_{0.95}$ can be determined from Eq. 44:

$$t_{0.95} = 1.645 + \frac{1.576}{n-2} - \frac{2.4}{(n-2)^2} + \frac{57.6}{(n-2)^3} \tag{44}$$

where $d_F = n - 2$.

The adequacy of the approximate confidence level computed by Eq. 43 is measured in terms of its closeness to the exact Fieller type limit derived from the assumption that the W_i values are normally distributed about the straight line.[4] Experience with Type A leak tests has shown this approximation to be entirely adequate. However, to obtain the exact upper confidence limit, let:

$$a = B^2 - t_{0.95}^2 \, S_B^2 \tag{45}$$

$$b = AB - t_{0.95}^2 \, S_{AB} \tag{46}$$

$$c = A^2 - t_{0.95}^2 \, S_A^2 \tag{47}$$

The *exact* upper one sided limit of a 95 percent confidence level for the percentage per day leakage rate is given by Eq. 48:

$$Q_{am}:\text{UCL} = -2.4 \times 10^3 \times \frac{b - \sqrt{b^2 - ac}}{a} \tag{48}$$

Possible Reasons for Rejection of Erroneous Data from Pressure Change Leak Test

To obtain adequate accuracy in pressure change leakage rate testing, the instruments used for leakage measurements must be very accurate and sensitive. Nevertheless, fluctuations of leak test data points cannot be avoided. An outlying observation or an outlier is a datum widely different from the remaining observations in the data set. The outlying observation may be the result of an error in calculating a numerical value and could probably be corrected if properly identified. An outlier could also result from an instrument error or from an error in reading the instrument's indication. If this is known to be the case, the false reading should be removed from the data set. Hence, the testing engineer is always confronted with

TABLE 3. Tabular relationship between number of sets of leak testing data following initial W_0 and t_0, and 95th percentile of distribution, $t_{0.95}$, as a function of number d_F of degrees of freedom, after Fleshood.[2]

n	d_F	$t_{0.95}$
3	1	6.314
4	2	2.920
5	3	2.353
6	4	2.132
7	5	2.015
8	6	1.943
9	7	1.895
10	8	1.860
11	9	1.833
12	10	1.812
13	11	1.796
14	12	1.782
15	13	1.771
16	14	1.761
17	15	1.753
18	16	1.746
19	17	1.740
20	18	1.734
21	19	1.729
22	20	1.725
23	21	1.721
24	22	1.717
25	23	1.714
26	24	1.711
27	25	1.708
28	26	1.706
29	27	1.703
30	28	1.701
31	29	1.699
32	30	1.697
33	31	1.684
34	32	1.671
35	34	1.658
	∞	1.645

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the task of determining when a test datum is spurious or bad. A bad datum must be rejected; otherwise it will increase the standard deviation unduly. However, an apparently bad datum cannot be eliminated arbitrarily. There must be a valid basis for such rejection, such as a valid statistical criterion that identifies a true outlying observation.

Responsible Usage of Criteria for Leakage Test Data Rejection

Where a statistical criterion for testing an outlying datum is permissible, it cannot be applied selectively. That is, one should not apply the criterion to an outlier if its inclusion in the calculations would reduce the calculated leakage rate unless one is also prepared to reject an outlier whose inclusion would increase the calculated leakage rate or its upper confidence limit. For this reason, it is appropriate for the user to determine in advance of the leakage test whether or not the criterion is to be used and what the rejection level for data points will be if this criterion is applied.

It should be noted also that, if a high percentage of test points (such as two or more in 20 points) has to be rejected, the test engineer must conclude that either (1) the test instrumentation and procedure used on the leak test must be improved or (2) some systematic errors are not accounted for. In either case, the deviation does not follow a normal Gaussian distribution to which the statistical criterion could be properly applied. On the other hand, if most of the leak testing data are widely scattered, then an additional widely scattered datum is likely to be found to be acceptable according to the statistical criterion for identification of a true outlier. In this case, the standard deviations in measured leakage rates will be larger and the confidence limit will be smaller than the typical 95 percent upper confidence limit desired.

Data Rejection Criterion for Regression Data from Pressure Change Leak Test

Most traditional tests for an outlying observation are not appropriate for testing for an outlier in a regression situation, such as pressure change leakage rate testing, because the standard error of residual varies with time. An acceptable test criterion for a single outlier in a simple linear regression, however, is given by Tietjen et al.[5]

To use the technique proposed by Tietjen in pressure change tests, let t_i denote the ith time (hour) for the ith reading, W_i the corresponding air mass and $(W_i)_a = At_i - B$ the corresponding predicted mass from Eq. 33. Then the ith residual $w_i = W_i - (W_i)_a$ has a standard s_i:

$$\textbf{(49)} \quad s_i = S\sqrt{1 - \frac{1}{n} - \frac{\left(t_i - t_a\right)^2}{\sum\left(t_i - t_a\right)^2}}$$

In Eq. 49, the term S (standard deviation estimated from least square line) is given by Eq. 50:

$$\textbf{(50)} \quad S^2 = \frac{1}{n-2}\sum\left[W_i - \left(W_i\right)_a\right]^2 = \frac{\sum W_i^2}{n-2}$$

or

$$\textbf{(51)} \quad S = \sqrt{\frac{1}{n-2}\sum W_i^2}$$

and t is given by Eq. 52:

$$\textbf{(52)} \quad t_a = \frac{1}{n}\sum t_i$$

The standardized residual $r_i = w_i \cdot s_i^{-1}$ is next computed and the potential outlier W_i is the observation whose absolute value of the standardized residual r_i is the largest. Once $D = \max |r_i|$ is located, it is compared to a value in Table 4, to determine whether this quantity is significant. If D exceeds the table value, W_i is declared an outlier.

For a leakage rate test in which the data are collected at equal time intervals, Eq. 49 reduces to Eq. 53:

$$\textbf{(53)} \quad s_i = S\sqrt{1 - \frac{1}{n} - \frac{\left(i - i_a\right)^2}{\sum\left(i - i_a\right)^2}}$$

in which i_a is defined by Eq. 54:

$$\textbf{(54)} \quad i_a = \frac{1}{n}\sum i = \frac{1+2+3+\ \ldots\ +n}{n} = \frac{n+1}{2}$$

A still simpler form is shown in Eq. 55:

$$\textbf{(55)} \quad s_i = S\sqrt{1 - \frac{1}{n} - \frac{12\left(i - i_a\right)^2}{n\left(n+1\right)\left(n-1\right)}}$$

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TABLE 4. Values of critical deviation ratio D for data rejection for a one-sided statistical test, used in criterion for outlier data in containment leakage test.

Number of Observations (*n*)	5 Percent Rejection Level (*D*)	1 Percent Rejection Level
4	1.41	1.41
5	1.71	1.73
6	1.92	1.97
7	2.07	2.16
8	2.19	2.31
9	2.28	2.43
10	2.35	2.53
11	2.43	2.64
12	2.48	2.70
13	2.52	2.76
14	2.57	2.80
15	2.61	2.87
16	2.64	2.92
17	2.68	2.96
18	2.71	2.99
19	2.74	3.03
20	2.76	3.06
21	2.79	3.09
22	2.82	3.12
23	2.84	3.15
24	2.85	3.17
25	2.89	3.19
26	2.90	3.21
27	2.92	3.23
28	2.93	3.25
29	2.95	3.26
30	2.96	3.28
31	2.97	3.29
32	2.99	3.31
33	3.00	3.32
34	3.01	3.33
35	3.02	3.34
36	3.03	3.35
37	3.04	3.36
38	3.06	3.37
39	3.07	3.38
40	3.08	3.39
41	3.09	3.40
42	3.09	3.40
43	3.10	3.41
44	3.11	3.42
45	3.12	3.42
46	3.13	3.43
47	3.14	3.44
48	3.15	3.45
49	3.15	3.45
50	3.16	3.46
51	3.17	3.46
52	3.17	3.47
53	3.18	3.47
54	3.18	3.48
55	3.19	3.48
56	3.19	3.49
57	3.20	3.49
58	3.21	3.50
59	3.21	3.50
60	3.21	3.50

Example Application of Criterion Technique for Outlier Datum

This example illustrates the technique used for identifying and evaluating an outlier datum. For every point in time, Table 5 shows the containment air mass, its deviation from the linear least squares fit, the standard error of the residual and the standardized residual. In this example, with data generated at 15 min intervals from an actual test, the number of data points $n = 36$. With the measurements made at equal time intervals using Eq. 54:

$$i_a = \frac{36 + 1}{2} = 18.5$$

and

$$\sum W_i^2 = 28\,848.83$$

and using Eq. 50,

$$S = \sqrt{\frac{28\,848.83}{36 - 2}}$$

The estimated standard deviation of the containment air mass from the linear least square fit is given by Eq. 55 so that

$$s_i = \sqrt{\frac{28\,848.83}{36 - 2}} \times \sqrt{1 - \frac{1}{36} - \frac{12\left(i - 18.5\right)^2}{\left(36\right)\left(37\right)\left(35\right)}}$$

The maximum absolute standardized residual is found from the last column of Table 5 for $i = 28$, a where $D = |r_i| = 2.08$. The absolute magnitude is indicated by $|r_i|$.)

From Table 4, it is seen that, for $n = 36$, a D statistic as large as 2.08 occurs more often that 5 percent of the time; hence, the potential outlier should not be rejected on statistical ground. Because the largest standardized deviation is not rejected, no other datum can be rejected statistically, either.

If for the datum, the residual were –96 instead of –59.07, just to illustrate the 5 percent data rejection criterion, one would have obtained $D = 3.38$. From Table 5, it is seen that the datum would have occurred less than 5 percent of the time and could have been rejected statistically.

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System Ability to Measure Leakage Rate

The purpose of a leak testing instrumentation selection guide is to determine the ability of a specific instrumentation system to measure the overall leakage rate of a pressurized system adequately. This selection guide is not based on a statistical analysis of the leakage rate calculations, but has been developed for the purpose of selection of instrumentation adequate for the required leakage measurements. In evaluations made using one guide,[6] the errors of individual instruments used for measurement of pressure and temperature or dew point are combined using a statistical root-sum-square formula:

$$\text{(56)} \quad \delta Q = \text{ISG} = \pm \frac{2.4 \times 10^3}{t} \times \sqrt{2\left(\frac{e_P}{P}\right)^2 + 2\left(\frac{e_T}{T}\right)^2 + 2\left(\frac{e_{P_v}}{P}\right)^2}$$

ISG is the instrumentation selection guide; δQ is the standard deviation δ of the leakage rate Q (percent per day); t is the test duration (hour); P is the containment atmosphere total absolute pressure; P_v is the containment atmosphere partial pressure of water vapor; T is the containment atmosphere weighted average absolute dry bulb temperature; e is the error associated with measurement of change in a given parameter; E is the error associated with sensor sensitivity.

TABLE 5. Example of calculations for a single outlier test datum in pressure change test for leakage rate.

Datum i	Air Mass W_i	Linear Least Squares Fit W	Residual from Least Squares Fit $w_i = W_i - (W_i)_a$	Standard Error of Residual s_i	Standardized Residual $r_i = w_i \cdot s_i$ $D = \lvert r_i \rvert$
1	735478.1	735443.37	34.73	27.53	1.26
2	735473.5	735442.46	31.04	27.67	1.12
3	735475.8	735441.54	34.26	27.79	1.23
4	735451.1	735440.63	10.47	27.91	0.38
5	735439.8	735439.71	0.09	28.02	0.00
6	735449.6	735438.80	10.80	28.12	0.38
7	735444.2	735437.88	6.32	28.21	0.22
8	735426.6	735436.97	–10.37	28.30	–0.37
9	735415.1	735436.06	–20.95	28.38	–0.74
10	735396.7	735435.14	–38.44	38.45	–1.35
11	735391.3	735434.22	–42.92	28.51	–1.51
12	735426.3	735433.31	–7.01	28.56	–0.25
13	735440.7	735432.39	8.31	28.61	0.29
14	735424.8	735431.48	–6.68	28.64	–0.23
15	735432.3	735430.56	1.74	28.68	0.06
16	735435.3	735429.65	5.65	28.70	0.20
17	735409.1	735428.73	–19.63	28.71	–0.68
18	735423.5	735427.82	–4.32	28.72	0.15
19	735436.4	735426.90	9.50	28.72	0.33
20	735436.4	735425.99	10.41	28.71	0.36
21	735391.8	735425.07	–33.27	28.70	–1.16
22	735392.1	735424.16	–32.06	28.68	–1.12
23	735452.8	735423.24	29.56	28.64	1.03
24	735455.5	735422.33	33.17	28.61	1.16
25	735448.9	735421.41	27.49	28.56	0.96
26	735371.3	735420.50	–49.20	28.51	–1.73
27	735387.9	735419.58	–31.68	28.45	–1.11
28	735359.6	735418.67	–59.07	28.38	–2.08
29	735395.4	735417.75	–22.35	28.30	–0.79
30	735375.0	735416.84	–41.84	28.21	–1.48
31	735407.8	735415.92	–8.12	28.12	–0.29
32	735445.5	735415.01	30.49	28.02	1.09
33	735446.5	735414.09	32.41	27.91	1.16
34	735447.0	735413.18	33.82	27.79	1.22
35	735464.2	735412.26	51.94	27.67	1.88
36	735437.0	735411.35	25.65	27.53	0.93

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Nature of Systematic Errors and Random Errors

In estimating the magnitude of the uncertainty or error in the value assigned to a quantity (mass of air in containment) as the result of measurements, a distinction must be made between two general classes of error, systematic and random. Systematic errors are those errors associated with a difference between the true value and the measured parameter produced by predictable or identifiable effects. Calibration of the leakage rate measuring system traceable to the National Institute of Standards and Technology removes systematic errors or reduces them to an acceptable magnitude. Random errors are those whose magnitude and sign fluctuate in a manner that cannot be predicted from a knowledge of the measurement system, the system calibration certification or the conditions of measurement.

Techniques for Verification of Accuracy in Leakage Test Measurements

An acceptable technique to verify that a significant calibration shift or system change has not occurred is to make a definite, known change in the magnitude of the measured value using a different, independent, calibrated instrument. This is accomplished with the verification test. Such comparison provides a check to verify that a significant calibration shift or other system change has not occurred and that the measurement system systematic error has remained essentially constant. Therefore, a successful verification test confirms that the leakage rate test system systematic error is within acceptable limits. Any other error associated with leakage rate measurement is then due to random error.

For verifying the validity of the leakage rate test measurements during the change leak tests, the following supplemental techniques described in Appendix C of ANSI/ANS-56.8-1981 may be used.[6]

Supplemental Technique Using Calibrated Leak[6]

A calibrated or measurable leak is intentionally superimposed on the existing leaks in a system under test. A practical and simple arrangement uses the orifice leak of a microadjustable instrument flow valve installed at a convenient penetration of the containment system. The flow through the valve is measured by means of a calibrated flow meter or rotameter. The leak orifice is selected to provide a flow under the test pressure condition equivalent to 75 to 125 percent of the leakage rate specified for the acceptance test.

The test procedure involves placing the calibrated leak system into operation after the leakage rate test in progress is completed. The flow meter readings are then recorded at least hourly. Concurrently, readings of the containment system leakage measuring system record the composite leakage of both the containment system leakage rate and the superimposed leakage rate.

The readings of the flow meter as a function of time enable calculation of the average leakage rate through the calibrated orifice. From the analysis of the readings taken with the leakage measuring system, the composite leakage rate Q_c is determined.

The duration of the superimposed leakage verification test depends on the leakage rate involved and generally requires at least 4 h with a minimum of ten sets of data.

Supplemental Technique Using Metered Mass Change

A mass step change verification test using a metered quantity of air. A small quantity of air is either metered into or out of the containment over a short time interval. This mass change indicated by the leak test instrumentation prior to and following the metered mass change is compared to the metered mass change.

The mass step change verification test is conducted as follows. At the end of the leak test a mass of air is metered through a flow meter, either into or out of the containment over a short time interval. This metered mass change is compared to the mass change indicated by the leak test instrumentation before and after the metered mass change. The change in mass calculated from the test instrumentation must agree within 25 percent with the metered mass change.

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PART 2. Pressure Change Leakage Rate Tests in Pressurized Systems

Operating Principles of Pressure Change Leakage Rate Testing

Leakage rate testing by measurement of pressure changes in closed volumes requires that the system under test be maintained at a pressure other than ambient atmospheric pressure. Pressure change leak tests can be made with either an evacuated or a pressurized test system. The leakage rate Q is equal to the measured pressure change ΔP multiplied by the test system's internal volume V and divided by the time interval Δt, required for the change in systems pressure to occur:

$$Q = V \frac{\Delta P}{\Delta T} \quad (57)$$

where Q is leakage rate ($Pa \cdot m^3 \cdot s^{-1}$); V is enclosed system volume (cubic meter); $\Delta P = P_1 - P_2$, which is pressure change during leak test (pascal); $\Delta t = t_2 - t_1$, which is time interval during leak test (second).

The pressure change leak testing procedure is used primarily for leakage measurement in large systems. However, with minor modifications, the pressure change technique can be used to measure leakage rates on test systems of any size. This procedure is used only for measurement of leakage and is not well suited for location of individual leaks. However, a leak may be localized to a closed part of a system under test by pressure change test techniques.

Sensitivity of Pressurized Mode Leakage Tests by Pressure Change Techniques

The sensitivity of leakage measurement during leak testing of pressurized systems with the pressure change technique depends on the minimum detectable magnitude of pressure variation. Static pressure is measured at the start, at intervals and at the end of the leak testing period. The sensitivity of this static leakage measurement largely depends on the time duration of the test and the sensitivity and accuracy of the pressure measuring instruments. In the absence of uncontrolled temperature changes or severe outgassing effects, longer time intervals between initial and final measurements permit more sensitive measurements of leakage rate.

The accuracy of measurement of leakage rates in the pressurized mode of pressure loss leak testing depends on how precisely the test volume V is calculated and on how accurately the changes in pressure and temperature can be measured. If the leakage rate is measured as a percentage of total enclosed fluid (mass) lost per unit of time, then precision in calculating the enclosed volume may not be required. When using properly calibrated pressure measuring instruments in the pressurized mode, the accuracy of leakage measurement by the pressure loss technique can often be traced to the National Institute of Standards and Technology.

Sources of Error in Pressurized Mode Leakage Tests by Pressure Change Techniques

The test procedure for the pressurized mode of leakage measurement consists of filling the test system with gas and observing any pressure decrease. The fundamental relationship is given in Eq. 57.

Two large sources of error exist in this technique. The volume of the test system is difficult to calculate for a large or complex system; however, it can be measured by the additional leakage technique, which is also known as a verification test or a proof test in practice. An additional known leak is added to the system under test. The system volume is then calculated from the effect of the additional leakage on the observed rate of pressure decrease.

The second source of error inherent in the pressure change technique exists when temperature variations during the test cycle tend to vary the pressure in the system. This error can be corrected by measuring system temperature during the leak test. The pressure effect of temperature variations can be calculated by using the ideal gas laws. In an

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alternative technique for correction for interfering effects, a reference volume is placed in the system under test and the variations of pressure differential between this closed reference system and the test system are observed. Specific illustrative examples of such calculations are given later in this chapter.

Advantages and Limitations of Pressure Change Techniques of Leak Testing

Two major advantages of the pressure change technique of leak testing are the following.

1. Instrumented large scale pressure or vacuum systems can often be leak tested by using pressure gages already installed on the system to be tested.
2. No special tracer gas is required.

Two major disadvantages of the pressure change technique of leak testing are the following.

1. The time required for leak testing can be rather long.
2. This test technique does not permit precise leak location without auxiliary techniques.

Pressure change leak tests can be conducted on any contained volume that will withstand the internal pressure used to apply the necessary pressure differential across the boundaries of the test volume.

Pressure Change Leakage Rate Testing of Constant or Variable Volume Systems

Pressure change leak testing is a nondestructive test technique used for determining the total leakage rate through the walls or pressure boundaries of a structure tested at a specific pressure. Pressure change leak tests can be conducted on any contained volume that will withstand either an internal pressure differential (pressure system) or an external pressure differential (vacuum system) across the boundary of the test volume. For constant volume or variable volume pressure systems with gage pressure greater than atmospheric pressure, the pressure change leak test is also commonly identified by names such as pressure hold test, pressure loss test, pressure decay test or leakage rate test.

A constant volume system is a rigid structure such as a pressure vessel where the physical change in the size of the system due to temperature variation is so small relative to total contained volume that it can be ignored. A variable volume system is a flexible structure such as a vapor tank in which the volume changes to maintain a uniform internal pressure. For large volume systems, the gas temperature and dewpoint in the system under test should be measured if possible throughout the time period used for the pressure change leak test.

Selection of Pressurizing Gases for Pressure Change Tests for Leakage Rates

Pressurizing gases used for pressure change leakage rate testing should obey the ideal gas laws to a reasonable degree. The most commonly acceptable gases in this category are air, nitrogen, helium, argon and carbon dioxide. Use should never be made of hazardous pressurizing gases such as toxic gases or oxygen (which supports combustion of oils, grease or hydrocarbons). Similarly, combustible gases such as propane, butane or acetylene should never be used for pressurizing because of the dangers of explosion.

The common halogen rich tracer gases (such as refrigerant-12 or refrigerant-22) should not be used as pressurizing gases for absolute pressure leak testing because they do not obey the ideal gas law and can produce erroneous leak testing results. If refrigerant gases have been used in a system as the tracer gas for preliminary halogen detector probe leak testing, these chlorinated hydrocarbons must be purged from the system under test prior to performing a pressure change test for leakage rates.

Precautions in Preparation for Pressure Change Leakage Rate Testing

The following preliminary leak testing techniques and practices are desirable before pressure change leak testing during fabrication or erection of large items such as pressure vessels or liners, test channels, double gasket flange interspaces or airlocks, for example. Before conducting a pressure change test, preliminary leak testing should be performed to detect and eliminate leakage from connections external to the test object. Otherwise, such external leaks could affect the results of the pressure change leak test. The type of preliminary testing that should be performed is usually given in the written

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procedure for leak testing of the specific products or assemblies.

When preliminary leak testing includes a halogen detector probe test, the halogen mixture should be purged from the test system before conducting a pressure change test. Also, before starting the pressure change test, the operator should always close the inlet isolation valve and disconnect the pressurizing line or manifold; then, tests to locate all leaks should be performed on this valve connection and the pressure gage connection.

If adverse working conditions are encountered during the day work shift, it is often best to perform a short duration pressure hold leak test of a small volume system during a less busy shift or when there is less interference. A longer overnight leak testing period with more stable ambient temperature conditions may make it possible to pass a test object or a channel test zone which otherwise might improperly have appeared to have failed during the usual 1 or 2 h leak test during variable daytime conditions. For such reasons, if a test object is on the borderline of acceptance for a pressure hold leak test, it is advisable to continue the test overnight or during some other convenient longer period not subject to interference from other work activities.

Typical Test Sequence for Pressure Change Leak Testing in Industry

After completing all required preliminary testing and after purging of the test system (if halogen rich refrigerant was previously used), the pressure change leakage rate test is performed in the following steps:

1. A calibrated pressure gage is connected to the contained volume under test. When necessary, calibrated equipment to measure dry bulb temperature and dewpoint temperature (humidity) is also installed and verified after installation.
2. A pressurizing line is then attached to a valve connection on the test system. The test object is pressurized to the designated test pressure (usually with compressed air). The pressurized test system is next isolated from the pressurizing source with the valving system. The pressurizing source is then disconnected and a solution film bubble emission test is next performed on the seat and stem of the pressurizing connection valve.
3. The pressure gage is observed to detect any consistent loss in pressure not related to temperature change. If the pressure remains reasonably stable, the leak test can be started. If the pressure constantly decreases more rapidly than the allowable rate of pressure decrease, additional preliminary testing for leakage should be performed.
4. Only after it has been established that no detrimental leakage exists in external connections, valves or other components should the pressure change leak test be started and test data be recorded.
5. If, during the course of a pressure change leakage rate test, any leak testing instruments malfunction or become damaged, they should be replaced with properly functioning instruments (if these instruments are indispensable to the satisfactory completion of the test). Then the leakage rate test should be repeated from the start.
6. A pressure change leakage rate test may be concluded at the end of the required test period if the magnitude of the pressure loss or leakage is within the specified allowable rate. If the test results are borderline, consideration should be given to continuing the test time period to increase the reliability of the test data. If the pressure loss or leakage rate is in excess of the allowable limits, the system should be reinspected by other testing techniques to detect the location of the excess leakage.
7. When leaks with unacceptable leakage rates are located, each such leak should be repaired; then local retests should be used to prove that the leakage has been eliminated or reduced to an acceptable level for each leak. Finally, the entire system should be retested by the specified pressure change leak testing technique to ensure that total leakage rates are within acceptable limits.

Relation of Pressure to Temperature (Volume Constant)

Calculations of leakage rates from absolute pressure readings in constant volume test systems depend on test variables including test time, temperature and pressure. For tests of large systems, it is also necessary to consider the effects of water vapor pressure within the contained volume. The static relation between the pressure, volume and temperature of a fixed of gas can be written as Eq. 58:

$$\frac{PV}{T} = \text{constant} \quad \textbf{(58)}$$

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where P is absolute pressure (pascal or $lb_f \cdot in.^{-2}$ absolute); V is volume of container (cubic meter or cubic inch); T is absolute temperature (kelvin or degree rankine). Consistent units, such as SI only or English units only, should be used for each term in Eq. 58 and in succeeding equations relating the same parameters.

The basic equation for pressure change leak testing used when comparing two different conditions for a given mass of the same gas (derived from Eq. 58 for test conditions 1 and 2) is given by Eq. 59:

$$\textbf{(59)} \quad \frac{P_1 V_1}{T_1} = \frac{P_2 V_2}{T_2}$$

or

$$\frac{P_1}{P_2} = \frac{T_1}{T_2} \frac{V_2}{V_1}$$

For a pressure change of a given (constant volume) system, the initial volume V_1 and final volume V_2 remain essentially the same. Therefore, for the constant volume test systems, $V_1 = V_2$ and Eq. 59 can be written more simply:

$$\textbf{(60)} \quad \frac{P_1}{P_2} = \frac{T_1}{T_2}$$

or

$$P_1 = P_2 \frac{T_1}{T_2}$$

As can be seen from the first form of Eq. 60, absolute pressure varies in direct proportion with the absolute temperature. In the absence of significant leakage, the absolute pressure increases in proportion with an increase in contained gas temperature. Conversely, lowering the gas temperature lowers the absolute internal gas pressure proportionately.

Calculation of Pressure Change with Absolute Pressure and Temperature Readings

The pressure change leak test is performed by pressurizing a closed system to a specific pressure and isolating the system. Time, temperature (internal) and system pressure are recorded systematically for some test period. For large volume systems, dewpoint would also be measured to permit determination of the partial pressure of water vapor. Comparison of initial pressure P_1 and final pressure P_2 can be used to determine the total system leakage rate for the case of the specific test pressure selected for the leak test. Working equations for these calculations are presented below.

If the pressure change during the test is designated by ΔP, Eq. 61 corrects for a change in temperature.

$$\textbf{(61)} \quad \Delta P = P_1 - P_2 \frac{T_1}{T_2}$$

To calculate the pressure change per unit of time, use can be made of Eq. 62, in which the time duration of the test (between successive readings in a sequence of readings or between start and finish of a leak test) is taken as Δt:

$$\textbf{(62)} \quad \frac{\Delta P}{\Delta t} = \frac{P_1 - P_2 \frac{T_1}{T_2}}{\Delta t}$$

Extending the time duration or length of a pressure change leakage test will increase the magnitude of the pressure change and usually result in an increase in the accuracy and reliability of the leak test results.

Calculation of Pressure Change with Gage Pressure and Thermometer Readings

With small systems, pressures are sometimes measured as gage pressures and gas temperatures are measured with ordinary thermometers or surface temperature indicators on the celsius or fahrenheit temperature scales. These pressures must be converted to absolute pressures and the temperatures must be corrected to absolute temperatures in kelvin or degree rankine. If the pressure change test is made under conditions that do not require measurement of the barometric pressure, the barometric pressure can be assumed to be one standard atmosphere (101.3 kPa or 14.7 $lb_f \cdot in.^{-2}$ absolute). The gas pressure change is computed by either Eq. 8 for celsius temperatures or Eq. 9 for fahrenheit temperatures.

If barometric readings of the pressure of the earth's atmosphere are required and barometric pressures vary, each individual gage pressure measurement must first be corrected to the absolute pressure value by Eq. 63:

$$\textbf{(63)} \quad P = P_{\text{gage}} + P_{\text{barometer}}$$

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where P is absolute pressure (kilopascal or $lb_f \cdot in.^{-2}$ absolute); P_{gage} is gage pressure (kilopascal or $lb_f \cdot in.^{-2}$); $P_{barometer}$ is barometric pressure (kilopascal or $lb_f \cdot in.^{-2}$) obtained in uncorrected form from local weather bureau or read from a precision barometer and converted to pressure units. Where pressures are measured in other units such as torr, inch of mercury or foot of water, the pressures must be converted consistently either to English units or preferably to SI units. After conversions have been made, the rate of absolute pressure change can again be calculated by use of Eqs. 10 or 11.

Data Acquisition, Analysis and Recording Systems for Leakage Rate Testing

Data recording for large scale pressure change leakage tests is made simpler by sophisticated numerical data acquisition systems. There systems automatically multiplex the conditioned signals from the pressure, temperature, dewpoint and flow measuring sensors (during the verification phase of leak testing) at preset automatically timed intervals. Data are transmitted through an interface for numerical analysis by computer, recorded on magnetic tape or disk systems, displayed by printout or graphical recordings and evaluated by error analysis and statistical techniques.

In many cases, this numerical test data analysis system can analyze the data by progressive analysis (with least mean squares fit to straight line approximations of leakage as a function of testing time). Computers provide the fastest and most accurate technique for analysis of the pressure change leak test data. The data can be fed into the computer directly from the acquisition system interface, from tape or manually from printer or recorder readouts. This absolute technique analysis of leakage rate may be performed by mass point or leakage rate point-to-point, point-to-point cumulative and total time statistical analysis techniques.

Minicomputer Integrated Leakage Rate Measurement System

Integrated leakage rate measurement systems are available that include all components from input sensors to minicomputer analyses of test data. This system will measure and record the absolute pressure, the dewpoint temperature and the dry bulb temperature of the air within the system under leakage test. In addition, the system displays the ambient pressure and temperature conditions. Flow measurements, vital to leakage verification tests, are also integral functions. This system accommodates the superimposed leak tests technique or the pumpback technique (the mass change verification leak test). The leakage test data are represented by a visual display, a printed record of the raw test data and a concurrent minicomputer calculation of the leakage rates, in several forms.

Components of Integrated Leakage Rate Measurement System

Figure 21 shows a schematic diagram of the components and system used in an integrated leak testing system. Typical leakage rate computations are based on measurements of the changes in the absolute pressure, water vapor pressure and the dry bulb temperature. The absolute pressure is measured with a fused quartz Bourdon tube. The low internal viscosity of fused quartz makes it the most perfectly elastic material available. This type of pressure sensor has no measurable hysteresis. It also has fast response, high resolution and high accuracy. The water vapor pressure is measured by use of chilled mirror dewpoint sensors and is presented to the minicomputer as a dewpoint temperature in degrees celsius or fahrenheit.

The dry bulb temperature is measured by resistance temperature detectors and is also presented to the minicomputer as digital data. Because the changes in the test parameters are small in magnitude, all input sensors must be capable of high sensitivity, accuracy, repeatability and resolution. Similar high accuracy, high resolution and reliability are required of the electronic networks and digital computer analyses.

Microprocessor Data Acquisition and Analyses with Leakage Rate Measurement System

A microprocessor (minicomputer) controls the minicomputer data acquisition and raw data recording system. The microprocessor system includes both read-only memory (ROM) and random access memory (RAM), a scanner system and various interfaces with sensor and output system components. Digital data for pressure, dry bulb temperature and dewpoint temperature are presented in ASCII (American Standard Code for Information Interchange) to the computer which then operates on these raw test data and calculates the leakage rate (see discussion below).

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Example of Analysis of Data from Pressure Hold Test of Small Volume Test Object

Table 6 shows leak test data analysis for pressure hold tests of a small volume test object with an allowable temperature corrected pressure loss of 0.5 $lb_f \cdot in.^{-2}$ in 2 h. In these tests, corrections for variations in barometric pressure were not required.

Analysis of data for the first pressure hold test on day one by means of Eq. 8 shows that the test object has failed the requirements of the pressure hold test:

$$\frac{\Delta P}{\Delta T} = \left[(42.0 + 14.7) - (42.0 + 14.7)\frac{89.5 + 460}{95.8 + 460}\right] \cdot 2\ h^{-1}$$

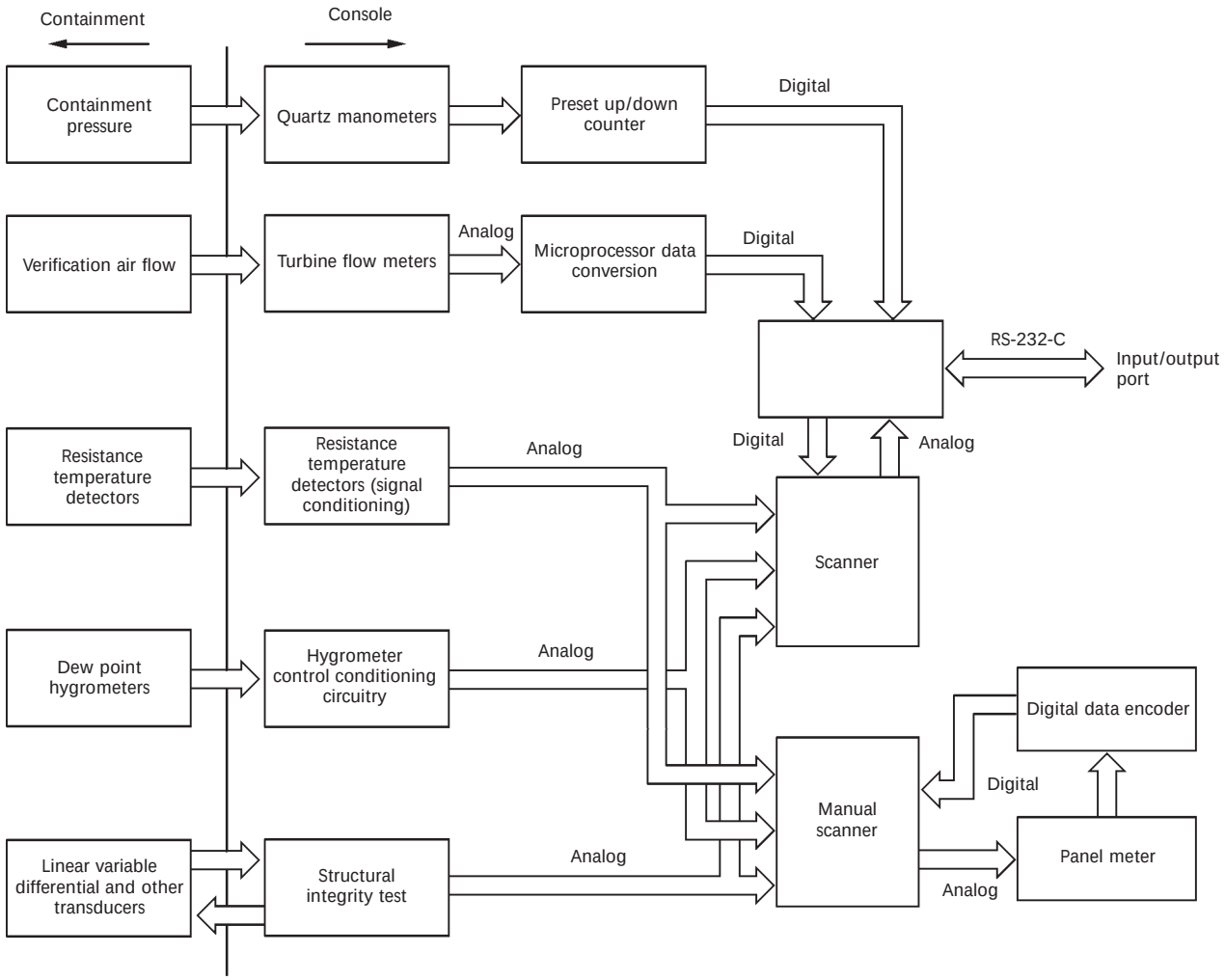
$$= 56.7 - 56.7\ \frac{549.5}{555.8}\ \text{loss in 2 h}$$

$$= 0.6\ lb_f \cdot in.^{-2}\ \text{loss in 2 h}$$

$$= 0.3\ lb_f \cdot in.^{-2} \cdot h^{-1}$$

This leakage rate exceeds the maximum acceptable temperature corrected pressure loss of 0.5 $lb_f \cdot in.^{-2}$ in 2 h, so the test results are not satisfactory. A leak was located in the pressure zone and the welds repaired. The pressure hold test was then repeated four days later with the results shown in the last two columns of the table in the left hand column. In this case, analysis of the pressure hold test data showed:

FIGURE 21. Information flow diagram for minicomputer controlled integrated leakage rate measurement system using a microcomputer, dual disk memory and instrument display console.



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$$\frac{\Delta P}{\Delta T} = \frac{56.7 - 56.7\frac{550.0}{550.7}}{2\text{ h}} + \frac{56.7 - 56.6}{2\text{ h}}$$

$$= 0.1\,\text{lb}_\text{f}\cdot\text{in.}^{-2}\text{ loss in 2 h}$$

$$= 0.05\,\text{lb}_\text{f}\cdot\text{in.}^{-2}\cdot\text{h}^{-1}$$

This leakage rate is well below the maximum allowable temperature corrected pressure loss rate of 0.5 $\text{lb}_\text{f}\cdot\text{in.}^{-2}$ in 2 h, so the test object is now acceptable.

The calculations in SI would reflect the fact that 1 $\text{lb}_\text{f}\cdot\text{in.}^{-2} \cong 6.9$ kPa.

Determining If Pressure Hold Test is Completed or Should Be Extended

A pressure hold leakage rate test may be concluded at the end of the required test period if the magnitude of the pressure loss is within (lower than) the specified allowable rate of pressure loss. If the test results are subject to question, the test can be continued over a longer time period to increase the reliability of the test data. If the extended test confirms that the actual pressure loss rate is in excess of the specified allowable limits, the system should be reinspected to detect the locations of the excess leakage. The system leaks should then be repaired and the system retested to the same specifications and procedures.

Leak Testing Techniques Using Cyclic Repressurization with Compressor

Intermittent operation of a compressor can be used for leakage measurements by evaluating the load cycle (duty cycle) when a compressor of known capacity maintains a specified pressure in the system under test. (The duty cycle is the ratio of the time the pump operates, *on* time, to the total time testing — *on* time plus *off* time for the pump.) With large rates of leakage, the compressor must operate for a large proportion of the test time. With low leakage rates, the same compressor need operate only occasionally for relatively short time periods. If it is desired to measure the absolute value of leakage by this technique, the capacity of the compressor at the test operation pressure should be known or be determined in a separate compressor calibration test.

When leak testing by the cyclic pressurization technique, operators need two stopwatches and a rapid response pressure gage with a clear scale. Prior to the first measurements, the compressor is allowed to charge the system under test to its normal operating (or delivery) pressure. The compressor control valve is then shut off to isolate the test vessel. The compressor is allowed to operate under no load conditions while the pressure in the test vessel falls off to a suitable lower pressure limit value, well below the initial compressor delivery pressure. When this pressure limit is reached, one stopwatch is started and the compressor is put under load by manual opening of the isolation control valve. During this operation step, the compressor pumps air or gas into the test vessel to raise its internal pressure. This first stopwatch is stopped when the test pressure has risen to a predetermined upper pressure limit.

When this upper limit is reached, the compressor is cut off by closing the isolation valve of the test vessel and the second stopwatch is started. When the internal pressure of the test vessel or system again falls to the original lower pressure limit, the second stopwatch is stopped and reset. Then the first watch is started as the compressor is again put under load to repressurize the test system. A new cycle of pressurization is initiated and the alternating stopwatch readings for pressurization time and leakage time are taken. Four or five cycles of repressurization and pressure decay are carried out in succession to ensure that the compressor is running under constant and reproducible conditions, as required to obtain accuracy in the leak test measurements.

TABLE 6. Pressure hold test data.

Date:	Day One		Day Four	
Time:	**09:50**	**11:50**	**15:30**	**17:30**
Average surface temperature, °C (°F)	31.9 (89.5)	35.4 (95.8)	32.2 (90.0)	32.6 (90.7)
Actual test pressure, kPa ($\text{lb}_\text{f}\cdot\text{in.}^{-2}$ gage)	290 (42.0)	290 (42.0)	290 (42.0)	290 (42.0)
Final temperature corrected test pressure, kPa ($\text{lb}_\text{f}\cdot\text{in.}^{-2}$ gage)		285 (41.4)		289 (41.9)
Loss in test pressure, kPa ($\text{lb}_\text{f}\cdot\text{in.}^{-2}$)		4.1 (0.6)		0.7 (0.1)

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The cyclic repressurization leak test technique is based on the assumption that no gas is supplied to the system under test during the no load period. With a reciprocating pump compressor unit, when the load is removed it is not uncommon for the compressor to continue to deliver some gas during the no load period. This can be avoided by ensuring that the compressor is provided with a delivery system that enables all gas in the intercooler to be discharged into the atmosphere during the no load period.

Advantages and Limitations of Cyclic Repressurization Leak Testing

The cyclic repressurization leak testing technique has the advantage of requiring only very simple equipment. Its accuracy in leakage measurements is less than the accuracy of more direct leak testing techniques. It is subject to random errors caused, for instance, by malfunctioning compressor valves. Therefore, these valves should be checked for satisfactory operation before starting each cyclic pressurization leak test. The cyclic pressurization test does not indicate the volume of the system under test, nor does it provide means for leak location.

When several compressors are available, the compressor selected for the leak tests should be one which if possible gives charging times at least as long as the leakage times. It is not advisable to operate the compressor under part load conditions, because its delivery capacity is rarely determined with the same accuracy for lower loads as for full load. Compressors with dead space regulation have a part load capacity that may differ between (1) a compressor calibration test and (2) a system leakage test, if the quantity and temperature of the compressor cooling water are different in these two cases.

Localizing Leaks in Low Pressure Gas Mains

Gas utilities use a modification of the pressurizing mode of leakage measurement to localize leaks in gas mains. The main is tested, section by section, with the leak locator inside the pipe. The leak locator consists of the following parts:

1. A flexible frame on which are spaced two rubber gas bags jointed by a rubber tube. These bags are pressurized to seal off a short section of gas pipeline for the leakage test.
2. A rubber dual tubing (two separate tubular passages) of length sufficient to reach from the rubber bags to a leak test control panel. One of the tubular passageways connects to the rubber bags and is used for their pressurization. The second tubular passageway extends through the adjacent rubber bag and opens into the pipeline interior space between the two rubber bag seals. This tube transmits the contained natural gas pressure to the control panel, where any loss of pressure due to leakage of gas from the test volume can be monitored.
3. A control panel with an inclined water gage connected to the test volume by the rubber tube. This gage is used to measure any variation in gas pressure in the gas line section between the rubber bag seals. A spring gage is used to indicate the air pressure within the sealing bags.
4. Connections are provided to a pressure pump used to inflate the rubber sealing bags and to a suction pump that deflates the bags.
5. A steel rod is used to propel the bag frame and tubing along the inside of the gas main. The rod and bag frame have sufficient flexibility to be passed through a tape on the gas line and yet have stiffness sufficient to avoid buckling when the apparatus is pushed along inside the gas main.

Procedure for Leak Testing of Natural Gas Mains with Rubber Sealing Bags

When testing for leakage in natural gas mains, the sealing bags described previously are inserted into a main pipeline containing gas under moderate distribution pressure. The rubber sealing bags are spaced a set distance apart on the frame. These bags seal off a portion of the main line when they are inflated with a pressure tire pump to 20 to 40 kPa (3 to 6 $lb_f \cdot in.^{-2}$). The gas pressure in the test volume between the two sealing bags is indicated on the inclined water gage on the test control panel as soon as the bags are inflated to form pressure seals. When the main gas distribution line is sealed off completely by both bags, the pressure in the test volume between the bags will remain constant, as in a pressure hold leak test. Loss of pressure would indicate gas leakage through the wall of the distribution pipeline, in the length between the two sealing bags.

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PART 3. Pressure Change Tests for Measuring Leakage in Evacuated Systems

Introduction to Pressure Measurements in Evacuated Systems

By popular usage, atmospheric pressure is taken as the upper limit of vacuum. Any pressure less than standard atmospheric pressure (101 kPa) is some form of vacuum. On Earth, vacuum pressure can be anything between absolute zero pressure and the barometer reading at the particular location and time. Earlier, the vacuum pressure was measured in inch of mercy (in. Hg) or millimeter of mercury (mm Hg) below atmospheric pressure. A vacuum of 28 or 29 in. Hg was considered to be a fairly good vacuum. Now, using SI units, this same vacuum level would be expressed as an absolute pressure of 3 to 6 kPa, which is 3 to 6 percent of normal sea level atmospheric pressure, 101 kPa (1 atm).

Meaning of Absolute Pressure and Gage Pressure in Vacuum Systems

As suggested earlier, the concept of a vacuum is related to the pressure exerted by the earth's atmosphere. Atmospheric pressure indicates the weight of a column of atmospheric air of unit cross sectional area measured at a particular altitude above sea level. With increasing altitude, the pressure decreases until, at some indefinitely great height above the earth's surface (where only empty space exists), the pressure approaches absolute zero. An enclosure is said to be under vacuum if its internal pressure is less than that of the surrounding atmosphere. Because of atmospheric pressure changes due to meteorological factors and altitude, the numerical value assigned to gage pressure in vacuum is referred to atmospheric pressure under standard conditions at sea level (an absolute pressure of 101 kPa). As vacuums were improved, it became necessary to provide a scale of absolute pressures (somewhat analogous to the scale of absolute temperatures). The concept of a perfect vacuum corresponds to the hypothetical state of zero absolute pressure.

International System of Units (SI Units) for Vacuum Pressures

The SI unit for pressure is the pascal (Pa) and is introduced here as the unit of pressure in vacuums. Many processes require medium levels of vacuum of the order of 0.1 to 1 Pa. However, for many applications such as high altitude simulation chambers, pressures much lower than 0.1 Pa are required. Units of millipascal (mPa) or micropascal (μPa) are used to describe pressures in this range of hard vacuum, to avoid negative exponents or powers of ten. The previously used unit of torr (1 torr = 1 mm Hg) must be multiplied by 133 to equal the pressure in pascal. The millitorr is equal to pressure of 133 mPa. Because the pressure of the standard atmosphere at sea level is 1.01×10^5 Pa or 101 kPa, it follows that perfect vacuum would have a (negative) gage pressure of (–) 101 kPa because the gage pressure in vacuum is referred to the standard atmospheric pressure at sea level.

Conversions of Vacuum Pressures from Prior Units to Pascal

The twentieth century has seen many change in the units used to describe pressure levels in vacuums. Early investigators described their vacuum pressure in terms of millimeter of mercury, or torr, where the atmospheric pressure at standard conditions was taken as 760 torr. Hard vacuum pressures were later described in terms of micrometer of mercury (1 μm is one millionth of a meter of mercury). Vacuum pressures are variously expressed in pound per square inch absolute pressure ($lb_f \cdot in.^{-2}$ absolute), inch of mercury, torr and the SI unit pascal. For example, 1 μm Hg = 0.001 torr = 10^{-6} m Hg = 133 mPa = 0.133 Pa. The pressure of the standard atmosphere is then equal to 760 torr. The (negative) gage pressure for a perfect vacuum would then be –760 torr in this system of units. (An absolute pressure of 1 torr is equal to 133 Pa.) The preferred unit is pascal.

Conversion factors relate the various units used to describe pressures in evacuated systems, including the pascal, atmosphere (atm or torr and the

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micrometer of mercury). Figure 22 shows a scale useful for approximate conversions of vacuum pressures between SI units of pascal and earlier units of millimeter of mercury (equal to torr) for several typical ranges of vacuum pressure. These comparisons may also help personnel to convert their data into SI units.

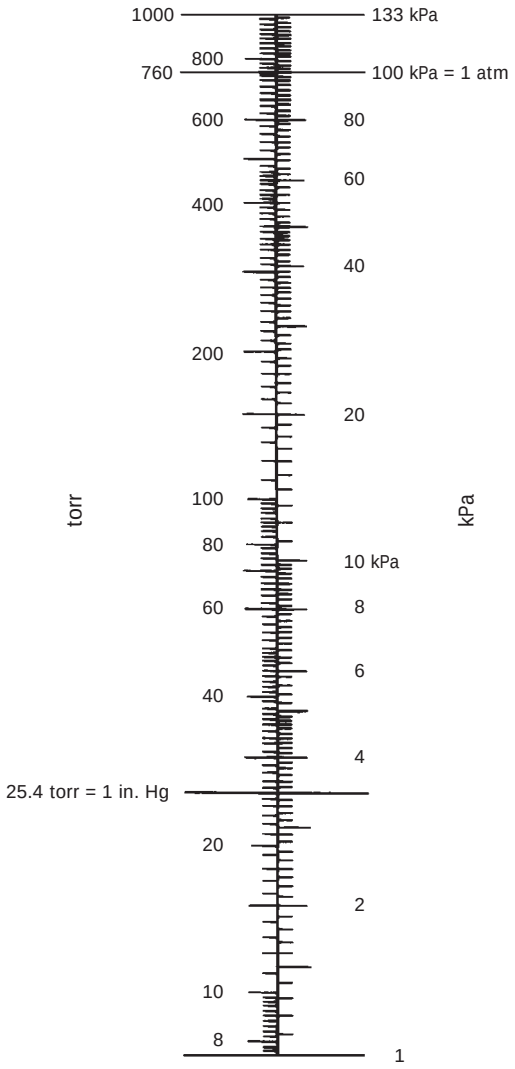
Limitations on Ultimate Vacuum Pressure Caused by Leakage and Outgassing

During evacuation of a container, molecules are constantly being removed by the pumping process. Therefore, it might seem that eventually a pressure of absolute zero would be obtained. This would be true if the only molecules to be removed were those in the gas space. However, other gas sources do exist and must be considered. The predominant gas sources are leakage and outgassing. Leakage is the direct transmission of gas molecules, driven by the higher external pressure, through holes or porosities in the vacuum chamber wall, in welds or in the various seals used in the system. Outgassing refers to all forms of gas coming from the materials in the vacuum system. It includes gases that are adsorbed on the surface, dissolved in the material and occluded in gas pockets, as well as those due to evaporation or decomposition.

The continual addition of gas from these sources represents the major limitation on the ultimate pressure that can be obtained in evacuated systems. Mathematically, the ultimate pressure P_u is given by the influx of gas Q divided by the pumping speed S, so that $P_u = Q/S$. Because the vacuum pump is itself a source of outgassing, it can contribute a limiting component P_p to the ultimate vacuum pressure. Its effect is frequently included in the prior equation for ultimate pressure. In this case, $P_u = Q/S + P_p$, where the term Q now refers to the influx of gases from all sources except the vacuum pump.

Even though the pump may be operating at a particular limit pressure for one type of gas, because of a leak or outgassing, it can still pump other gases to extremely low partial pressure. This is true because, in molecular flow, all types of gases flow independently of each other. Typically, a gas analysis of an ultrahigh vacuum system operating at a total pressure of 10 nPa (~1×10^{-10} torr) will show hydrogen and carbon monoxide as the residual gases still coming from the walls of the vacuum system in this ultrahigh vacuum range. This occurs even when the partial pressure of the original nitrogen and oxygen are too low to be measured.

FIGURE 22. Histogram for conversion of vacuum absolute pressures between prior unit of torr and SI unit of pascal (1 std atm = 100 kPa = 760 torr).



Pumping Requirements for High Vacuum Systems

The ideal gas laws apply to ideal gases even at very low vacuum pressures. They do not apply, however, to condensable vapors such as water vapor or refrigerant gases. The implications of the ideal gas laws become evident when considering the effect of reduced pressure on the volume of a fixed quantity of ideal gas held at constant temperature. A liter of gas at standard atmospheric pressure

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would increase in volume as pressures are lowered in the vacuum region (Table 7).

Tremendous multiplying factors come into existence as the pressure drops in an evacuated system. The pumping speed in cubic meters per second does not increase as pressure is lowered, so much smaller masses of gas (fewer gas molecules) are removed per unit of time, as system pressure drops.

Residual Gas Molecule Densities in High Vacuum Systems

Because the mass reduction factor is so great when evacuating a test system, it might be assumed that after pumping to reach low pressure, there is really nothing in the container to affect any work that may be inserted within it. However, one must consider the number of molecules that remain at various pressures. It may be recalled that there is a physical relationship stating that 22.4 L (0.89 ft^3) of any gas will contain 6.023×10^{23} molecules at 0 °C (32 °F) and 101.325 kPa. The natural constant, 6.023×10^{23}, is known as *Avogadro's number*. If the gas pressure is now reduced to 0.1 Pa or one millionth of its previous value, the 22.4 L (0.89 ft^3) volume of gas still within the container contains 6×10^{17} molecules. Even at 1 μPa, some 6×10^{12} molecules will still remain in the 22.4 L (0.89 ft^3) volume. This provides a residue of 3×10^{11} molecules or almost one trillion molecules per liter of volume (one billion per cubic centimeter). To obtain low ultimate vacuum pressures, one must reduce the various sources of gas within the system being pumped down. Leakage can be eliminated only by first locating each leak and then properly repairing it or by placing an adequate temporary seal over it. Maintaining cleanliness and avoiding introduction of moisture into the test system before the vacuum pumpdown are vital. However, where moisture has contaminated the interior volume of a test system, vacuum pumping can help to remove the moisture, if the total amount of moisture is very small (such as water adsorbed over a small surface area).

TABLE 7. Gas volume variation with pressure.

Gas Pressure		Volume of Gas		
Pa	(atm)	m^3	(ft^3)	Description
10^5	(10^0)	10^{-3}	(3.5×10^{-2})	atmosphere
10^3	(10^{-2})	10^{-1}	(3.5×10^0)	
10^0	(10^{-5})	10^2	(3.5×10^3)	
10^{-3}	(10^{-8})	10^5	(3.5×10^6)	high vacuum
10^{-6}	(10^{-11})	10^8	(3.5×10^9)	very high vacuum
10^{-9}	(10^{-14})	10^{11}	(3.5×10^{12})	

Ensuring Cleanliness of Welded Vessels to Be Evacuated for Leak Testing

In preparation for leak testing by pressure change or helium tests with a tracer probe or hood, the interior of the system under test is evacuated. A sensitive vacuum pressure gage is then used to measure pressure change or a helium mass spectrometer is used to detect helium tracer gas that reaches the vacuum pump input. Joints for high vacuum vessels are far more critical than joints in pressure vessels that also operate under 100 kPa (1 atm) of differential pressure.

Microporosity in the weld, entrapped gases or solids and surface layers that outgas become major problems with high vacuum equipment or equipment that will be evacuated for leak testing. Extremely small defects or inclusions in welded joints may not be detectable with the usual nondestructive testing techniques. The leak testing of the evacuated system may be compromised because of such small leak and gas sources.

For valid leak detection and location by the tracer probe technique or leakage measurement by the hood technique, cleanliness of the test object surfaces and the leak testing system is essential. Tracer gas can accumulate in surface dust and oil or grease, including that within leak passageways, possibly causing small leaks to remain undetected when they are exposed to the tracer probe gas only briefly. Alternatively, evaporation of condensed vapors and gases from such contamination layers may cause a sensitive leak detector system to indicate leakage when the system is actually leaktight. The larger the system under test, the more important it is to ensure cleanliness (including weld crevices and surface discontinuities). The inert gas tungsten arc welding (GTAW) process produces clean welded joints with minimum permeability to atmospheric or tracer gases. The absence of welding flux minimizes post weld cleaning operations and problems of outgassing from slag inclusions.

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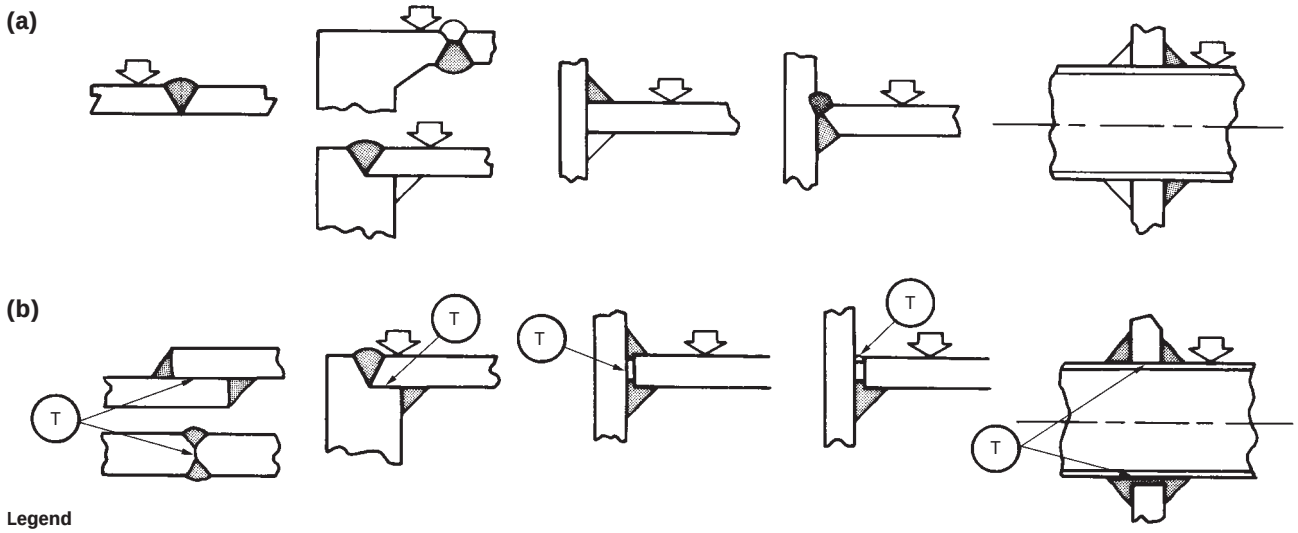
Effects of Weld Joint Design on Leak Testing of Evacuated Vessels

For pressure vessels to be evacuated during leak testing (and vessels designed for vacuum operation), the weld joint design and preparation should avoid trapped volumes or unwelded faying surface areas that will be exposed to the vacuum side of the joint. Both form crevices that may hold foreign matter that can outgas during evacuation or may provide traps for tracer gases. Because cleaning of such crevices is often impossible, joint design and welding procedures must eliminate such traps. Welding should be performed from the side of the joint that will be evacuated whenever practical. The under bead often contains unavoidable microporosity too small to affect most strength and toughness properties of the welded structure. However, if exposed to the vacuum, these voids could act as trapped volumes. Leakage from this source can be avoided by welding the cover (or seal) pass from the side of the pressure boundary that will be evacuated. Figure 23a shows examples of preferred joint designs for systems that will be exposed to high vacuum. Figure 23b shows undesirable joint designs which provide dirt traps and create trapped volumes (at the roots of butt welds made from two sides of the plate, or fillet welds with unwelded areas between abutting plates).

Factors Influencing Speed of Vacuum Pumping of Large Volume Systems

The pumpdown time or time required for evacuation of large vessels and systems from atmospheric pressure is highly dependent on the condition of the vacuum system, the volume to be evacuated and the pumping speed. Any significant amount of water contained in the system will have a powerful effect on the time required for pumpdown because water has a vapor pressure of 2.26 kPa (17 torr) at 20 °C (68 °F). When water is present within the system to be evacuated, the pressure will not drop below this value until the bulk of the water has been pumped out. (Drying by evacuation is often a useful way to remove water trapped or condensed within pressure vessels, piping and components.) Consequently, water or other vaporizing liquids should not be introduced into test systems before leak tests that require evacuation, if it can possibly be avoided. Evacuation rates attained by mechanical pumps drop rapidly as the pressure is reduced by

FIGURE 23. Weld joint designs for welded vessels: (a) preferred designs have no crevices or volume traps open to evacuated side of pressure boundary; (b) undesirable joints trap contamination and tracer gases, which may outgass during evacuation or leak testing with sensitive mass spectrometer or other vacuum leak detectors.



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pumping. Gas evolution by evaporation of liquids at very low pressures increases rapidly and prolongs the pumping period required to attain desired vacuums.

Techniques for Estimating Time Required for Pumpdown to 10 kPa (75 torr)

A technique for approximating the mechanical pumpdown time for very large systems as given by Guthrie in *Vacuum Technology*[7] uses the relation:

$$\textbf{(64)} \quad T = 2.3 \frac{V}{S}$$

where T is approximate pumpdown time (2.3 time constants) to ten percent of initial atmospheric pressure (to about 10 kPa or 75 torr); V is volume of test system to be evacuated from atmospheric pressure (100 kPa or 750 torr); S is pumping speed of evacuation pumps, volume unit per unit of time.

Consistent units must be used for each term in the above equation, such as those in Table 8.

Equation 64 indicates the pumpdown time required to reduce pressure to one tenth of an atmosphere or about 10 kPa. To attain lower vacuum system pressures, much more pumping time is required. The term on the right side of Eq. 64 must be multiplied by the factors in Table 9, for various indicated final pressures within the system being pumped down.

For example, to evacuate the system to a pressure of only 1 Pa (7.5 mtorr), the right side of Eq. 64 is multiplied by a factor of 5, so that the pumpdown time is estimated as:

$$T = 2.3 \times 5 \frac{V}{S} = 11.5 \frac{V}{S}$$

TABLE 8. Consistent units for pumpdown calculation.

Time	Volume	Pumping Speed
Second	Cubic meter	Cubic meter/second
Second	Liter	Liter/second
Minute	Cubic foot	Cubic foot/minute

TABLE 9. Calculation of approximate pumpdown time.

Final Pressure		Multiplying Factor for Equation 64
Pa	(millitorr)	
10	100	4
1	10	5
0.1	1	6

Alternative Technique for Estimating Pumpdown Time to 10 kPa (75 torr)

An alternative approximation technique for estimating pumpdown time of practical industrial systems with prior contamination is also presented by Guthrie.[7] This technique applies for many average industrial systems that may have various sources of gas, vapor and leaks that will require larger pump sizes for any given pumpdown time. For example, gas may be trapped on interior surfaces by mechanisms such as absorption (which refers to binding of gas in the interior of solid or liquid materials) or adsorption (which refers to condensation of gas or vapor on the surface of a solid). Despite efforts to maintain or restore cleanliness to the system, there will be variations from system to system in the rates of outgassing of these trapped gases and vapors, which will change the required pumpdown time to achieve specific vacuum pressures.

This approximation technique makes use of pumping down curves such as would apply typically to clean systems of known interior volume. For typical industrial systems with contamination, leaks or outgassing conditions, the time indicated on the pumpdown curve for clean systems would be multiplied by a service factor that accounts for the effects of nonideal systems. The service factors to be used for average industrial systems are listed in Table 10, in terms of the pressure region to which the system will be pumped down.

For example, suppose that a specific system pumpdown curve shows a pumpdown time of 200 min to pump from 100 Pa (750 mtorr) to a final pressure of 10 Pa (75 mtorr). The service

TABLE 10. Service factors for pressure regions in pumpdown calculations.

Pressure Region		Service Factor
Pa	(torr)	
10^5 to 10^4	(760 to 100)	1.0
10^4 to 10^3	(100 to 10)	1.25
10^3 to 10^2	(10 to 0.5)	1.5
10^2 to 10^1	(0.5 to 0.05)	2.0
10^1 to 10^0	(0.05 to 0.005)	3.0
10^0 to 0	(0.005 to 0.0002)	4.0

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factor corresponding to this final pressure range is 2.0. The estimated pumpdown time for this pressure range is then obtained by multiplying this pumpdown time of 200 min for an ideal system by a factor of 2.0, to obtain an estimated pumpdown time of 400 min in this pressure range for the average industrial system with contamination or leaks.

Comparison of Theoretical and Actual Pumpdown Curves for Welded Steel Tank

The following is an example of a theoretical and an actual pumpdown curve for the annular space of a double wall vacuum insulated liquified natural gas tank. Figure 24 shows typical pump curves relating pumping speed to pressure for a combination of mechanical pumps with booster pumps. For the test to be reported here, the pump unit's performance curve is typical of several shown in Fig. 24. Before the pumpdown tests, the annular space welds were deslagged. The metal surfaces were examined with a near ultraviolet light (used for fluorescent tracer inspection) to detect any deposits of hydrocarbons (which also fluoresce under ultraviolet radiation). All deposits detected were then removed with solvent cleaner to reduce absorption of water vapor or other condensable vapors within the interior of the test volume. The entire interior surfaces were then cleaned with a broom to remove loose dust and dirt to eliminate these particles as surfaces on which vapors might condense and later outgas.

The technique for calculating pumpdown time for very large systems is used to predict the pumpdown time periods Δt between the initial pressure P_1 and the final pressure P_2 in accordance with the approximation Eq. 65 and 66:[8]

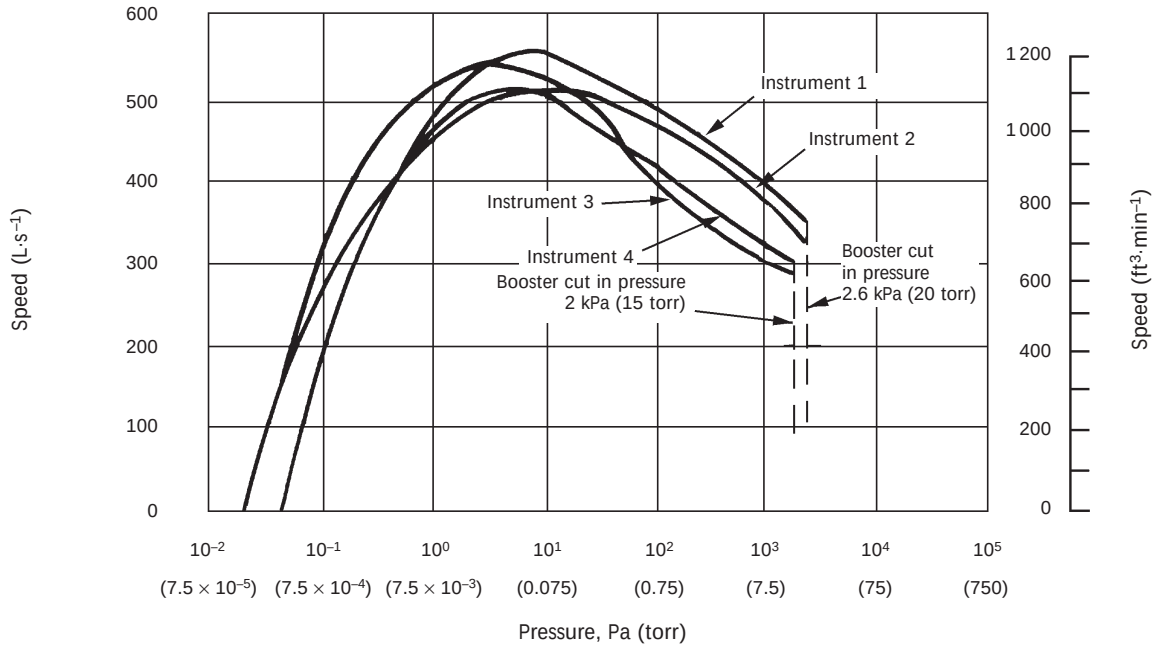
$$\textbf{(65)} \quad \Delta t = K \frac{V}{S} \ln \frac{P_1}{P_2}$$

$$\textbf{(66)} \quad \Delta t = 2.3\, K \frac{V}{S} \log_{10} \frac{P_1}{P_2} = K' \frac{V}{S}$$

In Eq. 66, $K' = 2.3\, K[\log_{10} (P_1/P_2)]$; Δt is the pumpdown time between the initial pressure P_1 and the final pressure P_2. Reasonable values for the K and for the K' factors are given in Table 11.

K values cannot be added. However, for calculating the pumpdown time Δt for a pressure range that spans two or more of the pressure ranges listed above, the K' value to be used is equal to the sum of the K' values given for the two or more ranges covering the pressure difference for which Δt is to be calculated. For example, if the

FIGURE 24. Curves relating pumping speed to pressure in vacuum chamber for various mechanical pumps with booster pump units.



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pump speed is fairly constant from atmospheric pressure (101 kPa) to a pressure of 100 Pa (1 torr), an estimated Δt for the pressure range could be determined in one calculation using a K' value of 7.3 or (4.0 + 3.3).

The values of K and K' for computing pumpdown times as listed above apply only for the case of clean mild steel tanks. At pressures below 0.1 Pa (0.001 torr), the pumpdown times are primarily determined by outgassing conditions and the relationships of Eqs. 65 and 66 are no longer valid.

Vacuum Pumpdown Technique for Leakage Measurements

The evacuation pumpdown technique of leak testing involves the determination and evaluation of a pressure time response curve for a vacuum test chamber within which the test object is placed for testing. Leakage measurement can be performed in either of two ways.

1. Determining leakage rate at equilibrium pressure attained during pumpdown. The vacuum test chamber is pumped down to equilibrium pressure. Test object leakage and outgassing from the test chamber are measured and then subtracted from the value of outgassing measured in a leakfree system.
2. Deriving an allowable pressure time curve for the pumpdown of a system under test. Systems deviating from this relationship are considered to be leakers.

With either type of test system, it is possible to set up an automated leak test station involving a carousel system. The carousel moves the test samples into position, pumps them down and measures the resultant pressures. The biggest difficulty with this type of leak test is the false reading produced by outgassing of dirty samples.

TABLE 11. Values of pumpdown time estimation factors *K* and *K'*.

Pressure Range			
Pa	(torr)	*K*	*K'*
101 000 to 2600	(760 to 20)	1.1	4.0
2600 to 133	(20 to 1.0)	1.1	3.3
133 to 13.3	(1 to 0.1)	1.5	3.45
13.3 to 6.6	(0.1 to 0.05)	4.0	2.77
6.6 to 1.3	(0.05 to 0.01)	4.0	6.44
1.3 to 0.13	(0.01 to 0.001)	4.0	9.21

Equations Used in Analysis of Vacuum Pumpdown Leak Tests

The fundamental response curve for a vacuum system during pumpdown is described by Eq. 67:

$$\frac{dP}{dt} = \frac{Q}{V} - S\frac{P}{V} \quad (67)$$

where P is pressure in system being evacuated; t is time elapsed from start of pumping; S is effective pumping speed of vacuum pump; V is volume of system being evacuated; Q is total in-leakage rate plus outgassing load of test system; dP/dt is time rate of change of pressure. The gas load may be due to leakage, evolution of gas from the walls of the evacuated system or both.

In the lower vacuum pressure range where outgassing has significant effect, integration of Eq. 67 leads to the pumpdown response characteristic of Eq. 68:

$$t_2 - t_1 = -\frac{V}{S}\ln\frac{1 - P_2\frac{S}{Q}}{1 - P_1\frac{S}{Q}} \quad (68)$$

Equation 68 describes an exponential decay curve with a time constant equal to S/V, which becomes asymptotic to an equilibrium pressure defined by Eq. 69:

$$P = \frac{Q}{S} \quad (69)$$

This ultimate pressure is approached in approximately five time constants, when $t_2 - t_1 = 5(S/V)$.

Procedure for Pressure Rise (Vacuum Retention) Test for Leakage Rate

The pressure rise test (also called a vacuum retention test) is a pressure change leakage measurement technique performed on a system evacuated below atmospheric pressure. It can be performed on systems at any vacuum level but is most effective on systems evacuated to an absolute pressure (vacuum) in the range from 10 to 0.001 Pa (100 to 0.01 mtorr). This leakage rate test is performed by isolating the system under test after it has been evacuated to the required (or specified) absolute pressure (vacuum). Then the pressure and, when exposed to ambient weather conditions, the surface temperature of the system are observed for a specific time to determine the rate of pressure rise per unit of time for the system. Figure 25 shows schematically the test arrangement and the connections

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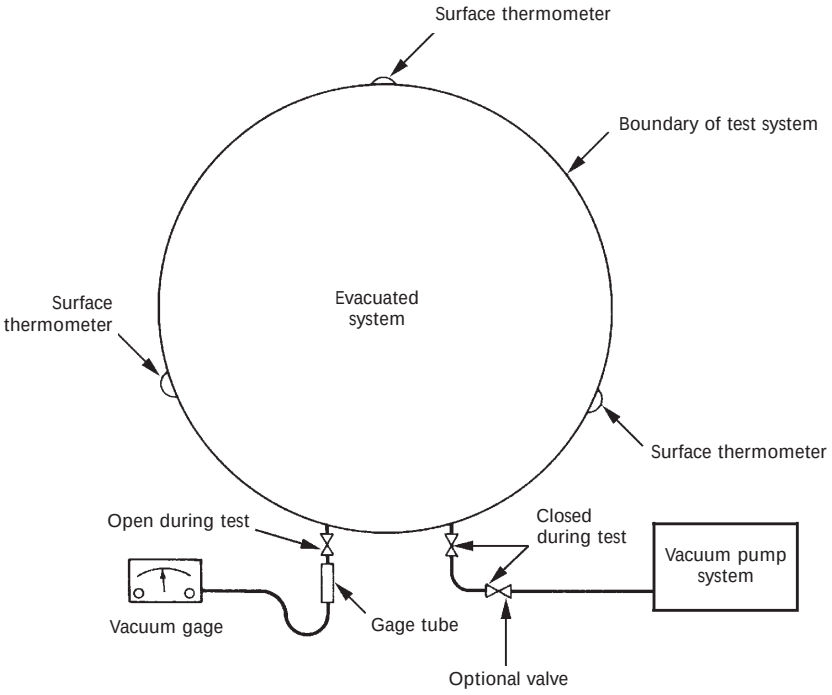
FIGURE 25. Arrangement of equipment for pressure rise leakage rate testing of an evacuated system. Also known as a vacuum retention test, this test measures overall leakage rates and requires use of a vacuum pumping system and a vacuum gage. For systems exposed to ambient weather conditions, surface temperature detectors are used to approximate internal air temperatures in the system. Ambient temperature must be measured in shade, not in direct sunlight.



between the test volume, the vacuum pump system and the instrumentation.

Effects of Condensable Vapors on Vacuum Retention Leakage Test

As noted earlier in this chapter, the behavior of vapors in an evacuated system deviates significantly from the General ideal gas law:

$$PV = nRT \quad (70)$$

A vapor is the gaseous form of any substance that usually exists in the form of a liquid or solid, such as water vapor. A pure liquid in equilibrium with its own vapor will have two phases (liquid and vapor) that coexist at a specific partial pressure known as the vapor pressure.

Because condensation or evaporation occurs with changes in temperature, vapor molecules enter or leave the gaseous phase with any change in temperature. This changes the number of molecules of a particular vapor and the partial pressure which that vapor exerts in a particular gas volume. These vapor effects, called *outgassing* in a vacuum system, are not included in the effects described by the General ideal gas law of Eq. 70. For this reason, in an evacuated system, it is not mathematically realistic to make accurate temperature corrections to the final pressure for pressure data taken at different temperatures.

Therefore, to establish a fairly accurate leakage rate by this pressure change technique for an evacuated system exposed to ambient weather conditions, it is necessary to compare pressure data at periods when the temperature is the same or nearly the same and the temperature trends are in the same direction. For a system enclosed in a temperature controlled building, such as a vacuum chamber evacuated to lower absolute pressure ranges, temperature measurements are usually not necessary. A pressure rise test of such an enclosed system can be used to determine both the leakage rate and the outgassing rate for that system.

Advantages of Pressure Rise (Vacuum Retention) Leakage Test Technique

The pressure rise leakage rate test is relatively simple in principle and fairly

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easy to perform on smaller test systems. The test is capable of attaining increased leakage sensitivity as the system size or volume decreases. That is, the total leakage rate that can be measured as a pressure rise per unit time becomes smaller as the system under test gets smaller in volume. This test technique can serve as a final test or as a preliminary test preceding other leak test techniques, depending on the size and configuration of the system to be leak tested. This quantitative leakage rate test can be used to determine the total leakage rate (in the form of a pressure rise per unit of time) through the test boundary of any system capable of being evacuated.

Limitations of Pressure Rise (Vacuum Retention) Leakage Test Technique

The sensitivity of the pressure rise leakage rate test diminishes as the size or volume of the system to be tested increases. Larger rates of leakage must exist if they are to be detected in large volume systems by this test technique. In addition, the location of unacceptable leakage cannot be determined by this test alone. If the actual total leakage rate exceeds the allowable value, another leak test technique must be used to locate any unacceptable leaks or the numerous small leaks that might contribute to an unacceptable high overall rate of leakage. Thus, performance of a pressure rise test on the evacuated annular space of a double wall vessel, with a resultant total leakage rate indication in excess of that allowable, will not reveal whether the unacceptable leakage is in the inner vessel, in the outer vessel or in a combination of both.

Because of the effect of vapors that do not obey the general gas laws for ideal gases, it becomes difficult to determine an accurate true gas pressure rise per unit of time for very large volume systems exposed to wide temperature variations during the leakage test period. Lowering the absolute pressure within the evacuated vessel in an effort to increase the leakage rate test sensitivity may be unfeasible because of the vacuum pumping system limitations. Alternatively, the rate at which gas can be pumped out may be limited by the size of the hole (penetration) through which it must be removed. Trying to increase the test sensitivity by increasing the duration of the test, in an effort to achieve the ability to read a smaller pressure rise per unit time more reliably, may prove unrealistic as costs increase and schedule completion is made more difficult.

Factors Affecting Leakage Sensitivity of Pressure Rise Test Technique

The leakage rate sensitivity of the pressure rise (or vacuum retention) leakage rate test is influenced by five major factors:

1. absolute pressure attained in the evacuated system, when the test is performed (this, in turn, affects the resolution of the smallest measurable pressure change);
2. internal volume of the system to be tested;
3. time duration of the leakage rate test;
4. ambient temperature and weather conditions; and
5. internal surface areas and cleanliness of the test system.

Each of these factors is discussed next, in greater detail.

Effect of Absolute Pressure in Evacuated System Being Tested

When vacuum retention leakage rate tests are performed within the absolute pressure range of 10 to 0.001 Pa (100 to 0.01 mtorr) on large systems, the lower the pressure, the greater the test sensitivity becomes. The limitation on the high pressure end of this range results from inability to measure very small pressure changes resulting from leakage from large volumes.

For example, it might be necessary to detect changes of a fraction of a pascal at 2.5 kPa (a few micrometers at 20 torr). The limitation on the low pressure side is the increase in the portion of the pressure change attributable to outgassing. At these very low absolute pressures, the pressure rise due to actual leakage is small in relation to the pressure rise due to outgassing. This makes it difficult to determine the true rate of pressure rise caused by real leakage.

Effect of Volume of Tested System

The test sensitivity and, in turn, the rate of pressure rise both vary inversely with the size or volume of the evacuated system being tested. For example, a leakage rate of 5×10^{-3} Pa·m^3·s^{-1} (5×10^{-2} std cm^3·s^{-1}) in a 570 m^3 (2×10^5 ft^3) system would cause a rate of pressure rise of only 0.8 Pa (5.8 mtorr) per day. This same rate of leakage in a 0.3 m^3 (10 ft^3) system would cause a rate of pressure rise of 1.5 kPa (11.6 torr) per day.

Effect of Duration of Leakage Test

The sensitivity of the leakage rate test increases directly with the elapsed time during the test. As the time duration of

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the test increases, the test sensitivity increases. The three factors of absolute pressure P, system volume V and time duration t of the pressure rise test are related by Eq. 71 and 72:

$$\textbf{(71)} \quad Q = \left(P_2 - P_1\right) \frac{V}{t}$$

where Q is leakage rate ($Pa \cdot m^3 \cdot s^{-1}$); P_1 is initial absolute pressure (torr); P_2 is final pressure (pascal); V is volume of evacuated system under test (cubic meter); t is time duration of test (second).

$$\textbf{(72)} \quad Q = \left(P_2 - P_1\right) \frac{V}{96.6\,t}$$

where Q is total leakage rate (std $cm^3 \cdot s^{-1}$); P_1 is initial absolute pressure (torr); P_2 is final absolute pressure (torr); V is volume of evacuated system (cubic foot); t is time duration of test (hour). For other systems of units, the conversion factor of 96.6 will change.

Effects of Weather and Ambient Temperature Conditions

In pressure rise (vacuum retention) tests of evacuated systems, the greater the exposure of the system to direct sunlight and the greater the variations in ambient temperature, the more difficult it becomes to determine an accurate pressure rise. Temperature variations lead to uncontrollable effects on the rate of outgassing or condensation of vapors within the system, which also influence the pressure variations in the system.

Effects of Internal Surface Area and Cleanliness of Test System

With evacuated systems under pressure rise leak testing conditions, the smaller the internal surface area and the cleaner that surface is, the less the outgassing in the systems. This reduces the effect on pressure change from outgassing due to temperature variations.

Estimating Leakage Test Sensitivity Attainable in Pressure Rise Tests

To determine the leakage rate sensitivity attainable with a pressure rise (vacuum retention) test, it is necessary to know in advance the volume (estimated or calculated) of the system and the absolute pressure at which the test must be performed. If the allowable pressure rise per unit of time is known or specified and it is realistic for the absolute pressure (vacuum) level at which the test is to be performed, the test sensitivity or detectable total leakage rate can be computed by Eq. 71. If instead the test sensitivity or total leakage rate Q is specified or known because of system performance requirements, the allowable pressure rise per unit of time for that total leakage rate can be determined by using the transposed form of Eq. 71 shown below as Eq. 73:

$$\textbf{(73)} \quad \frac{P_2 - P_1}{t} = \frac{Q}{V}$$

or in $torr \cdot h^{-1}$:

$$\textbf{(74)} \quad \frac{P_2 - P_1}{t} = 96.6\,\frac{Q}{V}$$

Units for variables in Eqs. 73 and 74 are given below Eqs. 71 and 72, respectively.

If the rate of pressure rise computed by Eqs. 73 and 74 is measurable with available test equipment at the specified test pressure, the required or specified leakage rate test sensitivity can be achieved. If it is not measurable, then an attainable test sensitivity must be established by Eqs. 71 and 72.

Example Computation to Determine Pressure Rise Test Feasibility

As an example of the application of Eq. 74, suppose that the performance specification for a system requires that the completed system contain no leakage in excess of 2×10^{-3} $Pa \cdot m^3 \cdot s^{-1}$ (2×10^{-2} std $cm^3 \cdot s^{-1}$). This 300 m^3 (10^5 ft^3) system can be evacuated to an absolute pressure of 1 Pa (or about 10 mtorr) with the permanent vacuum pump system. Would a pressure rise test be a realistic test technique for quantitatively verifying that this system meets the specification requirements? Because Q and V are known, Eq. 73 can be solved as follows in SI units: $(P_2 - P_1)/t = Q/V = (2 \times 10^{-3})/300 = 7 \times 10^{-6}$ $Pa \cdot s^{-1}$ or 0.6 Pa per day. For mixed units, Eq. 74 indicates that:

$$\begin{aligned} \frac{P_2 - P_1}{t} &= 96.6\,\frac{Q}{V} \\ &= 96.6\,\frac{2 \times 10^{-2}}{10^4} \\ &= 1.93 \times 10^{-2}\ torr \cdot h^{-1} \end{aligned}$$

These requirements can be met by the pressure rise (vacuum retention) leakage rate test technique. The time required for the test depends on the surrounding temperature conditions. If the system is in a building in a controlled temperature environment, a test duration of only a few hours should be adequate. If the system is exposed to the weather, then a

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comparable temperature cycle must be experienced. If the weather is cloudy and the temperature is stable, a few hours may be adequate. Normally, for an exposed system, a temperature cycle of 12, 24 or 36 h is necessary to achieve the necessary reliable comparison data.

Example of Pressure Rise Leakage Rate Test of Liquid Hydrogen Vessel

The following example illustrates test conditions and test results for the leakage rate of the annular inner space between concentric inner and outer spheres of a double wall vacuum insulated liquid hydrogen vessel. The outer sphere has a 15.81 m (51 ft, 10.5 in.) inside diameter and the inner sphere has an inside diameter of 13.9 m (45 ft, 7 in.) and a wall thickness of about 19 mm (0.75 in.). The volume of this annular space was calculated to be about 650 m^3 (2.3×10^4 ft^3). Critical areas of the inner sphere were tested by the more sensitive helium tracer probe or hood leak testing techniques.

The specification for the pressure rise (vacuum retention) leakage rate test required that the test be conducted over a period of 72 h. The allowable pressure rise was 3.3 Pa (25 mtorr) in 72 h. For this time span, this was a reasonable leakage allowance. For the 650 m^3 (2.3×10^4 ft^3) annulus volume and allowable pressure rise rate of 3.3 Pa (25 mtorr), the total leakage rate allowable was computed in SI units as:

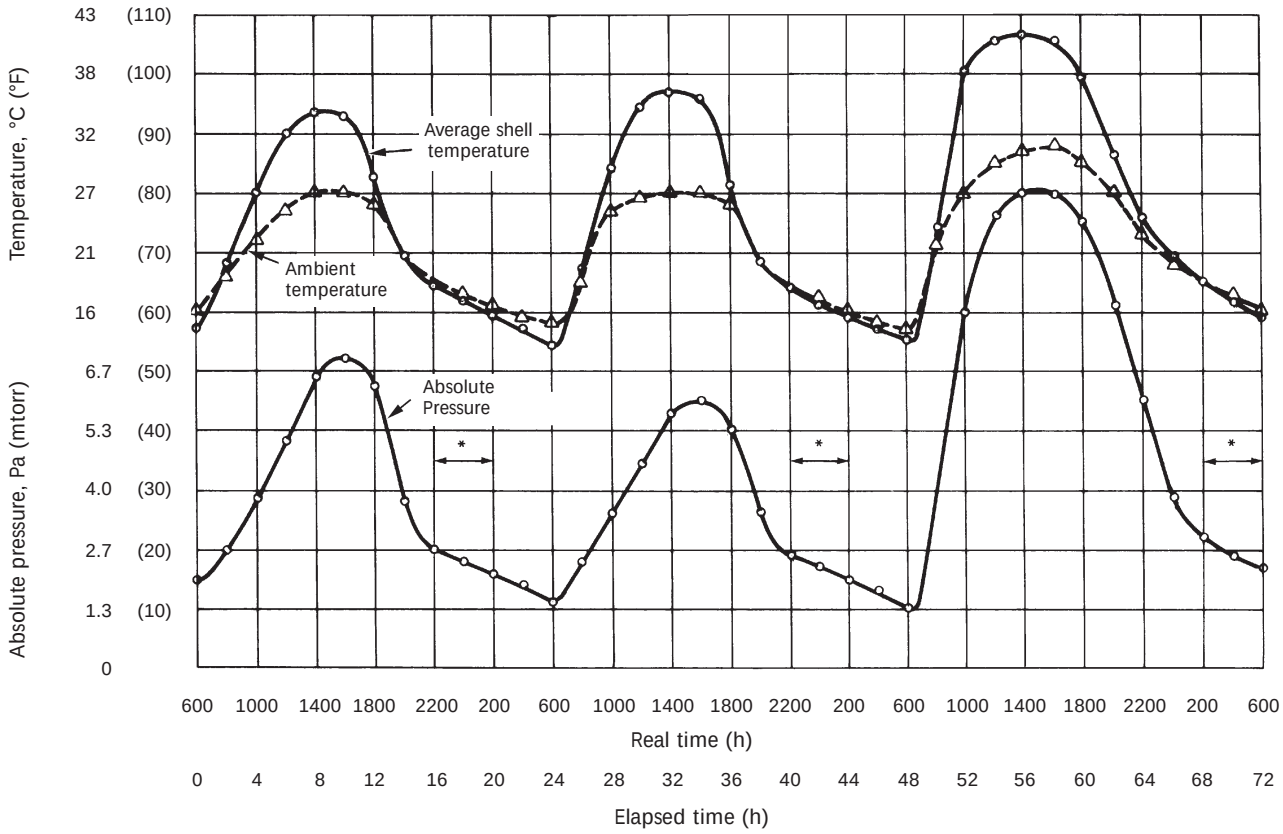
$$Q = \frac{3.3 \times 650}{72 \times 3600}$$

$$= 8.3 \times 10^{-3} \ \text{Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$$

$$= 8.3 \times 10^{-2} \ \text{std cm}^3 \cdot \text{s}^{-1}$$

The results of the pressure rise test performed on the annular space of this double wall liquid hydrogen sphere are shown in the pressure rise test data of Table 12 and are plotted in the graphs of pressure and temperature as a function of time during testing in Fig. 26. Pressure levels may be compared at any of the nearly equivalent temperature points during the night time periods marked

FIGURE 26. Graphs showing variations in temperature and absolute pressure of liquid hydrogen sphere annular space during 72 h pressure rise leakage rate test. Arrows with asterisks indicate time periods when temperatures and trends in change of temperature were comparable. Pressure rise test liquid hydrogen sphere with 13.9 m (45 ft, 7 in.) inside diameter inner tank and 15.8 m (51 ft, 10.5 in.) inside diameter outer tank.



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with brackets and footnotes on the elapsed time column of Table 12. These time points are also marked by arrows and asterisks on the graphs of Fig. 26. The results for this 52 h time span indicate that the pressure rise on this system could have been a maximum of 250 to 400 mPa (2 to 3 mtorr) in 72 h. This was an acceptable leakage test rate because it was much less than the allowable rate of 3 kPa (25 torr) in 72 h. The total leakage rate is equivalent to 6.6×10^{-4} to 9.9×10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (6.6×10^{-3} to 9.9×10^{-3} std $cm^3 \cdot s^{-1}$). Because this loss of 250 to 400 mPa (2 to 3 mtorr) is less than the error in reading of the Pirani or thermocouple vacuum pressure gage used for the test, the pressure rise was probably much less. A longer test period could have proved this but would have served no useful purpose.

Example of Pressure Rise Leakage Rate Test of Laboratory Vacuum Chamber

The following example illustrates test conditions and test results for a pressure rise test of a stainless steel solvent cleaned vacuum chamber. The purpose of the test was to determine the leakage rate and the outgassing rate of the chamber. The chamber had an inside diameter of 590 mm (23.25 in.) and a length of 1.6 m (63 in.). Its volume was calculated to be about 0.487 m^3 or 487 L (17.2 ft^3). Its inside surface area was calculated to be about 4.63 m^2 (49.8 ft^2). The results of the pressure rise test with the chamber vacuum conditioned for approximately 189 h are given in Table 13 and shown graphically in Fig. 27. Figure 27b is an enlargement of the upper linear portion of the graph of Fig. 27a, from whose slope the final leakage rate was determined.

TABLE 12. Test data for pressure rise test of liquid hydrogen sphere.

Real Time	Elapsed Time (h)	Shell Temperature, °F[a]				Ambient Temperature °F	Annulus Press		Information and Comments
		No. 1	No. 2	No. 3	Average		Pa	(millitorr)	
0600	0	59	56	57	57.3	60	2.0	(15)	Begin hold test
0800	2	65	77	62	68.0	66	2.7	(20)	
1000	4	72	100	69	80.3	72	3.9	(29)	Windy, clear and sunny
1200	6	78	112	81	90.3	77	5.1	(38)	
1400	8	81	100	100	93.7	80	6.6	(49)	
1600	10	83	97	97	93.0	80	6.9	(52)	
1800	12	79	87	82	82.7	78	6.3	(47)	
2000	14	70	69	69	69.3	69	3.7	(28)	
2200	16[b]	65	65	63	64.3	65	2.7	(20)	
2400	18[b]	63	62	61	62.0	63	2.4	(18)	
0200	20[b]	61	59	59	59.7	61	2.1	(16)	Clear and calm
0400	22	58	57	56	57.0	59	1.9	(14)	
0600	24	56	53	54	54.3	57	1.5	(11)	
0800	26	63	81	57	67.0	65	2.4	(18)	Clear, calm and sunny
1000	28	76	108	69	84.3	77	3.5	(26)	
1200	30	80	120	85	95.0	79	4.5	(34)	
1400	32	82	113	96	97.0	80	5.7	(43)	
1600	34	84	106	98	96.0	80	6.0	(45)	
1800	36	78	86	80	81.3	78	5.3	(40)	
2000	38	67	70	68	68.3	68	3.5	(26)	
2200	40[b]	63	65	63	63.7	64	2.5	(19)	
2400	42[b]	61	62	61	61.3	62	2.3	(17)	
0200	44[b]	59	60	58	59.0	60	2.1	(16)	Clear and calm
0400	46	57	58	56	57.0	58	1.7	(13)	
0600	48	56	55	54	55.0	57	1.3	(10)	
0800	50	65	87	70	74.0	71	4.0	(30)	Clear, calm and sunny
1000	52	82	120	100	100.7	80	8.0	(60)	
1200	54	85	129	103	105.7	85	10.1	(76)	
1400	56	90	124	106	106.7	87	10.7	(80)	
1600	58	93	120	104	105.7	88	10.7	(80)	
1800	60	87	110	97	98.0	85	10.0	(75)	
2000	62	80	93	86	86.3	80	8.1	(61)	
2200	64	73	78	75	75.3	73	6.0	(45)	
2400	66	70	72	66	69.3	68	3.9	(29)	
0200	68[b]	65	68	62	65.0	65	2.9	(22)	
0400	70[b]	62	63	61	62.0	63	2.5	(19)	
0600	72[b]	59	59	59	59.0	60	2.3	(17)	End of 72 h hold test

a. (°F – 32)/1.8 = °C.
b. Data comparison points.

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The total rate of pressure rise due to both outgassing and leakage during the entire 41 h, 44 min (≅ 2500 min = 150 000 s) test period was computed as:

$$\text{Total } Q = \frac{\left(1.64 \times 10^{-2}\right)\left(1.72 \times 10^{1}\right)}{96.6 \times 41.73}$$

$$= 7.0 \times 10^{-6} \ \text{Pa} \cdot \text{m}^3 \cdot s^{-1}$$

$$= 7.0 \times 10^{-5} \ \text{std cm}^3 \cdot s^{-1}$$

Based on the straight line portion of the last 23.5 h of the test as shown in the graph of Fig. 27b, the leakage rate Q for the chamber was computed as:

FIGURE 27. Pressure rise leakage rate test of a type 487-L stainless steel vacuum chamber: (a) pressure rise as a function of time; (b) enlargement of upper portion of curve, showing rate of pressure rise due to leakage, following outgassing of steel vacuum chamber. See Table 13.

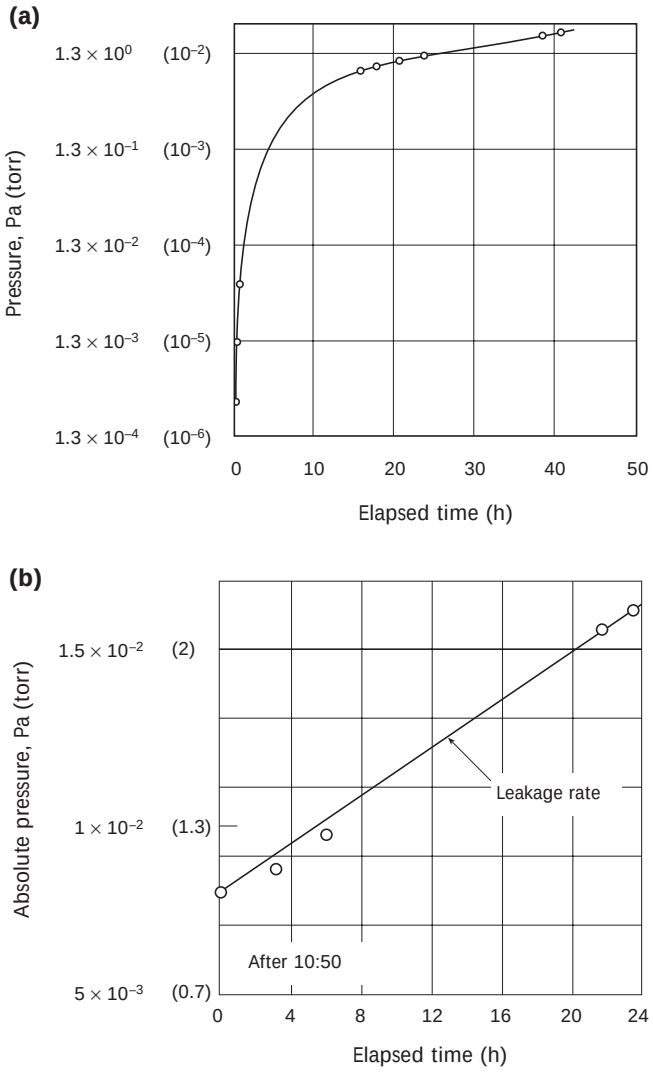

TABLE 13. Pressure rise leakage rate test of type 487 L stainless steel vacuum chamber. See Fig. 27.

Real Time (h : min)	Elapsed Time (h)	Chamber Pressure	
		Pa	(torr)
16:36	0	2.9×10^{-4}	(2.2×10^{-6})
16:38	0.033	7.2×10^{-4}	(5.4×10^{-6})
16:39	0.05	9.5×10^{-4}	(7.1×10^{-6})
16:40	0.067	1.2×10^{-3}	(8.7×10^{-6})
16:41	0.083	1.3×10^{-3}	(1.0×10^{-5})
16:44	0.133	1.9×10^{-3}	(1.4×10^{-5})
16:47	0.183	2.7×10^{-3}	(2.0×10^{-5})
16:53	0.283	4.1×10^{-3}	(3.1×10^{-5})
16:57	0.35	4.9×10^{-3}	(3.7×10^{-5})
08:50	16.23	1.0	(7.2×10^{-3})
10:50	18.23	1.1	(8.0×10^{-3})
14:00	21.40	1.2	(8.7×10^{-3})
16:50	24.23	1.3	(9.7×10^{-3})
08:30	39.90	2.1	(1.58×10^{-2})
10:20	41.73	2.2	(1.64×10^{-2})

$$Q = \frac{\left(8.4 \times 10^{-3}\right)\left(1.72 \times 10^{1}\right)}{96.6 \times 23.5}$$

$$= 6.4 \times 10^{-6} \ \text{Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$$

$$= 6.4 \times 10^{-5} \ \text{std cm}^3 \cdot \text{s}^{-1}$$

Subtracting the leakage rate from the total rate results in the outgassing rate computed as:

$$Q = 6.0 \times 10^{-7} \ \text{Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$$

$$= 6.0 \times 10^{-6} \ \text{std cm}^3 \cdot \text{s}^{-1}$$

$$= 4.6 \times 10^{-6} \ \text{torr-L} \cdot \text{s}^{-1}$$

This results in outgassing computed in torr-L·s^{-1}·cm^{-3} as:

$$\text{Outgassing} = \frac{4.6 \times 10^{-6}}{4.63 \times 10^{4}}$$

$$= 1.0 \times 10^{-10}$$

per square meter of surface area. This agrees very closely with published outgassing data for degreased stainless steel with 200 h vacuum conditioning.

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PART 4. Flow Rate Tests for Measuring Leakage Rates in Systems near Atmospheric Pressure

Principles of Leakage Testing by Measurement of Flow Rates

The flow measurement procedure for leakage testing consists of determining the extent of leakage by measuring the rate of flow of gas moving into or out of the system or component under test. Flow rates can be measured with a flow meter or by means of pumping at known volumetric pumping rates to maintain a fixed system pressure or to compare rates of change of pressure. The flow measurement leakage test procedure can be roughly separated into two broad classes of technique: (1) observation and measurement of gas flow rates or volume of gas displaced and (2) analysis of effects of pumping gas during pressurization or evacuation of systems, on pressure or rates of change of pressure. When leak testing by the flow observation technique, the amount of leakage is measured. The system under test is pressurized or evacuated and placed within a sealed enclosure. The enclosure volume is connected through a flow meter to a regulated pressure source. The gas transfer by leakage between the system under test and its enclosure causes a pressure difference between the enclosure volume and the regulated pressure source. The gas transfer between the sealed enclosure and the reference pressure source is measured by flow meters, by movement of a liquid (slug) indicator in a capillary tube in which the leaking gas is accumulated or by other techniques. In some cases, the reference pressure may be atmospheric pressure. Figure 28 shows a leakage testing system using a fluid slug indicator of the amount of gas leakage.

Pumping Technique for Measuring Leakage Rate from Evacuated Test Systems

In the pumping technique of leakage testing of evacuated systems, the system under test is evacuated by a vacuum pump. The rate of system pressure decrease during pumpdown is then compared with the rate of pressure decrease during pumpdown of a leaktight system.

In an alternative leak testing procedure, the sealed enclosure can be evacuated and allowed to reach pressure equilibrium with its vacuum pumps. The rate at which gas is being pumped to maintain this equilibrium is then measured to determine the rate of leakage from the test volume into the enclosure.

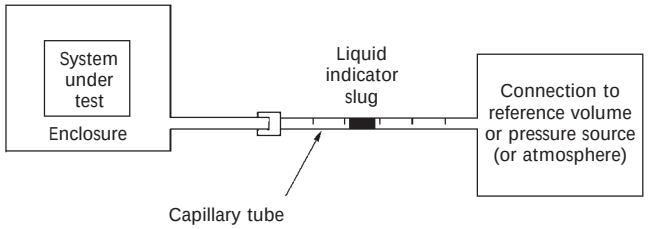
Pumping Technique for Measuring Leakage Rate from Pressurized Systems

In an alternative pumping technique for measuring leakage rates, the test volume can be pressurized and the compressor is then operated only sufficiently to keep the test system pressure constant. The leakage rate can then be calculated from the volumetric pumping speed ($m^3 \cdot s^{-1}$) and the length of time the compressor must operate to regain a predetermined system pressure.

Sensitivity of Flow Measurement Leak Testing Techniques

The sensitivity of leakage rate testing by flow measurements is relatively low, compared to the sensitivity of many other leak testing techniques described in this volume. In most cases, the leakage sensitivity depends on that of the

FIGURE 28. Arrangement for leakage rate testing of system enclosed in a sealed test enclosure connected to a capillary tube flow meter with an opaque visible liquid indicator slug. Leakage from pressurized system into enclosure would cause indicator slug to move to the right by a displacement proportional to the volume of gas leakage.



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instrument used to measure the flow rate and is relatively independent of the test system volume. In a flow observation technique, leakage rates between 10^{-3} and 10^{-5} Pa·m^3·s^{-1} (10^{-2} and 10^{-4} std cm^3·s^{-1}) can be detected, depending on the flow instrument used. If a sealed system is being evacuated, flow rates of the order of 0.1 Pa·m^3·s^{-1} (1 std cm^3·s^{-1}) may be observed. (Note that 1 Pa·m^3·s^{-1} is equivalent to 10 std cm^3·s^{-1}.)

The leakage sensitivity attainable with the pumping pressure analysis technique depends on the size (pumping speed) of the pumps. With evacuated test objects or test systems, leakage sensitivity depends critically on the outgassing within the system being measured.

Advantages and Limitations of Leak Testing by Flow Measurements

Flow measurement leak testing procedures are applicable to a large variety of test systems. The procedures are useful only for measurement of leakage. They are not appropriate for locating leaks. They are used to measure total leakage rates in small sealed parts. They can be used to measure total leakage rates in large sealed systems and in systems that can be pressurized or evacuated. The major advantages of leak testing by means of flow measurements are as follows.

1. No special tracer gas is necessary. the flow measurement leak testing procedure is applicable to whatever fluid is present within the system to be tested. The test system need not be placed in any special environment for leak testing. Instead, systems may be tested in their normal operating modes.
2. The cost of the equipment for flow measurement leak testing is low.
3. The sensitivity of overall leakage measurement is independent of system volume.
4. The leakage rate can be measured without extensive calibration. However, the accuracy of leakage measurement is not very high, as compared with that for many other techniques.
5. When calibration is required, it can be readily attained with standard flow or volume measurement equipment.

There are two major disadvantages of flow measurement leak testing.

1. The test sensitivity is low.
2. Flow measurement procedures have not gained wide recognition.

Flow measurement uses various types of equipment with little similarity and different techniques are used to solve individual leak testing problems.

Sealed Volume Technique of Leak Testing by Flow Measurements

Figure 28 shows the arrangement of leak testing equipment using the most common technique of flow measurement by observation of the movement of fluid in a glass capillary tube. The system under test is enclosed and sealed within the test enclosure. The system being tested can be either evacuated or pressurized. It can either be sealed or connected to a source of pressure or of vacuum.

Care must be taken to ensure that the leakage being measured is not occurring in the connection to the source of pressure or vacuum. The capillary containing the indicating fluid is attached to the test enclosure. This type of testing can be performed with the capillary fluid indicator connected between the test enclosure and a standard testing volume on the other end of the capillary. In this way, the leak test can be compensated for temperature variations, if both test enclosure and the comparison volume are subject to the same temperature conditions. Alternatively, the capillary can be connected between the test enclosure and the atmosphere. For accurate leakage measurements and rapid response, the enclosure containing the system under test should have a net volume as small as practical.

One advantage in the construction of the sealed volume type of leak testing equipment shown in Fig. 28 is that there are no critical, leaktight connections within the enclosure. This is because the system is operating at atmospheric pressure. Therefore, although it is possible that a leak could exist between the enclosure and the atmosphere, leakage does not occur through this leak because no pressure differential is applied across it. Any differences in pressure are compensated for by the pressure transmission through the liquid slug within the interconnecting capillary tube.

Measuring Leakage Rates with Glass Capillary (Pipette) Tubes

Glass capillary tubes containing a slug of indicating fluid provide a means for direct quantitative measurement of leakage rates if a record is made of the time required for the small liquid plug to move a given distance. Because the cross sectional area of the capillary bore is known, the volume swept out by the liquid plug during the measured time interval can be

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computed. A 1.5 mm (0.06 in.) diameter glass capillary tube is used to measure leakage rates in the range from 10^{-3} to 10^{-1} Pa·m^3·s^{-1} (10^{-2} to 10^{0} std cm^3·s^{-1}). A 0.5 mm (0.02 in.) glass capillary tube can be used to measure smaller leakage rates from 10^{-5} to 10^{-3} Pa·m^3·s^{-1} (10^{-4} to 10^{-2} std cm^3·s^{-1}). These capillary tubes are marked with scales given in convenient units for computing leakage rates. A stopwatch is commonly used for timing the movements of the liquid plug within the capillary tube. Pipettes used for liquid measurements provide convenient calibrated capillary tubes.

The upper limit on leakage rates measurable with capillary tubes is reached when the liquid plug moves so fast that timing is difficult. The lower limit on leakage rate measurement is determined by the accuracy desired and is influenced by errors introduced by the resistive and inertial forces affecting the movement of the liquid plug within the capillary tube. Changes in atmospheric pressure (barometric readings) may move the liquid slug in capillary systems with one end open to the earth's atmosphere. As the speed of movement of the liquid plug decreases, these errors are increased. This causes the leakage measurements to become more inaccurate with slow movements of the liquid plug.

Errors due to starting inertia are decreased with liquids of lower density. Errors can be reduced, for example, by using a water plug about 1 mm (0.04 in.) long and timing the movement of the water plug only after it reaches a constant velocity. If a water plug is used, the error due to the resistive forces of surface tension can be minimized by coating the inside (bore) surface of the clean capillary tubing with an organosilicon compound. This coating acts to prevent the water from wetting the glass.

Mercury is almost impossible to use for the liquid slug in a glass capillary. Mercury has a very high surface tension and it is almost impossible to force it into a very small diameter capillary tube bore. However, there should be negligible gas transfer through a mercury plug.

An ideal fluid for use as the indicator plug in a glass capillary should have the following characteristics.

1. It should be a fluid in which the leaking gas is not soluble, so that no gas transfer by diffusion can occur through it,.
2. The fluid should not wet the walls of the tube, so that the surface tension forces on either end of the plug are balanced.
3. The fluid should be opaque for easy visibility and measurement of its position.
4. The fluid should have a low surface tension so that it can be placed easily within the bore of the capillary tube.

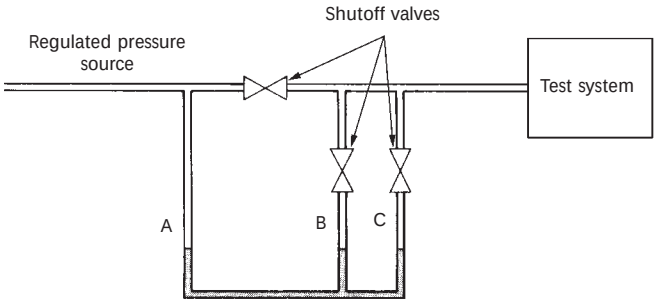
Alternative Flow Measurement Instruments Used in Sealed Volume Leakage Tests

The basic principles of sealed volume leak testing can be used in numerous ways. For large leaks, flow measuring devices such as a wet type gas meter or a rotameter may be used. These instruments produce accurate leakage rate measurements but are useful only on very large leaks. For measurements over a wide range of leakage rates, the instrument shown in Fig. 29 can form a U-tube capable of withstanding extremely high pressures. Tubes B and C have different diameters so that the proper tube can be selected for measuring various leakage rates.

When all the valves in Fig. 29 are open and the test components are pressurized, the liquid columns all reach the same height. By closing the shutoff valve in the main line between columns A and B, leakage is indicated by upward movement of fluid in columns B or C. The meter in Fig. 29 was designed primarily for determining leakage rates in hydraulic power systems. The principle of operations is to displace the leaking fluid with the indicating fluid. This can be done because there is a pressure loss in the leaking component. When the meter of Fig. 29 is installed in the hydraulic power line to the component being tested, leakage can be measured by the displacement of the separation level between the two different liquids in column A as compared with column B or C.

In another type of flow meter, leakage flow in the line between meter and component under test is measured by the

FIGURE 29. Connection of delta-vee U-tube manometer for leakage measurements, with tubes (A,B,C) of different diameters.



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displacement of a bellows. The deflection of the pressure difference sensing bellows system varies the setting of a potentiometer. An output electrical signal from the potentiometer indicates leakage directly in volume units. This bellows system replaces the observation of movements of a liquid slug in a capillary tube. Each of the preceding types of flow meter will work with liquids as well as with gases, provided that the indicating liquid slug is immiscible in the fluid whose leakage is being measured. This versatility makes the sealed volume leak testing techniques extremely useful for leak testing under operational conditions.

Fast Response Thermopile Mass Flow Meter

The flow meter (Fig. 30a) comprising a sensor, electronic circuitry and a shunt measures gas flow rate from 0 to 60 $Pa \cdot m^3 \cdot s^{-1}$ (0 to 600 std $cm^3 \cdot s^{-1}$). The shunt causes the flow to divide such that the flow through the sensor is a precise percentage of the flow through the shunt. The circuit board amplifies the sensor output linearly to a 0 to 5 V direct current signal proportional to the flow rate.

A thermal sensor measures flow through a capillary tube. This flow is a fixed percentage of the total flow through the instrument. This sensor develops an essentially linear output signal proportional to flow, which is about 0.8 mV full scale magnitude (Fig. 30b). This signal is amplified by the meter circuitry so that the full scale output is 5.00 V direct current. The output is routed to interface terminals and to decoding circuitry in the display.

Measurement of flow rates higher than 60 $Pa \cdot m^3 \cdot s^{-1}$ (600 std $cm^3 \cdot s^{-1}$) full scale is achieved by dividing the flow with a fixed ratio shunting arrangement. The measuring capillary tube is placed parallel with one or more dimensionally similar channels, call laminar flow elements. The sensor only needs to heat the gas passing through the capillary tube while retaining all the mass measuring characteristics.

The fast response of this instrument at very low rates of air flow permits fast, accurate leak testing by manual or automatic means. Table 14 lists multiplication factors for the air scale meter indications when this flow meter is used for gases other than air.

The metal capillary tube of Fig. 30b is heated uniformly by current from the transformer. The temperature distribution is symmetrical about the tube midpoint with zero flow (Fig. 30b). The external thermocouples TC_1 and TC_2 develop equal but opposing electromotive force outputs with a symmetrical temperature

FIGURE 30. Mass flow meter with thermal sensor that measures flow through capillary tube: (a) photograph; (b) schematic of components of thermal mass flow transducer; (c) temperature distribution under static (no-flow) and flowing conditions in flow meter transducer system.

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distribution. When air or gas flows through the tubing, heat is transferred to the gas and back again, creating an asymmetrical temperature distribution (Fig. 30c). For constant power input to the tube, the differential thermocouple output voltage is a function of the mass flow rate and heat capacity of the gas. Changes in gas composition requires only a simple multiplier of the air calibration to account for the differences in heat capacity. The flow meter can be used for a wide variety of gases during leakage rate testing. The full scale flow through the flow meter is about 1 $Pa \cdot m^3 \cdot s^{-1}$ (10 std $cm^3 \cdot s^{-1}$).

Figure 31 shows typical arrangements for leak testing of small items. Figure 31a shows a pneumatic bridge arrangement. The object to be tested and an identical, leaktight part used as a reference volume are charged with air at pressures up to 135 kPa (20 $lb_f \cdot in.^{-2}$) gage. The effects of adiabatic heating or cooling of the air during the pressurizing cycle should be avoided. The flow meter is then connected between the unknown and reference parts to detect any evidence of leakage (which would allow the pressure to decrease in the part under test). Because the adiabatic effects are nearly identical in the reference and the test parts, the thermopile flow meter quickly detects the leakage rate without requiring a waiting period for attainment of full equilibrium in temperatures and pressures. Leakage testing may also be done by a direct inline leak testing procedure, as sketched in Fig. 31b, but this test procedure requires a longer time cycle than the differential flow measurement technique.

TABLE 14. Multiplication factors for different gases of mass flow meter air scale.[a]

Gas	Symbol	Conversion Factor[b]	Density[c] ($g \cdot L^{-1}$)	Relative Specific Gravity[d]
Acetylene	C_2H_2	0.67	1.09	0.90
Air		1.00[e]	1.20	1.00
Ammonia	NH_3	0.77	0.71	0.59
Argon	A	1.43[e]	1.66	1.38
Arsine	AsH_3	0.76	3.25	2.70
Bromine	Br_2	0.88	5.98	4.96
Butane	C_4H_{10}	0.30	2.51	2.08
Butene 1	C_4H_8	0.34	2.40	1.99
Carbon dioxide	CO_2	0.73[e]	1.84	1.53
Carbon monoxide	CO	1.00[e]	1.17	0.97
Chlorine	Cl_2	0.85	2.98	2.47
Chlorine trifluoride	ClF_3	0.45	3.78	3.14
Cyclopropane	C_3H_6	0.52	1.75	1.45
Diborane	B_2H_6	0.50	1.15	0.95
Ethane	C_2H_6	0.56	1.26	1.05
Ethene (ethylene)	C_2H_4	0.69	1.17	0.97
Ethylene oxide	C_2H_4O	0.60	1.79	1.49
Fluorine	F_2	0.93	1.58	1.31
Helium	He	1.43[e]	0.17	0.14
Hydrogen	H_2	1.03[e]	0.08	0.07
Hydrogen chloride	HCl	1.01	1.48	1.23
Hydrogen fluoride	HF	1.00	1.53	1.27
Hydrogen sulfide	H_2S	0.85	1.43	1.19
Isobutane	C_4H_{10}	0.31	2.48	2.06
Krypton	Kr	1.39	3.49	2.90
Methane	CH_4	0.69[e]	0.68	0.56
Neon	Ne	1.38	0.84	0.70
Nitric oxide	NO	1.00	1.24	1.03
Nitrogen	N_2	1.02[e]	1.17	0.97
Nitrous oxide	N_2O	0.75	1.85	1.54
Oxygen	O_2	0.97[e]	1.33	1.10
Pentaborane	B_5H_9	0.15	2.83	2.35
n-Pentane	C_5H_{12}	0.22	3.18	2.64
Phosphine	PH_3	0.79	1.53	1.27
Propane	C_3H_8	0.32[e]	1.89	1.57
Refrigerant-11	CCl_3F	0.36	5.93	4.92
Refrigerant-12	CCl_2F	0.36[e]	5.13	4.26
Refrigerant-13	$CClF_3$	0.42	4.59	3.81
Refrigerant-14	CF_4	0.48	3.65	3.04
Refrigerant-22	$CHClF_2$	0.43[e]	3.65	3.03
Refrigerant-114	$CClF_2$	0.22[e]	6.99	5.80
Silane	SiH_4	0.68	1.33	1.10
Sulfur dioxide	SO_2	0.70	2.72	2.26
Sulfur hexafluoride	SF_6	0.28	6.43	5.34
Tungsten hexafluoride	WF_6	0.23	8.22	6.82
Uranium hexafluoride	UF_6	0.23	14.65	12.16
Water vapor	H_2O	0.80	0.76	0.63
Xenon	Xe	1.37	5.54	4.60

a. No corrections or compensations for temperature or pressure of gas required.
b. Multiply air scale by these conversion factors.
c. Density in grams per liter at 20 °C (70 °F) and 100 kPa (1 atm).
d. Specific gravity (air = 1.00).
e. Empirical data; other data is theoretical. Example: Flow meter NALL-1K, 0–1000 std $cm^3 \cdot s^{-1}$ in air would be $1000 \times 1.43 = 1430$ std $cm^3 \cdot s^{-1}$ at full scale in helium.

Orifice Flow Detector with Differential Pressure Transducer

Figure 32a shows a leakage test instrument system that uses an orifice to convert flow across the orifice element into a pressure differential sensed by the differential capacitance sensor (see also Fig. 14a). The orifice (which produces a pressure loss when air flows through it) is connected in series with the air supply line to the item under test, as shown in Fig. 32). This system is used with automatic flow and leakage testers providing fully automatic cycling and accept/reject test indicators and output signals. Leakage sensitivity and stabilization time are both programmable.

A compensation network provides a programmed electronic time base signal to match the dynamic characteristics of short time cycle flow measurements. Figure 32a is a photograph showing instrument connections to the differential pressure transducer (capacitance gage) and the orifice or flow restriction element (in this example, a short length of tubing). Typical ranges vary from 0.05 to 250 $L \cdot s^{-1}$ (0.002 to 9.0 $ft^3 \cdot min^{-1}$). The dynamic range is indicated to be 50:1 for operation with laminar flow pressure loss elements.

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Total leakage test cycle times from 0.5 to 2 s are obtainable. The flow compensation network allows dynamic air flow measurements without requiring a stabilization period.

Digital Electronic Flow Meter for Monitoring Leakage Rate Tests

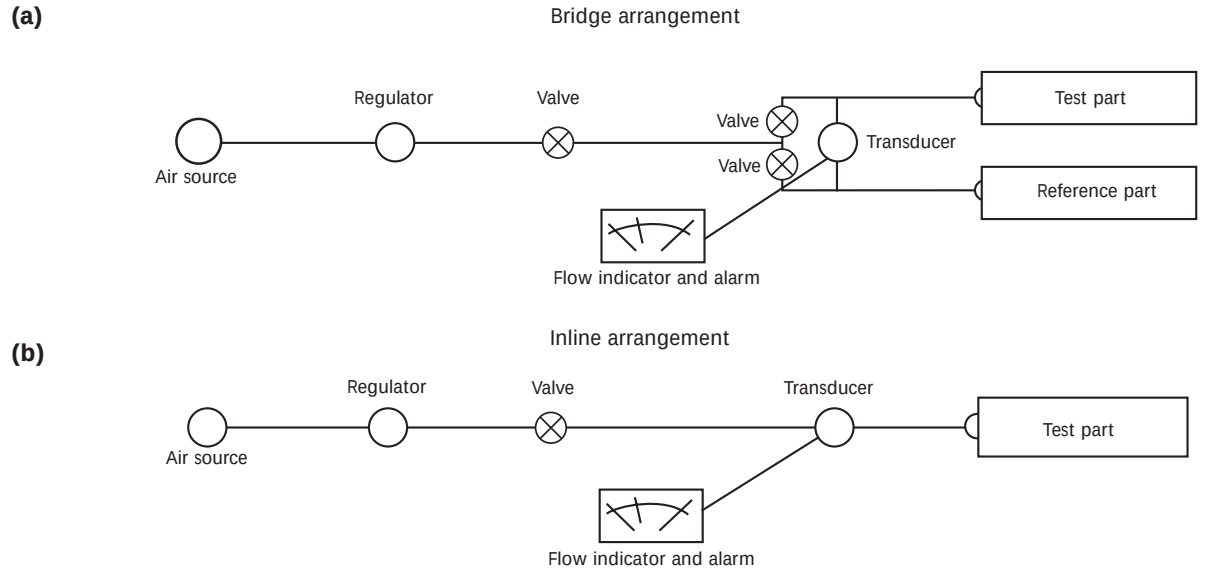
Figure 33 shows a portable, digital electronic flow meter designed for fast, accurate indication of leakage rates of pressurized components such as valves, O-ring seals, pressure vessels, holding tanks, tank cars and processing vessels. It provides a broad range of flow rate measurements up to 2×10^2 Pa·m^3·s^{-1} (2×10^3 std cm^3·s^{-1}) with a resolution of one part in 2000. Repeatability is indicated as ±0.2 percent of full scale and accuracy is ±1.0 percent of full scale values. The instrument includes a broad range, high accuracy solid state digital flow indicator, combined with an integral flow regulator and a digital pressure indicator. The only additional equipment required is a pressure source such as instrument air or nitrogen for tests as pressures up to 400 kPa gage (0 to 60 lb$_f$·in.$^{-2}$ gage) or 700 kPa gate (0 to 100 lb$_f$·in.$^{-2}$ gage) and means for making connections to the test unit. Once the system and object under test have been pressurized, operation is changed from the charge mode to the leakage test mode. The leakage rate indication is displayed instantaneously in standard engineering units and in the range selected. A self check mode provides means for verifying the integrity of the flow monitor both electrically and pneumatically.

Vacuum Pumping Technique of Leak Testing with Flow Measurements

If the test system can be safely evacuated, leakage can be measured directly by means of flow meter with vacuum pumping arrangement sketched in Fig. 34. The system under test is evacuated through an opened isolation valve connected to the vacuum pump inlet. The exhaust gases from the vacuum pump go through a surge tank to the flow meter. A bypassing valve around the pump provides an alternative path between the isolation valve and the surge tank. Before performing the leak test, the vacuum pumping system leak tightness is first determined by closing the isolation valve and measuring the rate of gas flow through the flow meter. If this flow is negligible, the isolation valve is then opened and the flow meter readings are taken only after an equilibrium (constant flow rate) condition has been achieved.

The vacuum pressure in the system under test is adjusted by means of the bypass valve, which controls the backflow of gas from, the exhaust port of the vacuum pump to its inlet port. The lower limit of vacuum pressure for which the

FIGURE 31. Arrangements for leak testing with thermopile air flow meter: (a) pneumatic bridge leakage testing arrangement with thermopile flow meter arranged to measure difference in pressure between test object and an identical leaktight object (reference volume); (b) inline leakage testing arrangement in which test part is pressurized, line valve is closed and leakage is indicated by pressure drop in flow meter sensing element.



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vacuum pumping leak analysis technique is useful is in the range of 3 kPa (25 torr). The lower limit of leak testing sensitivity is about 0.1 $Pa \cdot m^3 \cdot s^{-1}$ (1 std $cm^3 \cdot s^{-1}$) and is mainly dependent on the availability of suitable flow meters for the vacuum pressure range used during the leak test.

Sealed Volume Flow Meter Leak Testing of Nuclear Containment Systems

Sealed volume leak testing techniques are also used on large volume systems such as nuclear containment systems. For this application, this procedure is commonly called a verification test. Its purpose is to verify the accuracy of the leakage test results and instrumentation used in that test. It also verifies the validity of the dewpoint and temperature sensor locations within the containment structure. Flow meters used in these large scale leakage rate tests include thermal mass flow sensors, rotameters and integrating gas flow meters usually with ranges of 25 to 700 $Pa \cdot m^3 \cdot s^{-1}$ (0.5 to 15 std $ft^3 \cdot min^{-1}$). These flow meters are usually designed for the planned leak testing conditions and they produce readouts compensated to standard pressure and temperature conditions. The accuracy of the flow meter must be

FIGURE 32. Air flow meter with orifice and differential pressure transducer: (a) photograph; (b) pneumatic circuit.

(a)

(b)

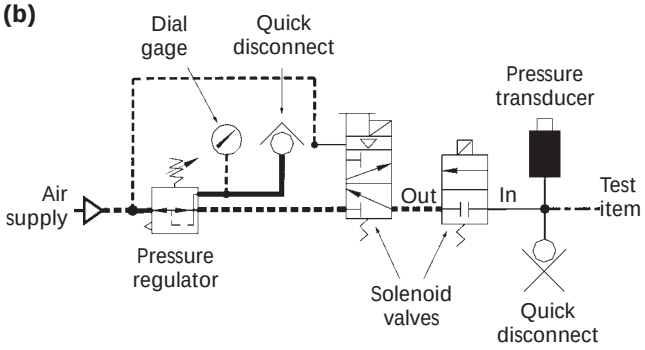
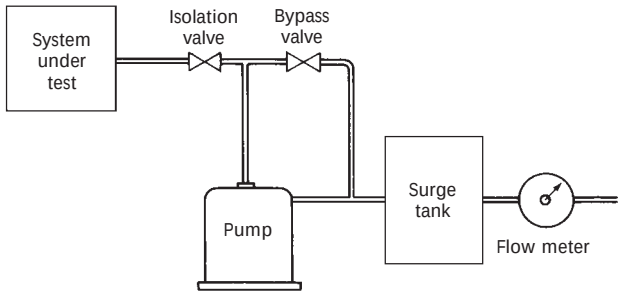


FIGURE 33. Portable digital electronic flow meter for monitoring leakage rates in pressurized systems.

FIGURE 34. Arrangement for vacuum pumping technique of leakage measurement with flow meter.



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commensurate with the accuracy of the leakage rate test instrumentation and also with the accuracy required in the containment leakage rate test results.

Procedures for Flow Meter Verification Test of Nuclear Containment Systems

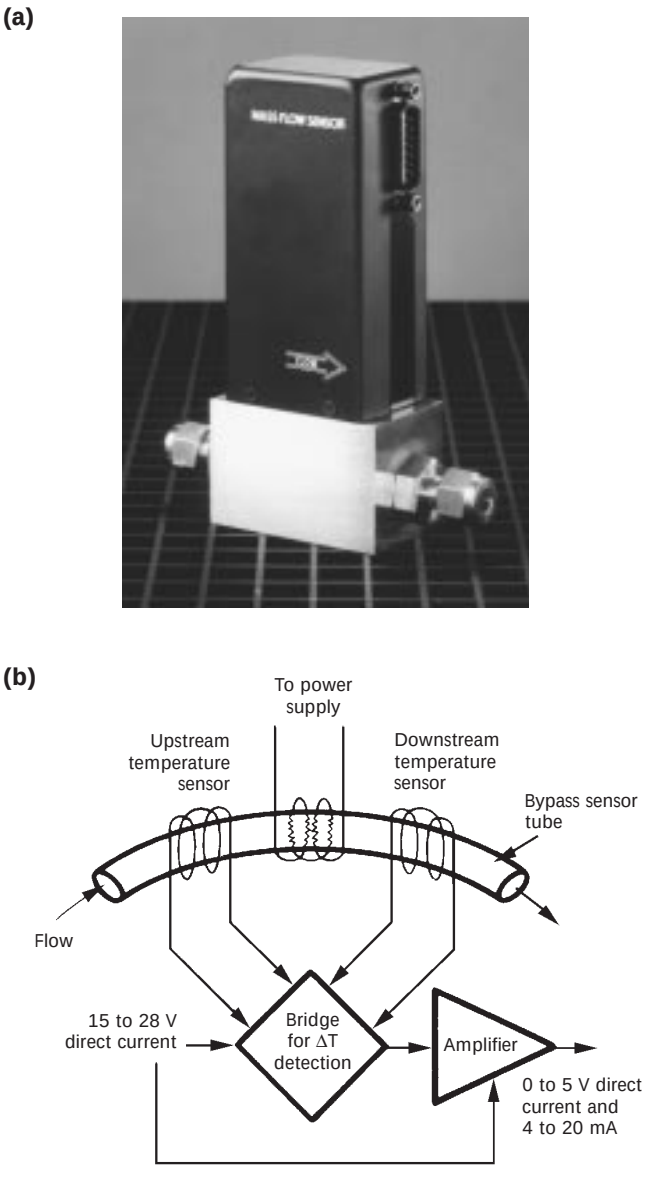
The verification test is normally performed as the last phase of a containment test. It follows the test for the system measured leakage rate Q_{am} (usually given as a percentage of air mass lost in 24 h). The flow meter is installed in the system with a valve to isolate it from the system under test. The verification test may be performed by measuring either the out-leakage or the in-leakage that passes through the flow meter. For either technique, a meter valve is placed downstream from the direction of leakage flow through the flow meter, to minimize the pressure loss across the flow meter. After opening the isolation valve between the test system and the flow meter, this metering valve is adjusted to produce a leakage flow through the flow meter from (or into) the test system that is some required percentage (usually 75 to 125 percent) of the allowable leakage rate Q_a for the system under test.

The leakage rate test of the containment is then continued. After a period of 4 to 6 h with a minimum of ten sets of data, the combined leakage rate Q_c of the containment system and flow meter and the leakage rate Q_0 of the flow meter are determined using the flow meter readings. The difference between these two leakage rates is $Q_c - Q_0 = Q'_{am}$. This difference Q'_{am} in reading is then compared to the leakage rate Q_{am} measured previously on the containment test system alone, before the inflow or outflow of air from the containment through the flow meter. The two values must agree with 25 percent of the measured containment leakage rate Q_{am}. This is to say that $Q_{am} - Q_{am}$ must be equal to or smaller than 0.25 Q_{am}.

True Thermal Mass Flow Meters for Accurate Flow Rate Measurements

The containment verification test just described requires a true mass flow meter that measures the mass of gas that passes through it. Figure 35 shows a true mass flow sensor element which does not require temperature or pressure compensation and provides ±1 percent of full scale accuracy and linearity. The sensor unit has a stainless steel flow tube. A heater coil is wound around the center section of its length. Sensor coils are wound around the flow tube on either side of the heater coil and are connected in a bridge circuit. The zero flow, the bridge circuit is balanced and the output signal is zero. With flow the sensor coils detect the resulting temperature difference, which is proportional to mass flow. The output electrical signal varies linearly with the gas flow rate. Signals can be used for measuring, recording or controlling gas flow rates with valves and an automatic controller. Sensors for specific gases such as air, nitrogen, hydrogen, oxygen and helium are

FIGURE 35. Thermal mass flowmeter uses a true mass flow sensor for measuring gas flow rates accurately: (a) sensor; (b) principle of operation.



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available with ranges from 0 to 0.015 up to 0 to 10 $Pa \cdot m^3 \cdot s^{-1}$ (0 to 10 up to 0 to 5×10^3 std $cm^3 \cdot min^{-1}$). Repeatability of indications is claimed as ±0.2 percent of full scale. Output signals from the thermal mass flow sensors of Fig. 35 can actuate indicating meters or provide 0 to 5 V direct current signals that can be transmitted up to 300 m (1000 ft) to recording instruments, digital indicators or controllers. The electrical output signal is linearly proportional to the mass flow rate through the sensor.

Flow Meter Tests to Locate Leaks in Gas Filled Electric Power Cables

Electric utility companies have made use of a U-tube manometer equipped with appropriate valving to use as a flow meter for locating gas leaks in gas pressurized electric power cable sheaths. When the manometer is installed in a segment of the pressurized gas filled cable sheathing, oil will rise in the glass tube of the manometer, on the side closer to the leak. In this test, the manometer measures the pressure loss in the segment of cable across which it is connected, when gas flows toward the leak.

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CHAPTER 6

Leak Testing of Vacuum Systems

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PART 1. The Nature of Vacuum

Definition of a Vacuum

The word *vacuum* is derived from the Greek word meaning empty. In practice, use is made of some type of vessel (vacuum enclosure, chamber or container) to contain a vacuum. When the enclosure is closed to the surrounding atmosphere and air or gas is removed by some pumping means, a vacuum is obtained. Various degrees of vacuum can be obtained, depending on how much air is removed from the enclosure. Common terms such as partial vacuum, rough vacuum, high vacuum and ultrahigh vacuum are used to describe degrees of vacuum. A vacuum is any pressure below the prevailing atmospheric pressure. Practically speaking, a vacuum such that the containing vessel is empty, i.e., free of all matter (molecules), is never obtained. If this were possible, the vacuum would be called a perfect or absolute vacuum.

Applications of Vacuum Environments

Vacuum is used to reduce the interaction of gases or air with solids and to provide control over electrons and ions by reducing the probability of collision with molecules of air. Vacuum pumps are used by industry and laboratories to create a vacuum environment for these operations. Most gases react with solids to cause effects such as oxidation, which it may be necessary to avoid. In a vacuum environment, the necessary operation may be performed so that undesirable effects are reduced or eliminated. For example, unless most of the air is removed from an incandescent light bulb, oxygen in its atmosphere will react with the hot tungsten filament, causing it to burn out prematurely. An electron tube could not operate at atmospheric pressure. Electron flow would be impeded by collision with air molecules due to the extremely small mean free path. In addition, elements within the tube may react with the air. Other examples can be cited where vacuum is necessary to produce desired results that could be unattainable in any other way.

Vacuum is required in many industries and products. In addition to light bulbs and computer chip manufacturing, vacuum is used in magnetrons, cathode ray tubes, television picture tubes, semiconductor devices, solar cells, plating metals and plastics, thin film deposition, lifting objects, plasma physics, cryogenics, metallurgical processing, electron beam welding, brazing, distillation organic chemistry, packaging, mass spectrometry, space simulation and leak detection. Many other areas find application for vacuum equipment.

Changes in Pressure Units Used for Vacuum Measurements

The presently preferred SI unit for pressure is the pascal (Pa). The standard atmospheric pressure at sea level and 0 °C (32 °F) is equal to 101.325 kPa. Earlier units used for pressure in vacuum relate to atmospheric pressure indicated by the height (nearly 760 mm) of the mercury barometer column at sea level and 0 °C (32 °F). The unit known as the torr was defined as 1/760th of the pressure of the mercury column. The torr was named in honor of an Italian physicist, Evangelista Torricelli (1608-1647), inventor of the mercury barometer. The torr is almost identical to the millimeter of mercury (mm Hg), because there are 759.96 torr in a standard atmosphere. The difference between the two units amounts to so little that torr and mm Hg have been used interchangeably.

Variation of Atmospheric Pressure with Altitude

The mercury barometer is a device for measuring atmospheric pressure. As the altitude increases, the pressure decreases because fewer gas molecules press on any surface. A knowledge of how the pressure changes with altitude is very important in connection with various space studies. Table 1 shows the relationship between pressure and altitude in the earth's atmosphere.

At an altitude of 50 km (27 mi) the pressure is about 0.1 percent of standard atmospheric pressure or 100 Pa ($0.015\ lb_f \cdot in.^{-2}$). Air at this altitude

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contains one thousandth of the number of molecules per unit volume in air at sea level. At 400 km (250 mi) altitude, the pressure is in the range of 1 μPa or 10^{-11} parts of sea level pressure. Table 2 gives a relative measure of gas characteristics at sea level and at 1 nPa (10 ptorr). Compared with the number of molecules in a cubic centimeter at atmospheric pressure, it is seen that there are one hundred thousandth of one millionth as many molecules at 10^{-6} Pa (1.5×10^{-10} $lb_f \cdot in.^{-2}$). However, a tremendous number of molecules (3×10^8) still remain in a cubic centimeter at a pressure of 1 μPa (10 ntorr). Pressures around 1 μPa (10 ntorr) are not uncommon in good vacuum systems.

Diffusion and Adsorption of Gases in Vacuum Systems

The kinetic molecular theory of gases and the ideal gas laws (Boyle's, Charles', Dalton's and the general gas law), are applicable to vacuums. In vacuum, fewer molecules are dealt with, but their basic behavior is predictable by the molecular theory of gases and does not change. The ability of a gas to diffuse increases when its pressure is reduced. Consider the example of ammonia vapor being released in a room. The reason that it is not detected immediately at the other end of the room is that the path each ammonia molecule takes is restricted by the air molecules with which it collides. It is only after many billions of collisions with air molecules that the ammonia molecules finally make their way across the room. If the room were pumped down a high vacuum, there would be many fewer air molecules and far fewer collisions to impede the path of the ammonia molecules. Thus, an ammonia molecule in a high vacuum takes less time to complete its trip across a given distance than in gases at higher pressures.

Only those molecules that are in motion within a vacuum chamber create a pressure through collisions with its walls. A molecule that is adsorbed to the wall surface is stationary and does not produce collisions. Therefore, adsorbed gas molecules do not contribute to the total pressure. However, molecules adsorbed on surfaces can be returned to the gas phase by thermal agitation produced by the application of heat. Thus, outgassing effects can contribute to pressure in an evacuated system, because a molecule can undergo repeated collisions and exert pressure only when it is in the gaseous state.

TABLE 1. Change in atmospheric pressure with altitude.

Altitude		Pressure		
km	(mi)	kPa	(atm)	Remarks
0		101.325	(1.00)	international standard
1	(0.6)	89.90	(0.887)	
2	(1.2)	79.50	(0.785)	
5	(3.1)	54.00	(0.533)	
10	(6.2)	26.50	(0.262)	jetliner altitude
20	(12.4)	5.53	(0.055)	
50	(31.1)	7.98×10^{-2}	(7.9×10^{-4})	
100	(62)	3.2×10^{-5}	(3.2×10^{-7})	
200	(124)	8.5×10^{-8}	(8.39×10^{-10})	low orbit
500	(311)	3.0×10^{-10}	(3×10^{-12})	
1000	(621)	7.5×10^{-12}	(7.4×10^{-14})	

Mean Free Path of Gases in Vacuum Systems

At normal atmospheric pressure, gas molecules make many collisions with each other. The average distance that a molecule travels before colliding with another molecule is known as the *mean free path*. The mean free path of two different gases at the same pressure will not be the same; this is because the mean free path depends on the molecular size, which varies from one gas to another. In spite of this fact, it is still possible to give a useful relationship between mean free path and pressure. The approximate values of mean free paths for air and

TABLE 2. Comparison of atmospheric properties at sea level and at high altitude.

Condition	At Sea Level	At 400 km (250 mi) Altitude
Pressure	101.325 kPa (760 torr)	1 μPa (10 ntorr)
Number of molecules in 1 cm^3 (0.06 $in.^3$)	2.7×10^{19}	3×10^8
Mean free path	93 nm (3.7×10^{-6} in.)	9.3 km (5.8 mi)
Time to form a monolayer of adsorbed gas on a clean surface	>10 ns	120 s
Average speed of nitrogen molecule at room temperature 20 °C (68 °F)	1600 $km \cdot h^{-1}$ (1000 $mi \cdot h^{-1}$)	1600 $km \cdot h^{-1}$ (1000 $mi \cdot h^{-1}$)

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other gases are given as a function of gas pressure in Eq. 1:

$$\lambda = \frac{0.0095}{P} \quad (1)$$

where λ is mean free path (meter) and P is gas absolute pressure (pascal).

Effects of Molecular Friction and Gas Viscosity in Viscous Flow

As shown by Eq. 1, the mean free path length varies inversely with absolute gaseous pressure. The concept of mean free path is useful in describing vacuum ranges. The mean free path at atmospheric pressure is very short (see Table 2), due to the large molecular density. Therefore, collisions occur much more frequently between gas molecules than between molecules and the walls of the container. Thus, the gas acts much like a fluid. Under a pressure differential this gaseous fluid moves as a unit and is considered to flow. The molecules, while drifting slowly in the direction of flow, move rapidly along random paths. Any resistance to this flow is due to the viscous properties of the gas. The term viscous refers to molecular friction and is used to describe the flow characteristics of a fluid. Water, for example, is less viscous than syrup because it flows or pours more easily. The cross sectional dimension of the container or tube through which the gas flows is important because it determines the velocity of the molecules within the flowing gas. The viscous properties of the gas are functions of the gas viscosity and the gas velocity. When viscous properties control gas flow rates, the situation is termed viscous flow.

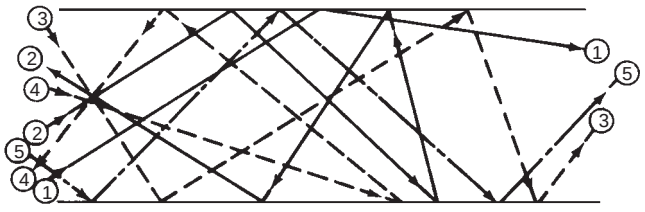
Effects of Mean Free Path and Flow Cross Section on Molecular Flow of Gas

As the pressure of the gas within a system is reduced, the mean free path of the molecules increases and the flow characteristics change gradually. As the mean free path becomes comparable to the cross sectional dimensions of the tube, collisions occur less frequently between molecules and the apparent viscosity of the gas decreases. Under these conditions, the event that is most likely to affect the direction of the molecules; travel is a molecular collision with the tube wall. As the pressure is further reduced, the mean free path becomes greater than the tube's cross sectional dimensions. The diameter of the tube alone then determines the resistance to flow; this situation is called molecular flow. The motion of a particular molecule is entirely random and unpredictable; it is as likely to move in one direction as in any other direction. To a molecule, tube wall appear very rough and irregular. The direction of molecule rebound after impact with the tube wall thus tends to be independent of the direction of incidence. (This is an over simplified description.) Figure 1 is a sketch of particle motions during molecular flow of gases through a tube. Note that not all of the molecules entering at the left exit at the right. The gas flow will continue as a net movement to the right only as long as there is some driving force causing movement from left to right. As gas concentration gradient is such a force. A pressure differential is another force that can control the direction of net flow of a gas. Both can contribute to flow of a tracer gas through a leak.

Specifying Gas Flow Rates

The flow rate of liquids is expressed simply as so many volume units per unit time, such as liters per second. When, however, the flow rate of gases is considered, it is necessary to know not only the volume of a gas but its pressure and temperature as well. A cubic meter volume of gas at 100 kPa (15 $lb_f \cdot in.^{-2}$) pressure and a temperature of 20 °C (68 °F) will contain ten times as many molecules as a cubic meter volume of gas at 10 kPa (1.5 $lb_f \cdot in.^{-2}$) and 20 °C (68 °F). Only a complete statement of volume, displacement rate, gas pressure and temperature can accurately describe the total quantity of gas that flows per unit of time. In both liquids and gases, it is mass flow that is of interest. For liquids of constant density, the mass rate of flow is directly proportional to volume flow rate. With gases, density varies both with temperature and with pressure. Thus, for a given gas, volume displacement rate, pressure and temperature must be known to define the mass flow rate.

FIGURE 1. Molecular motion along a tube, with particle mean free path far larger than tube diameter.



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The Concepts of Gas Quantity and Pumping Speed

From the gas laws, it is known that the product PV of pressure P and volume V is proportional to the number of molecules in a sample of gas. In static systems, the PV product is constant at a given temperature. This product PV is known as the *quantity* of gas. Common units of gas quantity include torr liter (torr-L); the atmospheric cubic centimeter (cm^3 of volume at standard sea level atmospheric pressure or std cm^3); and the bar liter (bar-L). The preferred SI unit of gas quantity is the pascal cubic meter ($Pa \cdot m^3$).

In steady flow, the same quantity of gas (number of molecules) that enters one end of a tube must leave at the other end, even though there may be different volumes of gas entering and leaving per unit time. If the PV product is used as a measure of the amount of gas flowing through a tube, computation may be done with a minimum of complication.

The volumetric pumping speed S is the time rate of volume displacement, as given by Eq. 2:

$$S = \frac{V}{t} \quad (2)$$

Typical units of pumping speed S would be cubic meter per minute ($m^3 \cdot min^{-1}$), cubic meter per second ($m^3 \cdot s^{-1}$), liter per second ($L \cdot s^{-1}$) and cubic foot per second ($ft^3 \cdot s^{-1}$).

Concepts of Throughput and Leakage Rate

In vacuum practice, the preferred description of the rate of flow of gas is commonly called throughput. Throughput is the quantity of gas or a measure of the total number of molecules at a specified temperature, passing an open section of the vacuum system per unit time. Leakage rate is a similar measure of the total number of molecules at a specified temperature passing through a leak per unit time. Q is the symbol commonly used for gas throughput per unit time, in pascal cubic centimeter per second:

$$Q = \frac{PV}{t} \quad (3)$$

By combining Eqs. 2 and 3, the product of pumping speed S and gas pressure P can be equated to throughput by Eq. 4:

$$Q = S \times P \quad (4)$$

Equation 4 is the universal relationship on which vacuum pumping throughput calculations are based. As an example of its use, suppose the gas in the pipe between Sections 1 and 2 of Fig. 2 passes Section 1 in 1 s and this volume V is 100 L (0.1 m^3) and pressure P at Section 1 is 10^{-4} Pa and displaced volume V = 0.1 m^3, divided by the time t = 1 s:

$$Q = S \times P = \frac{PV}{t}$$
$$= 10^{-4} \times 0.1 = 10^{-5}$$

Comparison of Gas Flow with Liquid Flow

Before attempting a more thorough discussion of gas flow, it may be helpful to compare gas flow with water flow. To get any fluid to flow within a pipe, a pressure differential must be established between the two sections across which the fluid is to flow. (Gravitational effects are neglected in this introductory discussion.) The fluid would then flow from the high pressure region P_1 to the low pressure region P_2. Consider a closed system of pipes through which water is circulated as in an automobile. The water pump creates the pressure differential necessary for water to flow. Across each component (radiator, engine block, thermostat, different sizes of piping) in the system, the pressure drops. The sum of all these pressure drops equals the pressure differential across the water pump. The magnitude of the pressure drop across each component of the system depends on its physical geometry. Clearly, a smaller diameter pipe will result in decreased flow for the same size pump. similarly, increasing the length of the pipe will reduce the flow, whereas decreasing the length of the pipe will increase the flow. Shorter lengths and larger diameters reduce the resistance to flow through the pipe.

Analogy of Gas Flow to Electric Current through Resistance

The analogy may be carried further by comparing the gas flow system to an electrical circuit. Given an electrical circuit with a battery and a resistor in series with it, the battery may be considered to be the pump and the resistor the pipe. Increasing or decreasing the resistance decreases or increases the current flow (analogous to gas flow),

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respectively. If the circuit consists of a series of resistors and a battery, the sum of the voltage drops across each of the resistors (pressure drops) is equal to the total voltage generated by the battery (pressure differential created by the pump). The voltage drop across each resistor will depend on the magnitude of resistance of that component. The larger the resistance, the larger the voltage drop (see Fig. 3).

Gas Conductance and Its Electrical Analogy

In vacuum, one speaks not of the resistance a tube or component offers to gas flow, but instead uses the reciprocal term conductance. Conductance is a measure of the ability of a vacuum component to permit gas flow or not to impede it. Consequently, the greater the resistance, the smaller the conductance and vice versa. Figure 3 shows the electrical analogy of a tube in a vacuum system. The battery is analogous to the vacuum pump, current is analogous to gas flow and the resistor is analogous to pipe.

FIGURE 2. Rate of flow of a gas Q through tube with applied pressure differential $\Delta P = P_1 - P_2$ ($P_1 > P_2$). (See analogous electric circuit of Fig. 3.)

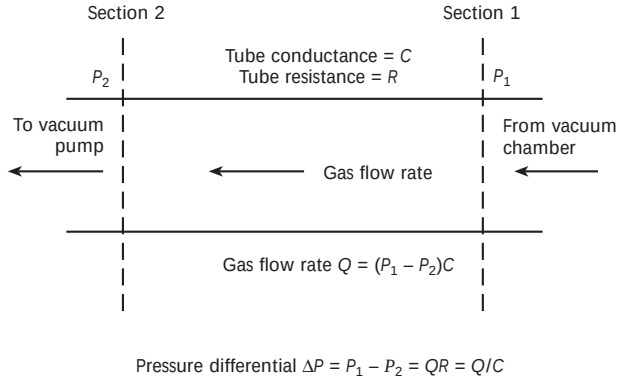
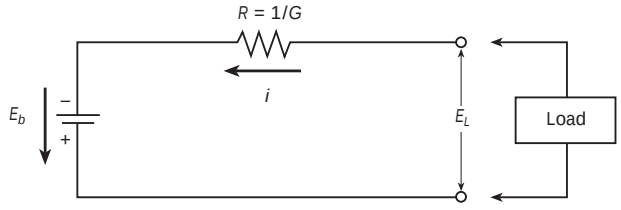


FIGURE 3. Electrical analogy of vacuum pumping pipe conductance system of Fig. 2. G is the electrical conductance, the reciprocal of electrical resistance R, so that $G = 1/R$. $E_R = E_b - E_L = E_1 - E_2 = IR = I/G$.



In an electrical circuit, the voltage drop across a resistor is the product of the current and resistance. In a vacuum circuit, the pressure differential across a pipe is the product of throughput (gas flow) Q and resistance R. Equation 5 states this relation mathematically for the pressure differential ΔP:

$$\textbf{(5)} \quad \Delta P = P_1 - P_2 = Q \times R = \frac{Q}{C}$$

Because R is equal to $1/C$, Eq. 5 may be written in the form of Eq. 6 for gas conductance C:

$$\textbf{(6)} \quad C = \frac{Q}{\Delta P}$$

Equation 6 is the defining equation for gas conductance: the ratio of throughput Q to pressure differential ΔP across the conductance.

Gas Conductance with Sequential Tubes of Passages

If two different diameter pipes with different conductance values are connected in series as in Fig. 4a, the total conductance of the connection between extreme ends decreases (resistance increases). From Eq. 6, the conductance of the pipe between Sections 1 and 3 may be expressed as in Eq. 7:

$$\textbf{(7)} \quad C_{13} = \frac{Q}{P_1 - P_3}$$

$$\textbf{(8)} \quad P_1 - P_3 = \left(P_1 - P_2\right) + \left(P_2 - P_3\right)$$

$$\textbf{(9)} \quad P_1 - P_2 = \frac{Q}{C_{12}}$$

or

$$P_2 - P_3 = \frac{Q}{C_{23}}$$

Now, by combining Eqs. 7, 8 and 9, the relationship for C_{13} becomes:

$$\textbf{(10)} \quad C_{13} = \frac{Q}{\frac{Q}{C_{12}} + \frac{Q}{C_{23}}}$$

or, in its reciprocal form:

$$\textbf{(11)} \quad \frac{1}{C_{13}} = \frac{\frac{Q}{C_{12}} + \frac{Q}{C_{23}}}{Q} = \frac{1}{C_{12}} + \frac{1}{C_{23}}$$

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In its general form, Eq. 11 may be written as Eq. 12:

$$\frac{I}{C_T} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3} + \ldots + \frac{1}{C_n} \quad (12)$$

In Eq. 12, the subscript T denotes the total conductance of a number of conductances C_1, C_2, C_3 ... C_n connected in series.

In the case of only two conductances connected in series (Fig. 4b), Eq. 12 should be written in the form of Eq. 13:

$$C_T = \frac{C_1 \times C_2}{C_1 + C_2} \quad (13)$$

This case is analogous to the special case of two electrical resistors connected in parallel.

Gas Conductance for Pipes or Tubes Connected in Parallel

Figure 5 shows two lengths of pipe connected in parallel. In this connection, the total gas load (throughput) flowing from the vacuum chamber divides between the two pipes as shown. The division is not equal but depends on the conductance of each component. From Eq. 6, the gas load in each parallel pipe may be written in the form of Eq. 14:

$$Q_A = C_A \times \Delta P \quad (14)$$

and

$$Q_b = C_b \times \Delta P$$

The total conductance between points 1 and 2 is

$$C_{12} = \frac{Q_a + Q_b}{\Delta P} \quad (15)$$

Substituting from Eq. 14, Eq. 15 gives:

$$C_{12} = \frac{C_a \Delta P + C_b \Delta P}{\Delta P} \quad (16)$$

Simplifying, Eq. 16 becomes:

$$C_{12} = C_a + C_b \quad (17)$$

In its general form, the total conductance for a number of pipes connected in parallel is equal to the sum of the individual conductances, as given by Eq. 18:

$$C_T = C_1 + C_2 + C_3 + \ldots + C_n \quad (18)$$

FIGURE 4. Electrical relationship of two conductances in series: (a) pipe conductances in series; (b) connection of two electrical resistances.

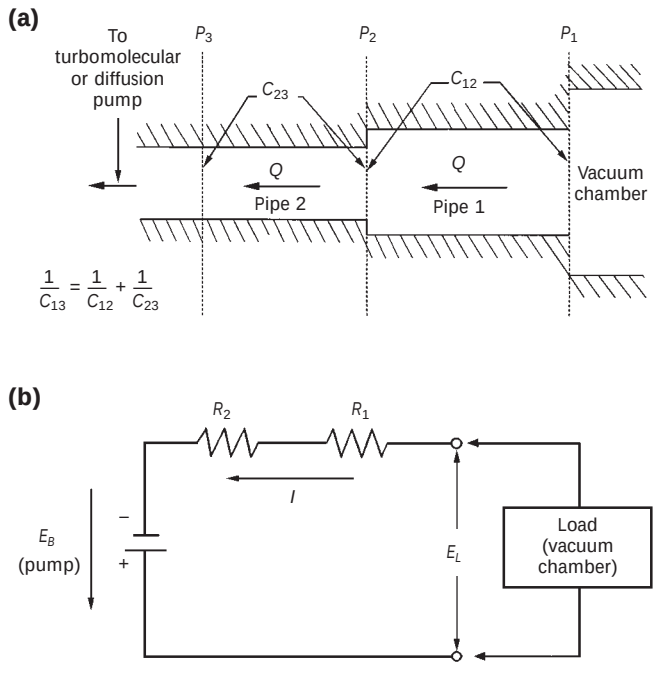
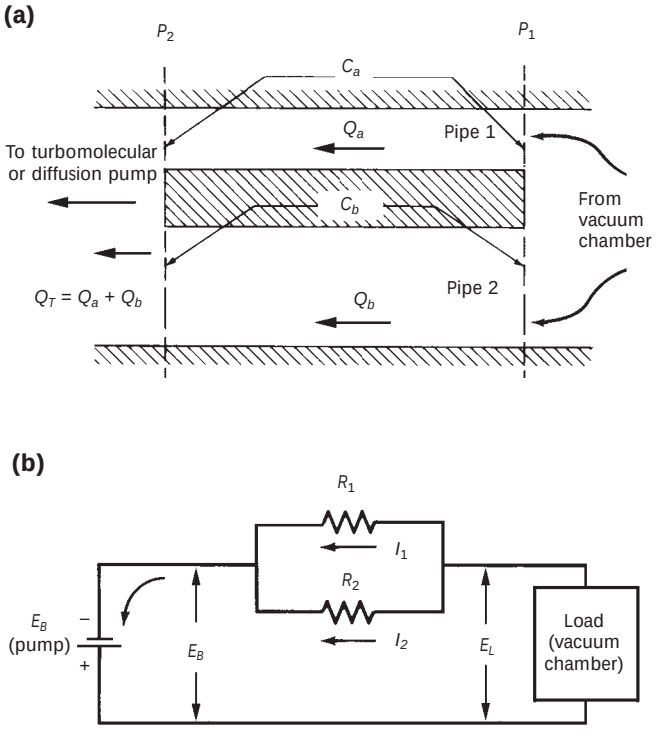


FIGURE 5. Electrical analogy of two gas conductances in parallel: (a) connection of two parallel gas conductances; (b) electrical circuit analogous to two gas conductances in parallel.



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Pumping Speed

In operating a vacuum system, there is an interest in how fast gases are removed from the system. The rate of removal of gases is measured by pumping speed *S*. From Eq. 4, pumping speed is defined as the ratio of the throughput *Q* to the pressure *P* at the point in the system. Mathematically, this relation is given by Eq. 19 ($m^3 \cdot s^{-1}$):

$$\textbf{(19)} \quad S = \frac{Q}{P}$$

If the inlet to a vacuum pump were connected directly to a vacuum vessel, then the pumping speed at the vessel would be the same as that at the pump inlet. Because it is physically impossible to join the pump and vessel without introducing a connector the pumping speed at the vessel will be lower than that at the pump. Pumping speed loss depends on the magnitude of the conductance that causes a loss in pressure or creates differential pressure between pumps and vessel. Figure 6 is used to help establish a relationship between the net pumping speed at the vacuum chamber, pumping speed at the port of a vacuum pump and the conductance between them. Although the connection is shown as a pipe in Fig. 6, it could be a combination of any number of vacuum components, each contributing a value of conductance. The flow of gas is from the chamber to the pump. From Eq. 5, the pressure drop is given by Eq. 20:

$$\textbf{(20)} \quad \Delta P = P_c - P_p = \frac{Q}{C}$$

In Eq. 20, the subscripts *c* and *p* refer to the chamber and pump, respectively. The throughput *Q* is the product of the speed *S* and pressure *P* where each is measured at the same point, such as at the pump or chamber. Throughput at the pump is therefore expressed as Eq. 21:

$$\textbf{(21)} \quad Q_p = S_p P_p$$

and pressure as:

$$P_p = \frac{Q}{S_p}$$

At the chamber being evacuated, throughput is expressed as:

$$\textbf{(22)} \quad Q_c = S_c P_c$$

and pressure as:

$$P_c = \frac{Q}{S_c}$$

Substituting Eqs. 21 and 22 into Eq. 20 results in the relation of Eq. 23:

$$\textbf{(23)} \quad \frac{Q}{S_c} - \frac{Q}{S_p} = \frac{Q}{C}$$

Rearranging terms:

$$\textbf{(24)} \quad \frac{Q}{S_c} = \frac{Q}{S_p} + \frac{Q}{C}$$

and multiplying by $1/Q$:

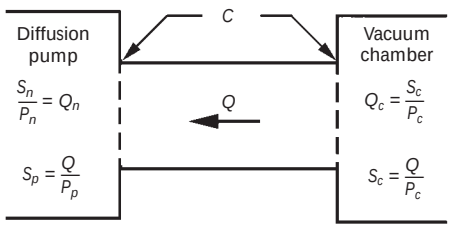
$$\textbf{(25)} \quad \frac{1}{S_c} = \frac{1}{S_p} + \frac{1}{C}$$

In the general case, the net speed S_n at any point in a vacuum system is related to the pump speed S_p and the total conductance C_t between that system point and the vacuum pump by Eq. 26:

$$\textbf{(26)} \quad \frac{1}{S_n} = \frac{1}{S_p} + \frac{1}{C_t}$$

Analysis of Eq. 25 shows that, except for the case of an infinite conductance (zero resistance), the net speed will always be less than the pump speed. How much less depends on the value of the tube conductance.

FIGURE 6. Net pumping speed relationship applicable to conductance *C* between vacuum pump and chamber being evacuated. Pressure at vacuum chamber (inlet to conductance *C*) is P_c, and pumping speed $S_c = Q/P_c$. Pressure at vacuum pump (outlet of conductance *C*) is P_p and pumping speed is $S_p = Q/P_p$. Because vacuum pump pressure P_p is lower than chamber pressure P_c, whereas *Q* is the same at each end of conductance *C*, the pumping speed is different at inlet and outlet of *C*.



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PART 2. Principles of Operation of Vacuum Systems and Components

Introduction to Vacuum Pumping

To attain vacuum in a container, some means of pumping must be used. A pump cannot reach into the system and extract molecules, but must simply wait for molecules to wander through the natural exit in the container and into the pump for isolation and discharge. When the pressure in the vacuum system becomes so low that there is no longer a net movement of molecules into the pump, the base pressure of the system has been reached under those conditions. How low this ultimate pressure is will be determined by conditions such as (1) the leak tightness of the vacuum system, (2) the nature and condition of materials within the vacuum system that might cause outgassing and (3) the operating characteristics of the pumps in combination with the specific vacuum system.

Molecular Conditions Limiting Rates of Pumping of Vacuum Systems

To evacuate a closed system initially at atmospheric pressure, numerous gaseous molecules must be removed from within the closed system. The fewer the gas molecules remaining, the lower the absolute pressure of gas within the system. However, the common concept that vacuum pumps draw out the air like a vacuum cleaner is wrong. Molecules in the gaseous phase are in constant motion and collide with each other and with the walls of the container. A certain number *R* of molecules strike each unit area of the container wall per unit time. The number of gaseous molecules striking a unit area (square meter) of the container wall per unit time (second) is given by Eq. 27:

$$R = \left(2.63 \times 10^{24}\right) \frac{P}{\sqrt{M\,T}} \quad (27)$$

where *R* is rate of molecular impact with the wall in molecules per square meter per second; *P* is absolute pressure in evacuated chamber (pascal); *M* is the molecular weight of gaseous particles, in unified atomic mass unit (u); and *T* is the absolute temperature of gas within the evacuated container (kelvin). A similar expression can be given for a mass of gas (kilogram) striking a unit area (square meter) of the evacuated container wall per unit time (second):

$$R' = \left(43.8 \times 10^{-4}\right) \frac{P}{\sqrt{M\,T}}$$

where *R* is rate of gas mass impact with wall ($kg \cdot m^{-2} \cdot s^{-2}$); *P* is absolute pressure within evacuated chamber (pascal); *M* is molecular weight of gaseous particles, in unified atomic mass unit (u); and *T* is absolute temperature of gas within evacuated container (kelvin).

If a hole of unit area were cut through the container wall, those gas molecules that would have collided with the container wall in the area of the hole and rebounded within the container will now pass through the hole at the rate given by Eq. 27a. If these escaping gas molecules are now prevented from reentering the container through that hole, the net effect would be that of reducing the number of molecules within the container and thus reducing the internal gas pressure. This is the basic concept of vacuum pumping, namely, to provide a natural exit for gas molecules and to isolate the escaping gas molecules so that they cannot reenter the container being evacuated. The vacuum pump cannot extract gas molecules from within the evacuated container; it merely aids those molecules that pass through the hole in the wall to naturally escape being reinjected into the vacuum container through that same hole. It is impossible to pump any gas out of an evacuated container at any rate faster than that at which internal gas molecules strike the hole area by their random kinetic motions.

Example of Limitations on Vacuum Pumping of Gaseous Nitrogen

For gaseous nitrogen with a molecular weight *M* = 28 at room temperature, 298 K (25 °C or 77 °F) and atmospheric pressure, 101 kPa (1 atm), Eq. 27a indicates that the number of molecules striking each square meter of container wall during each second would be calculated as:

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$$R = \frac{\left(2.63 \times 10^{24}\right) \times 101\,000}{\sqrt{28 \times 298}}$$

$$= 2.92 \times 10^{27}$$

Similarly, if the mass (kilogram) of nitrogen gas striking the unit area (square meter) per second were to be determined, Eq. 27b indicates that this mass would be:

$$R' = \left(43.8 \times 10^{-4}\right) \times 101\,000 \times \sqrt{\frac{28}{298}}$$

$$= 136$$

The volume flow rate corresponding to the above mass flow rate would be equal to $136 \times 0.8714 = 118\ m^3 \cdot s^{-1} \cdot m^{-2}$ ($7.08 \times 10^3\ ft^3 \cdot min^{-1} \cdot ft^2$).

For the case of molecular nitrogen (N_2) at 298 K (77 °F) and atmospheric pressure, the conversion factor is $0.8714\ m^3 \cdot kg^{-1}$. Thus, for gaseous nitrogen molecules (each of which contains two nitrogen atoms), the volumetric rate of exit of gas through a hole in the evacuated container would be $118\ m^3 \cdot s^{-1} \cdot m^2$ or $11.8\ L \cdot s^{-1} \cdot cm^2$ ($7.08 \times 10^3\ ft^3 \cdot min^{-1} \cdot ft^2$). This is the maximum rate at which nitrogen can be pumped from a container whose internal pressure was 101 kPa (1 atm). As system pressure decreases, the molecular or mass rate of pumping drops proportionally.

Conditions Limiting Rate of Pressure Reduction by Pumping

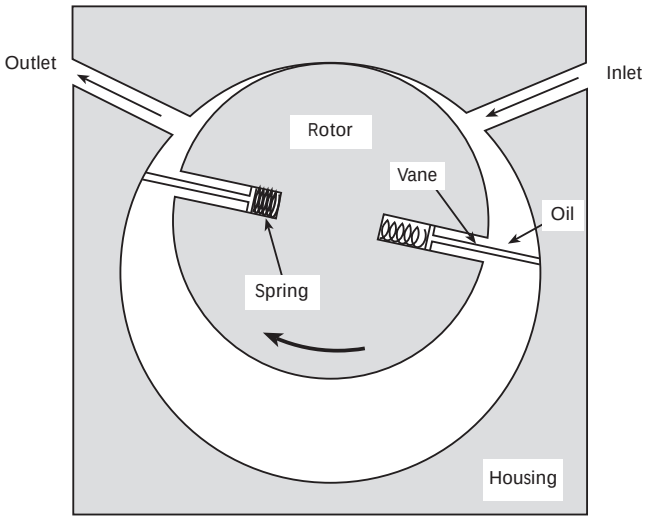
Pumping times greater than expected for reduction of pressure to desired levels can result from system contamination or system leaks. System contamination can be caused by processing of so-called dirty work materials or by allowing excessive time without thorough cleaning of the vacuum equipment. Contamination of this type results in many layers of various compounds, organic or otherwise, which build up on interior surfaces. The contaminated surfaces then outgas at such rates that the pump capacity may be unable to reduce pressure to desired levels within acceptable pumping times. Water vapor adsorbed to chamber walls is a common contaminant. Dirty walls are subject to more severe water adsorption. It is also possible for mechanical pump oil to become contaminated, which alone can cause poor pumping characteristics.

If pumping is slowed by system leaks, thorough mass spectrometer leak detection tests inspection should be performed and leaks repaired.

Operation of Mechanical Pumps for Vacuum Systems

The mechanical pump is an essential component used in vacuum systems to evacuate a chamber from atmospheric pressure to about 0.1 Pa (10^{-3} torr) absolute pressure. Of the various types of mechanical pumps, the rotary oil sealed vacuum pump shown in Fig. 7 is most common. The pump consists of a stationary housing, an eccentrically mounted rotor with two spring loaded vanes, an inlet port and a discharge port. Air enters the pump from the vacuum chamber through the inlet port. This air is trapped, compressed and ejected into the atmosphere through the discharge port by means of the rotor arrangement. Sealing of the eccentric rotor vacuum pump is done by an oil film between the two sliding spring loaded vanes that make contact between the rotor and the housing. Oil is used as the pump sealant. Close tolerances must be maintained to prevent leaks and by passing of gases. Consequently, care must be taken to prevent solid particles from entering the pump. Each rotation of the rotor discharges two volumes; each volume is a certain percentage of the volume to be evacuated. This would indicate that even a perfect pump could never evacuate to a vacuum linearly but could only approach this condition as an exponential function of pumping time.

FIGURE 7. Rotary mechanical vacuum pump with eccentric rotor and spring loaded vanes. Pump oil provides a sealing film at points of vane contact with stator housing.



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At low chamber pressures, air may leak back into the evacuated volume. This can be minimized by putting two pumps in series so that the discharge from the first pump chamber is not directly to atmosphere but to some intermediate pressure maintained by the second (or backing) pump.

Pump Oil Used in Rotary Vacuum Pumps

The operating fluid in any type of pump is called the pump fluid or pump oil. With rotary pumps, normally a good quality light petroleum oil, with the high vapor pressure factions removed, is used to provide pump sealing and lubrication between the rotor vanes and stator housing. The oil for lubricating and sealing is contained in an oil reservoir. The arrangement of the reservoir differs from manufacturer to manufacturer. In some small pumps, the pump chamber is actually immersed in the reservoir, whereas for the larger pumps the reservoir is usually separated from the pump chamber, often being mounted above the pump itself.

Prevention of Condensate Contamination of Pump Oils

Contamination of pump oil is one of the main difficulties with rotary pumps. As the gases and vapors are compressed, the vapors will tend to condense and contaminate the oil. Degassing of vapors from pump oil can limit the ultimate vacuum attainable. Pumps are available with a gas ballast valve incorporated, which minimizes the condensation of vapors in the pump oil. The gas ballast valve is a small valve that can be opened manually to admit a controlled amount of air to the pump cylinder during part of the compression cycle. This will dilute the vapors to the point where they do not condense during compression. The violent agitation of the oil by the additional air rushing through the pump causes reevaporation and exhaust of water that may have been pumped from the vacuum system in vapor form and condensed in the pump oil. To effect the removal of moisture when the surrounding air is saturated with moisture, connect a dry nitrogen gas supply to the gas ballast. Be careful to select a nitrogen flow rate and pressure that will not apply overpressure to the casing of the pump. The extent of use of the ballast valve is determined by the amount of such vapors handled by the pump. In normal high vacuum service, the ballast valve is usually kept closed because there is usually very little water vapor present. The minimum pressure obtainable is also slightly higher with the ballast valve open. (Because of the higher pressure in the chamber during compression with the ballast valve open, there is more leakage back into the vacuum system.) Some pump manufacturers recommend operating the pumps with the ballast valve open once each week for about 20 min to drive out any water vapor that may have accumulated in the pump oil.

Ultimate Pressure Attainable in Rotary Pump Vacuum Systems

The limiting absolute pressure approached in a vacuum system, after sufficient pumping time establishes that further reductions in pressure will be negligible, is called the ultimate pressure. The range of ultimate pressures of commercial rotary vacuum pumps extends from about 3 mPa to 1 kPa (20 µtorr to 5 torr). The low pressure of 3 mPa is reached only under the most ideal conditions. The ultimate pressure will be determined by:

1. outgassing of the pump,
2. the seal between rotor and stator,
3. contamination of pump oil and
4. the vapor pressure of the oil used.

A high vapor pressure pump oil will evaporate at a greater rate, which will create gas loads that saturate the pump and limit the ultimate pressure attainable.

A disadvantage of any oil sealed and lubricated pump is the backstreaming of oil vapors from the pump inlet when inlet pressures drop below or approach 70 Pa (0.1 torr). This has become a major concern to many industries, such as semiconductor producers, for whom backstreaming causes contamination of their products with oil vapors. As a result, several new pump designs classified as *dry* or relatively free of this problem have been available since the early 1980s. Two of these are called *scroll* pumps and *hook and claw* pumps.

Rotary Dry Mechanical Pumps

Unlike the rotary vane pump, which requires a low vapor pressure oil to lubricate and seal the internal surfaces, two commonly used pumps are designed with very small clearances between the moving and fixed surfaces and no need for oil. As a result, the contamination caused by vapors entering the evacuated space at low pump inlet pressures is

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eliminated. A slight disadvantage is that these pumps cannot quite reach the same low pressure as the lubricated pumps. Ultimate pressures for dry pumps is in the range of 2 to 3 mPa (20 to 30 µtorr).

Rotary Scroll Mechanical Pumps

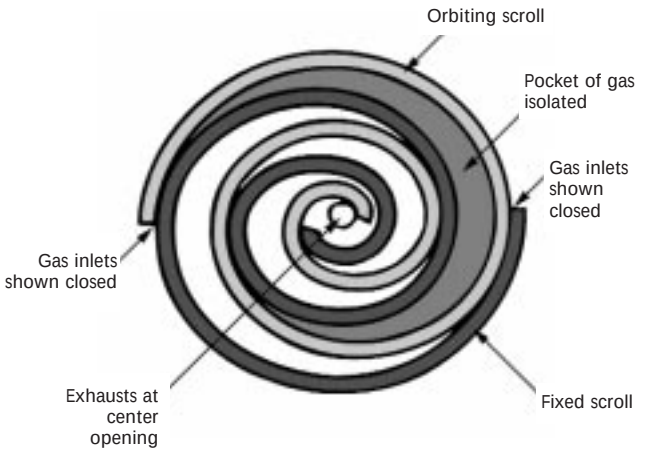
A scroll is a free standing involute spiral with a solid base on one side. A scroll set, the fundamental element of a scroll vacuum pump, is made up of two identical right and left hand involutes. When assembled, one scroll is indexed 180 degrees with respect to the other, to allow the scrolls to mesh (Fig. 8).

In operation, one scroll is fixed and the other is attached to an eccentric, driven by an electric motor. The pump inlet is at the periphery of the scrolls. As the moving scroll orbits (but does not rotate) about the fixed scroll, the entering gas is trapped in two diametrically opposed, crescent shaped pockets bounded by the involutes and base plates of both scrolls. The pockets shrink as they follow the involute spiral toward the center, compressing the gas. The compressed gas exhausts to atmosphere through the discharge port at the center of the fixed scroll.

Rotary Claw Mechanical Pumps

The hook-and-claw mechanism consists of several inline stages. The claw devices do not make contact with each other or with the chamber walls, obviating oils. Each rotation of a claw pair consists of three cycles: a start cycle, compression cycle and a finish cycle. The two claws, which divide the pump chamber, turn in opposite directions and, in so doing, open and close the intake and exhaust slots through which the gases pass. During pumping, gas is drawn in one side of the claws and compressed on the other as the claws rotate. During rotation, the right claw opens the intake slot, allowing gas to be drawn into the chamber. Simultaneously, the left claw opens the exhaust slot letting compressed gas escape. On completion of the compression cycle, the claws pass through a neutral position and cycle begins again.

FIGURE 8. Position of orbiting scroll shown before compression cycle.



Pumping Speeds of Rotary Mechanical Vacuum Pumps

Apart from the ultimate absolute pressure that can be achieved by an particular pump, there is an interest in how fast the pump can reduce the pressure in a vacuum system to the operating level. Manufacturers normally specify the pumping speeds of their mechanical pumps at atmospheric pressure. In general, rotary pumps start pumping at atmospheric pressure and, as the pressure is reduced, the pump becomes less efficient. It then is pumping the same volume, but at lower pressure. Eventually, the pumping speed becomes zero at the ultimate minimum pressure.

Figure 9 is a plot of pressure as a function of pumping speed for a 400 $L \cdot min^{-1}$ (15 $ft^3 \cdot min^{-1}$) mechanical pump. It is seen that at atmospheric pressure, the pump is rated at 400 $L \cdot min^{-1}$; at 0.1 Pa (1 mtorr), the pumping speed is 200 $L \cdot min^{-1}$ at 0.01 Pa (0.1 mtorr), the pumping speed is 16.7 $L \cdot s^{-1}$ (35 $ft^3 \cdot min^{-1}$). The pump speed reduces to zero at 10^{-3} Pa (10 µtorr), the ultimate pressure attained by this pump. At this point the gas handling capacity has been saturated by the gas load from the pump, thereby reducing its effective pumping speed to zero.

Blower Pump or Booster Pump

The blower or booster pump (Fig. 10) is a high throughput, low compression pump. This pump is usually used on systems where a large volume of gas must be pumped. It is also used with a mechanical pump to serve as the forepump for large diffusion pumps, turbomolecular pumps or even other blower pumps.

The pump consists of two figure eight shaped rotors or lobes mounted axially on parallel shafts, as shown in the drawing below. These rotors are synchronized by gears to prevent physical contact and damage and rotate in opposite directions. This rapidly displaces gas from the inlet to the outlet.

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How the Pump Works

These rotors are designed so that, while spinning, they approach each other and the housing within several thousandths of an inch. (See Fig. 10.) Rotor speeds vary from 40 to 60 s^{-1} (2500 to 3500 rotations per minute). Because of the high speeds and close tolerances of the rotating lobes, booster pumps are usually not started until roughing pressures of about 1.3 kPa (10 torr) have been reached. The typical blower *windmills* at atmospheric pressure, producing much heat and very little pumping action.

Blower or booster pumps are most useful in the 0.1 to 0.01 Pa (1.0 to 0.1 mtorr) pressure range. They are always backed by a mechanical pump as a result. Operating at high pressures will cause heating and expansion of the lobes. This can result in damage to the pump. No oil is used to seal the gap between stator and rotor. Oil is used in the forevacuum section of the pump to lubricate the gears and bearings located there.

Vacuum System Operation

Operating procedure consists of turning the mechanical pump on, then the blower (Fig. 11). Usually the mechanical pump has lowered the pressure sufficiently for the blower to begin pumping by the time the blower has reached operating speed. A bypass valve around the blower is sometimes used for high pressure roughing.

Blowers are commonly used where large volumes of gas need to be pumped. They are used when the lowest pressure needed is 10^{-2} to 10^{-3} Pa (75 to 7.5 µtorr). They also are used to help the mechanical forepump or backing pump maintain a low pressure and help reduce the possibility of oil backstreaming.

Turbomolecular Vacuum Pumps

The turbomolecular pump serves as an alternative to the diffusion pump and must also be backed by a forepump. Its

FIGURE 9. Mechanical pump speed as a function of gas pressure for a pump rated at 6.7 $L \cdot s^{-1}$ (14 $ft^3 \cdot min^{-1}$) at atmospheric pressure.

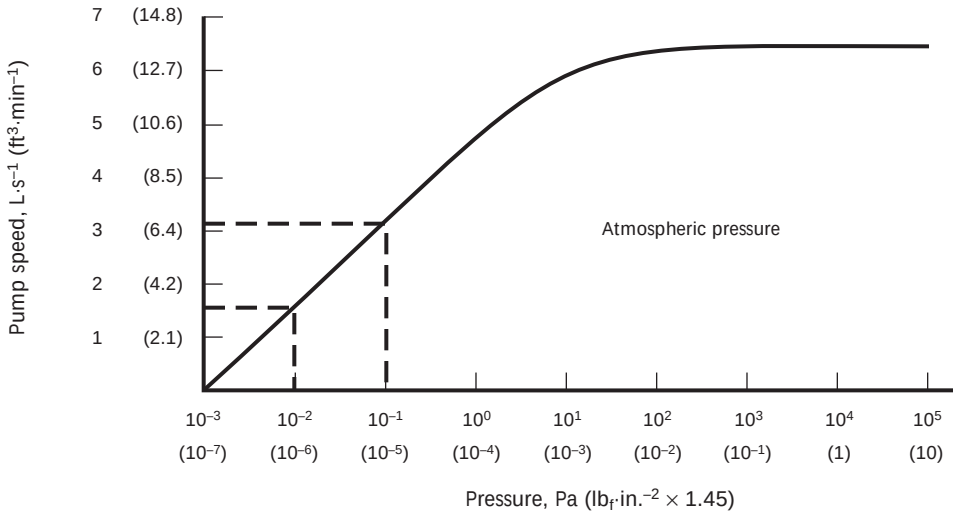
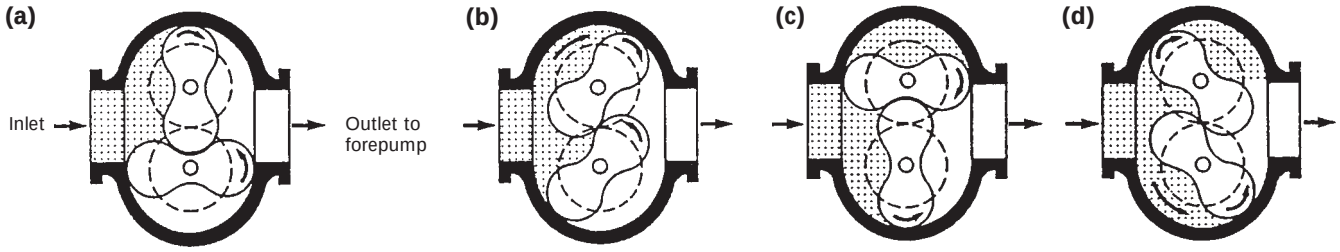


FIGURE 10. Blower pump operation: (a) at beginning of cycle; (b) after eighth of cycle; (c) after fourth of cycle; (d) after three eighths of cycle.



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principle advantage over the diffusion pump is that it provides an essentially vapor free vacuum without baffles or cold traps. Thus, for a system where the back streaming of vapor from a diffusion pump is undesirable or intolerable, a turbomolecular pump could be used. Its main disadvantage is that it has high speed rotating parts whereas the diffusion pump has not moving parts. It also requires air gap tolerance on the order of 2 to 5 µm (8×10^{-5} to 2×10^{-4} in.) between the high speed rotor and grooves in the stator. As with a diffusion pump, a molecular pump cannot operate at pressures above 13 to 1.3 Pa (100 to 10 mtorr) and must be backed by a mechanical forepump.

A turbomolecular pump (see Fig. 12) is a mechanical vacuum pump that creates a gas flow toward a suitable forepump by imparting momentum or motion to gas molecules by means of a rapidly rotating rotor with successive rings with inclined blades. These blades rotate with circumferential speeds comparable to the thermal motion of the molecules (speeds of 100 to 700 $m \cdot s^{-1}$ or 330 to 2300 $ft \cdot s^{-1}$). Some molecules are struck by the rotor blades and rebound in a favorable axial direction toward the stator blades. The molecules rebound from these stator blades in a direction favorable for their being impelled by the next stage rotor blades and so on as the process is repeated through all successive stages of rotor and stator blades. The series of impacts statistically favor motion through the turbine stages toward the discharge port and constitute a pumping action with a very high compression ratio.

The seal between the individual stages is achieved by very narrow air gaps. The dimensions of the grooves at the inlet port must be such that the molecules have a good chance of hitting the walls of the groove or the blades without making numerous collisions with other gas molecules (see Fig. 12). As the gas is compressed while passing through successive stages of the turbine, it is necessary to decrease the dimensions of the air passages to keep them comparable with the mean free path of the molecules. The system must already be evacuated by a forepump before a turbomolecular pump can start pumping. It can achieve pressures as low as 1.0 to 0.1 µPa (10 to 1.0 ntorr). Pumping speeds for air vary from about 70 to 9000 $L \cdot s^{-1}$ (1.5×10^2 to 1.9×10^4 $ft^3 \cdot min^{-1}$), depending on the size of turbomolecular pump selected. Pumping speeds for hydrogen and for helium vary only slightly from those for air whereas the exhaust pressure is in the range from 1.3 Pa to 1.3 mPa (10 mtorr to 10 µtorr). Higher exhaust pressures are achieved in compound turbomolecular

FIGURE 11. Vacuum system with blower pump.

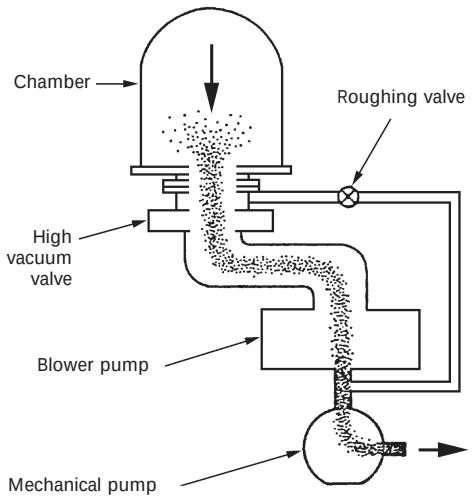
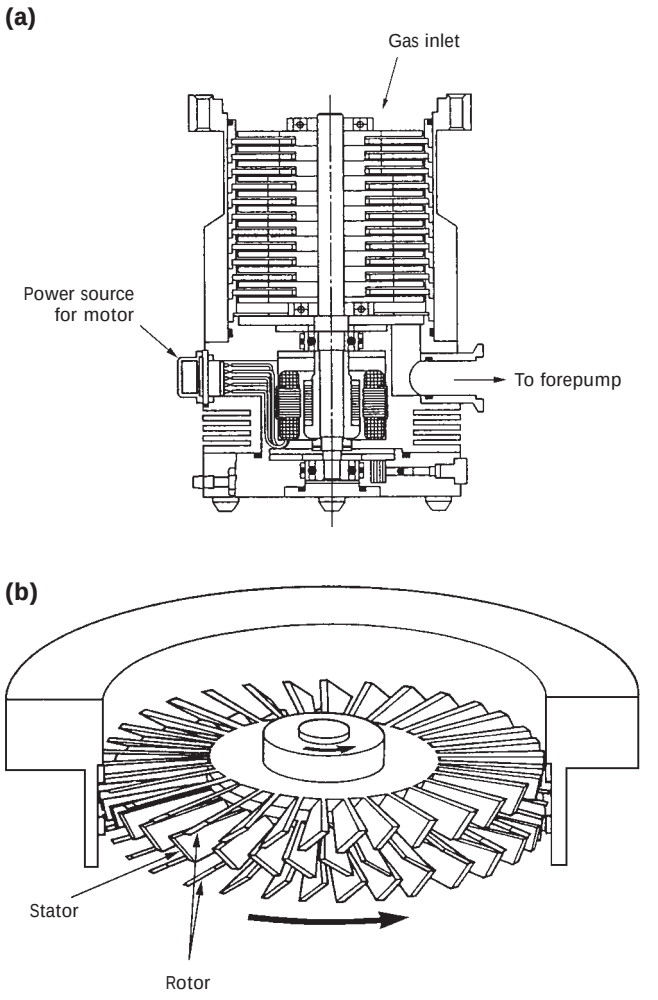


FIGURE 12. Turbomolecular pump: (a) schematic; (b) inlet port.



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pumps. These follow the turbomolecular stages with one or several molecular drag stages, which further compress the gas through the effects of viscosity.

Operation of Vapor or Diffusion Pumps for Vacuum Systems

Although mechanical rotary pumps sometimes attain pressure below 0.1 Pa (10^{-3} torr), they are generally used in the 100 to 0.1 Pa range. To obtain pressures well below 0.1 Pa, the vapor pump was at one time the most commonly used. However, in the 1990s it was largely replaced by turbomolecular pumps because of the backstreaming of vapors. The principle of operation of vapor pumps is entirely different from that of a rotary oil sealed pump, where the gases and vapors are compressed by a rotating mechanical member and exhausted to the atmosphere.

The vapor pump, or diffusion pump, operates in the molecular flow region. The basic principle involved is shown in Fig. 13.

The pump works by heating the pump fluid to its boiling point. The vapors travel upward inside the jet assembly and exit through the jet nozzles. In fact, they are accelerated downward through the jet nozzles. The vapor molecules travel very fast and can reach supersonic speeds.

These vapor streams are directed toward the outer walls of the pump. The walls are typically cooled by water. When the vapor hits the cooled walls, it condenses back into a fluid. This fluid then flows downward into the pump boiler for reboiling.

The actual pumping of gases happens when the large, heavy, high speed oil vapor molecules hit gas molecules. The gas molecules are knocked downward and compressed by the movement of the vapor jet stream.

The gas molecules are thereby compressed in several stages to higher pressures. They are finally pumped away through the foreline by the mechanical pump (Fig. 14). When the oil drops to the bottom of the pump, it is reboiled and the cycle repeats.

Vacuum Limitations of Vapor Diffusion Pumps

A diffusion pump (Fig. 14) cannot operate at pressures above 0.1 Pa (1 mtorr) because the oil vapor jets cannot form in the viscous flow region. Therefore, the pump must start pumping in a chamber that is already under vacuum (such as that attained with a rotary mechanical forepump). Oil is the most frequently used diffusion pump fluid because of its low vapor pressure at room temperature. Oil has a fairly steep curve relating its pressure to temperature. This is necessary for proper operation of the pump boiler. The lowest attainable pressure of the diffusion pump is determined in part by

FIGURE 13. Principle of operation of high vacuum vapor pump. Vapor forced through a narrow opening (nozzle) attains a high speed and is directed at a downward angle. Molecules of gas or vapor that wander along a path toward the jet stream will be struck by vapor molecules. The gas molecule B has diffused into the path of the jet stream where it is struck by the vapor molecule A. Molecule B is given a generally downward motion.

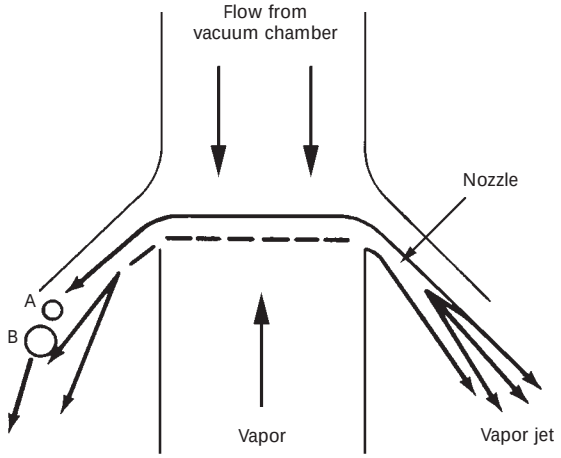
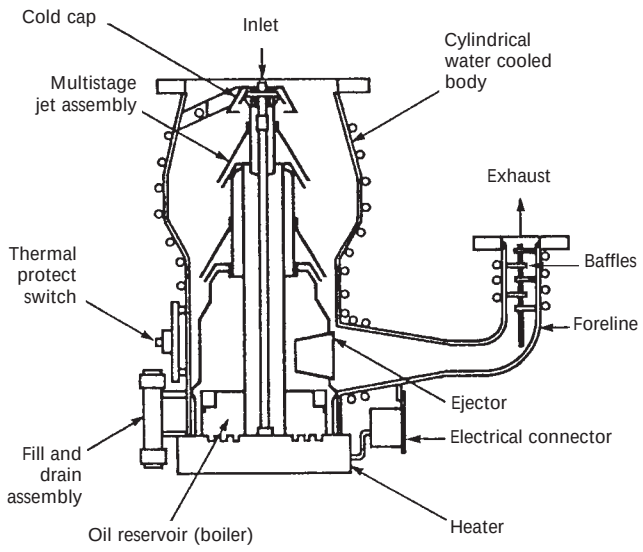


FIGURE 14. Construction of three-stage high vacuum vapor pump.



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the vapor pressure of the oil at the temperature of the available cooling water. Oils specified by pump manufacturers have vapor pressures, under these conditions, of about 0.1 μPa (1 ntorr). The popularity of the diffusion pump is due to its wide range of operation, low cost, reliability and lack of moving parts. The pump heaters are usually mounted from the outside and can be replaced during operation.

Should the diffusion pump be suddenly exposed to a burst of atmospheric pressure, the oil jet stream would collapse, thereby destroying the pumping capability of the vapor pumps and possibly acting to crack the oil. The term cracked oil refers to a decomposition of pump oil due to exposure to oxygen in the atmosphere while at or near the boiling point of the oil. Some fluids are less susceptible to cracking than are other diffusion pump fluids.

Operation of Baffles and Traps in Vacuum Pumping Systems

One of the objections to diffusion pumps has been the possibility of contaminating the vacuum chamber work area with the pump fluid. By providing suitable traps and baffles between the pump and the vacuum chamber, back diffusion of oil and oil vapor can be minimized and condensable vapors from the chamber may be trapped. As a general rule, the pumping speed of the system goes down as the trapping efficiency of baffles and traps goes up, due to decreased conductance.

The baffle or trap should normally be kept as cold as possible. However, the temperature of surfaces of the first baffling state above a pump should be cool enough to condense the oil vapors, but not so cold as to freeze the pump oil and prevent it from flowing back into the pump.

Characteristics Desired in Diffusion Pump Baffles

A baffle is simply a cool surface that is placed above the diffusion pump in the path of gas flow. This baffle is of metal with good thermal conductivity that keeps its surface at a uniform temperature. The refrigerant, usually cold water, is passed through tubing that is brazed to the baffle. A baffle should also be optically dense, that is, there should be no line of sight through it, to avoid back flow of molecules in molecular flow. Fig. 15 shows some typical designs of baffles. Because a baffle restricts the flow of gas, it should not sacrifice high conductance because that would impair the net pumping speed of the system.

Operation of Cold Traps in Vacuum Pumping Systems

A cold trap placed above the baffle ensures that those few oil molecules that may get by the baffle will not get to the vacuum chamber. A cold trap, therefore, stops back migration of pump oil vapors. It is also very effective as a cryogenic pump for pumping condensable vapors such as water vapor, the chief offender in most systems, as well as for grease vapors and other undesired contaminants.

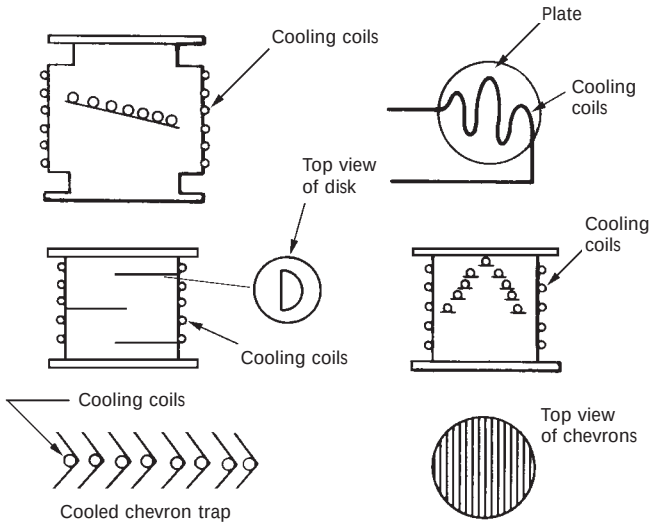
As a cryogenic pump, the cold trap reduces system pressure by taking molecules out of the gas or vapor phase and trapping them on its surface. These molecules are not pumped out of the vacuum system and discharged to atmosphere. The most common techniques used to obtain low temperatures for cold traps are mechanical refrigeration, dry ice and liquid nitrogen.

Some common forms of optically dense chevron and cold traps are shown in Fig. 16, which also shows thimble type traps used in mass spectrometer leak detectors. The reservoir is filled with liquid nitrogen through the filler tube. Use of liquid nitrogen requires that the thimble type trap be kept essentially in a vertical position.

Characteristics Desired in Vacuum Valves

Vacuum valves must (1) be free from leakage, (2) offer minimum flow

FIGURE 15. Typical baffle designs used in oil diffusion vacuum pump systems.



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resistance and (3) contain materials that do not outgas. The biggest problem in making leaktight valve is in sealing the operating shaft. Two types of valves that accomplish efficient sealing are the bellows sealed and diaphragm valves (Fig. 17). Usually preferred are brass or stainless steel bellows, more movement being obtained with brass. The bellows is brazed to the cover (bonnet) and dish, as shown in Fig. 17a. Figure 17b shows a valve using a diaphragm that can be a metal or elastomer. Compared to metal diaphragms, an elastomer has considerable flexibility but also has the disadvantages of outgassing and permeability to various gases. On the other hand, metal diaphragms are not as elastic but have better outgassing and permeability characteristics.

Precautions in Disassembly of Bellows Sealed Valves

Always open bellows valve before removing the stem assembly to prevent cracking the bellows. Never completely extend bellows when out of the valve.

Operation of Capture Vacuum Pump

Unlike the previously described pumps, which compress and exhaust gas either to atmosphere or into an attached forepump, two commonly used pumps collect and store gases in the pump body until eventually being released to atmosphere by a process called regeneration (for the cryopump) or until the pump is rebuilt as in the ion pump. These pumps are the mechanical *cryopump* and the sputter *ion pump*.

FIGURE 16. Cold traps used in vacuum pumping systems to condense vapor molecules: (a) combination baffle and trap with optically dense chevrons; (b) thimble trap used in leak detectors.

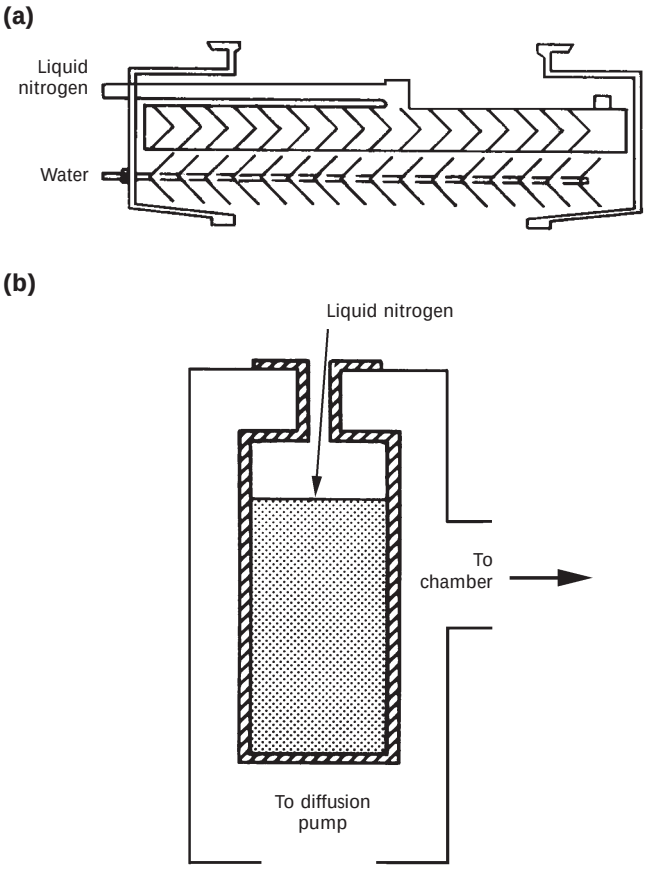
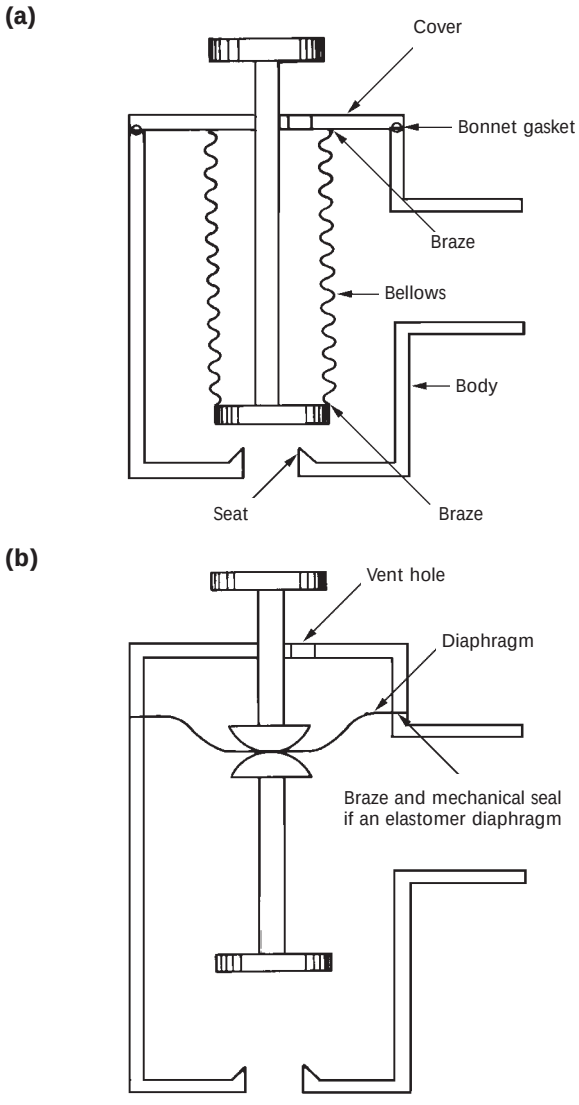


FIGURE 17. Operating principles of vacuum valves: (a) bellows sealed valve; (b) diaphragm valve.



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Operation of Cryopump

The cryopump is unique in that it pumps by getting the gases so cold that they freeze and are stored, or captured, in the pump. It is extremely clean, using no oil and having no moving parts in vacuum. It also has a very high throughput and is used in the high vacuum range in industrial applications where hydrocarbons cannot be tolerated.

A cryopump (Fig. 18)[2] is made up of two main components: a gaseous helium compressor and a pump consisting of an expander, cold head (chilled surfaces) and the pump body. These two components are connected by flexible hoses to form a closed loop refrigeration system. Gaseous helium is circulated between the compressor and expander.

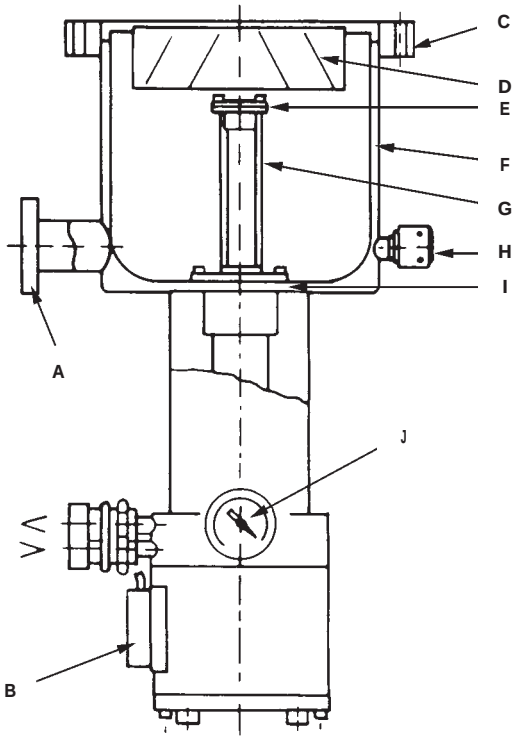
The pump module consists of the expander module, the first and second stage cryoarrays, the pump body, second stage temperature monitors and a pressure relief valve. In the expander, high pressure helium is supplied by the compressor. This gas is expanded in two stages to produce cryogenic temperatures. The actual operating temperatures will vary, depending on the thermal and gas loads that are imposed. The first stage operates between 50 and 80 K (–370 and –315 °F) and the second stage between 10 and 20 K (–440 and –420 °F). The cryoarrays are the pumping surfaces, cooled by the expander, on which gases from the vacuum chamber are condensed or adsorbed.

In cryopump operation, helium is compressed and gives up its heat to the surrounding walls of the compressor. This heat is removed by water or air cooling. The cooled, compressed helium then goes to the pump cold head. The expander at the cold head valving system lets the helium expand. The expanded helium now absorbs heat from the cold head and baffle array. This chills the cold head and baffle array to about 12 K (–440 °F) and 70 K (–335 °F), respectively. These chilled surfaces pump gases from the vacuum chamber in two ways. The gases are either condensed or adsorbed on the arrays. That most gases will stick to a surface in an icelike state at less than 20 K (–420 °F) is very likely. At this temperature, the combination of partial pressures of most gases is about 10^{-9} Pa (10^{-11} torr) or lower.

Most gases are condensed on the first and second stage cryoarrays. The first stage array is cold enough to pump water vapor and carbon dioxide by cryocondensation. The colder second stage array pumps nitrogen, oxygen, argon and most other gases by cryocondensation, but is not cold enough to condense helium, hydrogen and neon. These three gases are pumped by the process called cryosorption; a surface related phenomenon: the greater the available surface area at cryogenic temperatures, the more likely that gas molecules will stick to it. Although most gases are frozen or condensed between 12 and 20 K (21 and 36 °R), helium, hydrogen and neon are still very actively in motion at these temperatures. If we did not remove them, their partial pressures would continue to rise, perhaps to a point where the total system pressure would be unacceptable.

To solve this problem, activated charcoal is attached to the bottom side of the second stage (coldest) cryoarray where it is less likely to adsorb the easier to pump condensible gases. This reserves the charcoal for the helium, hydrogen and neon which are trapped in the maze like structures and surfaces of the charcoal. This is similar to a sponge soaking up water vapor at room temperature. This process is called cryosorption.

FIGURE 18. Schematic of cryopump.



Legend
A = Forevacuum port
B = Power connection
C = Inlet flange
D = Baffle
E = Second cold stage
F = Radiation shield
G = Cryoplates
H = Relief valve
I = First cold stage
J = Vapor pressure thermometer

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Before chilling the cryoarrays, the pump volume must be rough pumped to remove most of the gas below a valve at the pump inlet. During chilling, when the second stage drops to less than 20 °K, the pump is ready for use. During use, the pump can absorb very large amounts of condensible gas, but the second stage charcoal eventually saturates, usually with hydrogen and must then be turned off to warm up the pump (regeneration). To speed up this process, dry nitrogen is applied to the purge tube, through a valve, which flushes the pump and expels the previously stored gas out through the pressure relief valve. When the second stage rises to room temperature, the pump is ready to be rough pumped and chilled again.

The cryopump is normally used in the pressure range of 10^{-1} to 10^{-6} Pa (10^{-3} to 10^{-8} torr) but when operated continually at the upper end of this range, the time between required regeneration cycles is proportionately shorter; i.e. — more downtime.

Operation of Ion Pump

The ion pump (Fig. 19)[2] is also a gas capture pump but is not designed to pump heavy gas loads. For this reason, it is not generally used alone in high production applications. It is more often used in research and analytical applications where there is no need to cycle the work chamber repeatedly and rapidly from atmosphere to vacuum.

Ion pumps are clean operating electronic devices which use no moving parts or oils within the vacuum pump. It is possible to achieve pressures in 10^{-9} Pa (10^{-11} torr) range with overnight bakeout of the system. The bakeout process drives residual gas off the system walls, which is then pumped by the ion pump. In research and analytical applications, the ion pump's cleanliness, bakeability, low power consumption, and long life make it the pump of choice for most ultrahigh vacuum uses. They are available in various sizes and variations, but only the simplest (diode) pump will be described here for purposes of brevity.

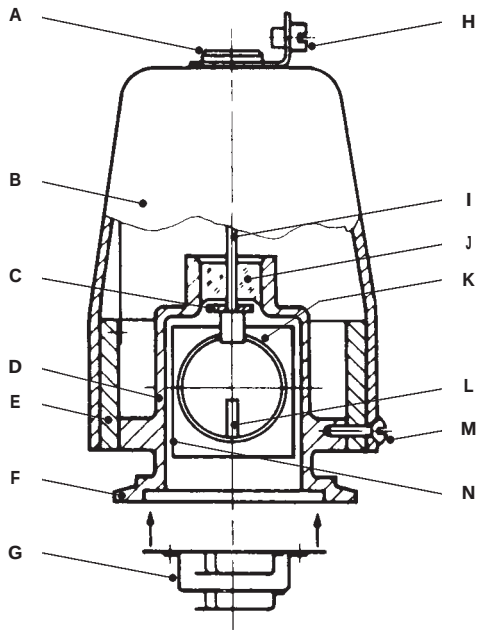
A stainless steel ion pump body contains a multicell anode assembly constructed of cylindrical parallel tubes spaced between two flat titanium cathodes. A very strong magnet is placed outside the pump body. After the ion pump is rough pumped to 1 Pa (10^{-2} torr) or less, a voltage of 5 to 7 kV direct current is applied between the cathodes and the anode assembly.

The magnetic field forces any free electrons within the anode into long helical paths instead of straight paths. This increases the probability of electron collision with molecules, as the electrons are accelerated toward the anode. This long path increases the probability of ionization and therefore the amount of useful pumping action that can be performed by the pump.

Because of the action of the magnetic field, the electrons do not easily come in contact with the anode. As a result, a cloud of electrons is formed within the anode space. This electron cloud becomes fairly stable during pump operation and is dense enough for the efficient ionization of gas molecules. The name for this process is *cold cathode discharge*. The positively charged ions, which are relatively heavy particles, are accelerated into the negatively charged titanium cathodes. This impact causes sputtering, or chipping away of the titanium cathode material.

Sputtered titanium deposits onto the internal structure of the anode. Then, when gas molecules come in contact with these clean titanium deposits, chemical

FIGURE 19. Section through a cold cathode ionization gage (Penning gage).



Legend
A = High voltage connection
B = Hood
C = Protective cap
D = Vacuum tight cast iron housing
E = Permanent magnet
F = Small flange connection
G = Baffle
H = Safety terminal
I = Leadthrough (anode lead)
J = Compressed glass-to-metal seal
K = Ring anode
L = Ignition pin
M = Fixing screw
N = Cathode plate (exchangeable)

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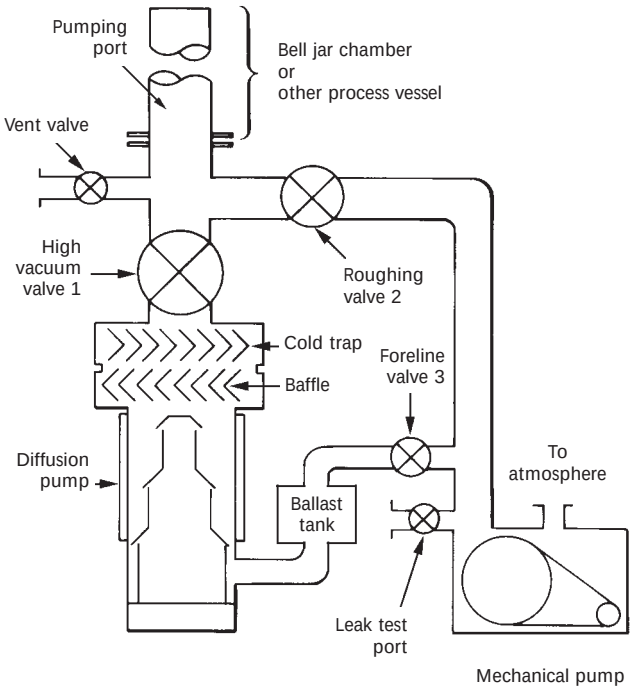
combination converts these gas molecules to solid compounds such as titanium oxide or titanium nitride. This process is called chemical gettering and produces the required pumping action. In addition, a second pumping action takes place. Some of the ionized molecules impact the cathodes with enough force to become buried in them, which prevents them from neutralizing and becoming a free gas again. A third pumping action occurs with hydrogen which diffuses directly into and reacts with the cathode plate. Also, neutral particles such as the inert gases can literally be buried or covered by the sputtered cathode material. Complex molecules may also be split apart in the discharge to smaller, more readily pumped molecules. Because these actions are not equally efficient, the chemically reactive gases such as hydrogen, nitrogen and oxygen are pumped at much higher speeds than the inert gases. A modification of the cathode design can be made to increase the efficiency for these inert gases.

Another characteristic of the ion pump, often referred to as a *sputter ion pump*, is that it is self-regulating. At higher pressures, where much ionization takes place, more current flows and at low pressures, less current flows. This characteristic current drain can be used to measure the pressure, or degree of vacuum achieved with the pump. This feature eliminates the need for an ion gage on the system. At lower pressures, ion pumps have long lives. Once they begin pumping, they quickly lower the pressure to the long life region. As long as they are not pumping against a leak, they will last for years. An example of this would be that a pump working at a constant pressure of 10^{-5} Pa (10^{-7} torr) would have a useful life of 20 years.

FIGURE 20. Schematic diagram of a typical complete vacuum system.



Procedures for Pumping and Operating Complete Vacuum Systems

By combining the components previously discussed with appropriate manifolding, plumbing and gaskets (O-rings), a complete vacuum system may be built as shown schematically in Fig. 20. The initial conditions are:

1. mechanical pump running,
2. diffusion pump operating and working in high vacuum,
3. cold trap filled with liquid nitrogen,
4. atmospheric pressure in bell jar chamber,
5. high vacuum valve closed,
6. vent valve open,
7. roughing valve closed and
8. foreline valve open.

An operational cycle for this vacuum system is as follows:

1. Close access to bell jar chamber, vessel or hood to be evacuated.
2. Close vent and foreline valves. The ballast tank permits the turbomolecular pump or diffusion pump to discharge to an expansion volume so that a high critical forepressure is not reached.
3. Start the roughing cycle by opening the roughing valve. This allows the mechanical pump to evacuate the manifolding between the high vacuum valve and the bell jar chamber.
4. After the pressure has been reduced to below 10 mPa (about 50 µtorr) or crossover point, close the roughing valve.
5. Open the foreline and high vacuum valves. This allows the diffusion pumping system (cold trap, baffle and turbomolecular diffusion pump) to continue pumping until the desired operating pressure is reached and work in the chamber may commence.

After completion of work in the bell jar or vacuum chamber, the system may be cycled to its initial condition by first closing the high vacuum valve and then opening the vent valve. This allows atmosphere to enter the bell jar chamber and system up to the roughing and high vacuum valves. The pressure equalization allows access to the chamber.

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PART 3. Materials for Vacuum Systems

Outgassing of Materials in Vacuum Systems

Adsorption refers to the condensation of gas (vapor) on the surface of a solid. As the pressure is reduced in a vacuum chamber, there is a spontaneous evolution of gas (and vapor) from materials in the vacuum; this is referred to as *outgassing*.

In vacuum systems the materials in the vacuum region may release adsorbed gases and vapors that increase the gas load of the system, resulting in a much longer pumpdown time. This phenomenon is most prevalent in new vacuum systems, unclean vacuum systems or vacuum systems that have been exposed to atmosphere for some considerable time. It will also occur when new materials or new work jigs and fixtures are installed in a vacuum chamber. Knowledge of the gas adsorption properties of various materials and, therefore, their outgassing properties, is very valuable in vacuum work.

Technique for Releasing Adsorbed Gases by Moderate Heating

Most metals in vacuum give off adsorbed or dissolved gases as well as gases resulting from the decomposition of oxide near the surface. To minimize this gas evolution, metals can be heated under vacuum before being used in vacuum systems. Gas adsorbed by exposure to atmospheric pressure can easily be released by heating to moderate temperatures. When pumping to pressures below 0.1 mPa (1 µtorr) where baking is not practical, great care must be taken in choosing the various materials in the system. This applies to choice of vacuum greases, elastomers, metals and various sealing compounds.

Factors Influencing Adsorption and Outgassing by Baking Vacuum System Materials

Gases and vapors are adsorbed by vacuum construction materials (metals and elastomers) and are gradually released. This set one limit on the lowest ultimate pressure that can be reached in a particular vacuum system. The usual technique of overcoming this problem is to degas the materials, usually by baking (raising the system to a high temperature while pumping). The bake-out temperature will depend on the temperature at which the material begins to change its properties. consequently, vacuum systems are degassed at fairly modest temperatures, say 300 to 400 °C (570 and 750 °F), for several hours while being pumped. This will eliminate much of the adsorbed gases and vapors.

The dissolved gas content of a metal or alloy will depend on factors such as (1) the nature of the metal, (2) the metallurgical process used in the production of the metal and (3) the degreasing and cleaning to which a metal was subjected. In comparing metals such as stainless steel and aluminum, stainless steel is found to outgas at a much lower rate. A cast aluminum surface outgasses at a rate about ten times higher than the rate at which a stainless steel surface outgasses. Therefore, during vacuum pumping, stainless steel vacuum systems are capable of reaching a desired vacuum in a shorter time than a comparable aluminum system with its higher rate of outgassing. Results are strongly influenced by the condition of a metal (its alloy, cleanliness, finish etc.).

Functions of Elastomers as Gaskets and Seals in Vacuum Work

Certain openings must be provided for the insertion, removal and sealing of equipment or materials for a given vacuum system. During the operation, these openings must be tightly sealed. Elastomers are the most widely used gasket material, where temperature and gas loads permit, because they offer reliable sealing. Elastomers are natural or synthetic rubbers that can be vulcanized to a state in which they have an inherent ability to accept and recover from extreme deformation.

For high vacuum service, leaks must be entirely eliminated and the gas evolved from the gasket material itself must be negligible. Both natural and synthetic rubber satisfy these requirements as long

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as negligible surface area is exposed. Frequently, gaskets must be exposed to oil or other gasket deteriorating substances and sometimes rather high or low temperatures must be tolerated. Gasketed joints should be readily accessible for tests for leakage. In designing a gasket these factors must be considered and specifications should be based on material capabilities as well as vacuum system operational requirements.

Selection of Gasket Materials and Design for Vacuum Seals

The choice of natural or synthetic rubber for a vacuum application depends on the combined qualities desired. In the case of rubbers, a wide range of characteristics is acceptable. Perhaps the most important single factor is that of allowable deflection under compression. This is a function of hardness and allowable permanent set. These materials generally contain volatile oils, plasticizers and coloring pigments that adsorb moisture and gases. Most of the chemicals used have low vapor pressure at room temperature. The outgassing rates for various elastomers depend on factors such as (1) the formulation used, (2) the area exposed, (3) the operating temperature and (4) the treatment of the elastomer before use. As a rule, there is no way to control the formulation of gasket materials because this is determined by the manufacturer. However, it is feasible to inform the manufacturer of intended service and ask for minimum volatiles. Exposed gasket area becomes critical as the operating pressure is lowered. Proper gasket groove design can help considerably in reducing exposed areas.

Because the outgassing rate of elastomers increases as the temperature is raised, the ultimate pressure can be reached more rapidly if the elastomer can be heated. However, all elastomers are damaged when heated too much. Also, the compression set increases more rapidly with temperature.

Because of these properties, elastomeric gaskets are not normally used in ultrahigh vacuum systems. Such systems are baked at temperatures well above the damage point of all known elastomers. In this case, it becomes necessary to use joints and seals of metals and alloys such as aluminum, brass, bronze, copper, indium, lead, silver, stainless steel and others.

Properties of Specific Elastomers for Vacuum Seals

Natural and synthetic rubbers are commonly used in systems that operate at room temperature and at pressures near 1 mPa (10 to 1 µtorr). Because of its temperature tolerance, silicone rubber is commonly used for low and high temperature operation. The fluorinated elastomers are highly resistant to most corrosive materials found in vacuum practice. Fluorocarbon resin is very good but suffers from *cold flow* under pressure at room temperature; suitable means for containing the fluorocarbon resin (spring loaded gaskets etc.) will eliminate this difficulty. In trying to reach very low pressures, the permeability of the elastomer as well as its outgassing characteristics must be considered. Permeability is the property that determines how readily gases will pass through a material.

Selecting Elastomers to Reach Low Pressure Vacuums

To reach low pressures at room temperature, elastomers with low vapor pressures and low permeabilities are desirable. consequently, considerable work has been done with fluorinated elastomers. Baking an elastomer at a temperature that does not damage it will reduce pumpdown time; however, it will still release some vapor after many hours of pumping.

Selection of Alloys for Use in Vacuum System Components

There are many alloys of copper, but only brasses and bronzes are used in vacuum practice. Brasses are copper zinc alloys, whereas bronzes are copper tin alloys. However, many brasses contain various other metals. Brasses are widely used for vacuum parts, such as diffusion pump parts, chambers, base plates, valves and fittings in high speed dynamic vacuum systems.

Many commercial bronzes contain zinc. Alloys containing zinc, cadmium, lead, antimony or bismuth should not be used in vacuum systems that are to be baked because of the high vapor pressures of these metals. Vacuum firing is likely to alter the composition and therefore the properties of such alloys.

Properties of Austenitic Stainless Steels in Vacuum Systems

Stainless steels have come into fairly common use in vacuum practice for turbomolecular pumps, diffusion pumps, manifolds, chamber baseplates etc. Austenitic stainless steels (types 302, 303 and 304) are commonly used in vacuum work and are often called 18-8 stainless steels because they contain about 18 percent chromium and 8 percent

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nickel. These steels are nonmagnetic and the melting points of austenitic stainless steels are over 1400 °C (2550 °F). Surfaces of stainless steels remain smooth because oxides and hydroxides do not occur as in other types of metals. This means that the effective surface area is less and vapors are adsorbed in smaller quantities. This leads to much easier degassing and quicker pumpdown.

Properties of Aluminum Alloys Used for Vacuum System Components

Aluminum is also being used in vacuum systems. The alloys of aluminum are generally readily worked in the shop without much difficulty, the workability depending on the composition. Surface hardening can be achieved easily by anodizing and other processes. Parts may be joined together by using aluminum solder. Cast aluminum alloy parts are used for a variety of purposes such as valves, turbomolecular pumps, diffusion pumps (particularly jet assemblies), grooveless flanges and gaskets. The design of the dies is important to get vacuum tight aluminum die castings. Although aluminum is difficult to de-gas thoroughly, it is commonly used for vacuum parts where good heat and electrical conductivity is required.

Properties of Other Metal Seals in Vacuum Systems

Certain specialty metals have almost the same coefficient of expansion as most glasses and have excellent sealing characteristics. They are used with vacuum flanges in the manufacture of ionization gage tubes and in other applications where metal-to-glass junctures and seals are necessary.

Applications and Limitations of Soft Metallic Vacuum Gaskets

Metal gaskets of some kind are used by vacuum seals that must be maintained at temperatures higher than about 125 °C (257 °F) or in which rubber cannot be used because of outgassing. Small gaskets of lead, copper, aluminum, gold, silver or tin have long been used for higher temperature vacuum services. Complete sealing demands high stresses and consequently the metal gaskets can only be used once. They are not designed for applications where the seals are often opened and then reclosed because the metal gaskets will take a permanent set and are not reusable in most applications.

Selection and Properties of Vacuum Greases and Oils

Vacuum greases are commonly used to help attain seals and to lubricate devices such as stopcocks and gasketed joints (static, rotating and sliding). In some cases, vacuum oils are used, including diffusion pump oils. Oils are generally not as satisfactory as greases for most types of seals, because they are more readily squeezed out, thereby leaving a dry seal. In general, vacuum greases should not have a vapor pressure of more than about 10 mPa (0.1 mtorr) at 30 °C (86 °F) and should maintain adequate viscosity at this temperature and can be used up to a few degrees below their melting point. In general, vacuum greases should be applied sparingly and surplus grease then wiped off, because greases absorb gases and vapors and are dirt catchers.

Diffusion Pump Oils

The ultimate vacuum of many vacuum systems is, in fact, limited by insufficient trapping of gas molecules by the diffusion pump fluid. Certain desirable properties that a diffusion pump oil must have include the following.

1. It should have low vapor pressure. Vapor pressures of typical diffusion pump oil recommended by manufacturers of diffusion pumps are in the range from 10 to 0.01 μPa (100 to 0.1 ntorr).
2. It should have low enough viscosity to flow back into the boiler.
3. It should have high molecular weight relative to the pumped gases to increase the efficiency of removal of gas from systems being evacuated by the vapor jets. Molecular weight of oil commonly used is in the range of 300 to 500 unified atomic mass units (u).
4. Oil should be thermally stable to avoid decomposition with heat. Decomposition often results in the evolution of more volatile fractions caused by cracking of the oil due to frequent exposure to atmospheric pressures.
5. The fluid should be chemically stable and noncorrosive in the presence of common metals, glass, elastomer gaskets and the gases and vapors usually present in vacuum systems.
6. It should be nontoxic.

The recommended hydrocarbon oils represent a very satisfactory low cost fluid for the normal vacuum range down to the low 10 μPa (100 ntorr) region. Ultrahigh vacuum is best obtained with oils specified by pump manufacturers. These oils are extremely stable, showing little change in properties even if the pump is exposed to atmospheric pressure with the heater on.

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PART 4. Vacuum System Maintenance and Troubleshooting

Maintenance of Vacuum Systems

The recognition, diagnosis, troubleshooting and treatment of vacuum system malfunctions and analysis of specific problems such as leaks commonly encountered in any vacuum system are important factors in maintaining vacuum systems at satisfactory levels of performance. The amount of maintenance service required by a vacuum system will depend on three basic factors:

1. The cleanliness of objects to be vacuum processed. Objects that are to undergo evacuation should be thoroughly degreased. Compounds or lubricants at connection points within equipment should always be held to a minimum.
2. The physical environment of the entire vacuum system. A clean temperature controlled environment is highly conducive to a long trouble free life of any vacuum system. Extreme ambient temperatures or high residual dust levels can appreciably affect the degree of trouble free operation to be expected from the system. When setting up a preventive maintenance schedule for any vacuum system, the actual environment in which the system is expected to function should be given prime consideration when selecting the rates and/or scheduled times at which specific preventive maintenance is performed. Under the heading of physical environment, one should also consider very carefully the reliability of available air, water and power sources. Although many vacuum systems are protected adequately against most emergencies, air, water or power failures with any vacuum equipment do not contribute to the overall well being of the machine.
3. The human element. The most serious consideration in maintenance of vacuum systems is that of personnel experience, care and training. Even with self-protected automatic vacuum machines, breakdowns do occur. If a unit is of the manual variety, particular concern should be directed to the human element. One cannot take too many precautions to prevent unauthorized personnel from tampering with a high vacuum evaporator or pumping station. This should be recognized in troubleshooting because it may well be the cause of certain problems.

Selecting Vacuum System Operating Schedules to Reduce Maintenance

Maintaining the cleanliness of internal machine parts exposed to high vacuum requires that the pumping system of a unit be kept running continuously as a machine cleaning function. In addition, the liquid nitrogen cold trap should not be permitted to run empty over night and over weekend periods. On manual as well as semiautomatic systems, strict attention should be paid to the proper manipulation of the system valves and to the selection of personnel having access to these valves. If the entire system has undergone cleaning, it is advisable to permit it to operate for a 24 h period without liquid nitrogen in the cold trap and with the port to the chamber or test volume blanked off. The preceding comment applies, although to a lesser degree, whenever the actual high vacuum portion of the system, i.e., that part of the system beneath the high vacuum valve, has seen atmospheric pressure, whether intentionally or otherwise, for more than a very brief period of time.

Delegating Responsibility for Operating Vacuum Systems

The human element problem is something best worked out within the individual company or group responsible for the vacuum system. Generally, it would seem best to delegate total responsibility for the operation and maintenance of the vacuum system unit to one responsible individual. Field experience tends to indicate that far fewer field problems occur with equipment that is owned and maintained under well defined levels of responsibility. Far more servicing is required for vacuum systems where no specific individual or group is held directly accountable for the condition of the equipment. Automation

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of startup and operating sequences minimizes these problems. Contractual service agreements can usually be obtained for the routing servicing and maintenance of vacuum equipment.

Preliminary Techniques for Locating Faulty conditions in Vacuum Systems

Frequently, maintenance checks show that the existing trouble with vacuum systems, although real enough, is not actually the result of a machine part failure. Consequently, on the assumption that the equipment was operating satisfactorily up to the point of failure, the following procedures for checks of basic power, water and air supplies should be followed:

1. Using a volt meter, check to make certain that the specified voltage is available at the power electrical outlet being used. Frequently, circuit breakers are opened within a plant. Occasionally workmen make power wiring changes within a plant and inadvertently disable parts of the electrical system. The operator should not assume that power is available at the wall receptacle unless he or she has personally checked and proven that the power is present.
2. If necessary, disconnect the outgoing water line from the system and be absolutely sure that cooling water is flowing through the water cooled component and exiting to the drain. Occasionally the water circuit will become plugged by debris in the line. Because some machines are protected against temperature rise in the diffusion pump, only roughing level vacuum may be achieved due to the automatic turnoff of the diffusion pump because of improper water flow. If the water flow is found to be blocked, correct this condition and continue with the machine startup procedure as specified in the manufacturer's operating instructions.
3. After checking water and power, be sure that proper air pressure is being maintained for actuating air operated valves. Low air pressure can cause some rather strange operational symptoms, which may be misdiagnosed as a vacuum controller failure or sticky valves. As often as not, low air pressure is the cause of sluggish or nonfunctioning valves.
4. Startup procedures should be reviewed to make certain that all operational switches are properly set and that the unit should indeed be running normally. No matter what the visible trouble symptoms may be, the aforementioned procedures should be followed before other service procedures are attempted.

Because power, water and other utilities vary considerably with the type of pumps and systems being used, the previous suggestions are only general. For more specific information, refer to the manufacturers instruction manuals.

Providing Necessary Information to Service Engineers

If, after completing the basic air, water and power checks described above, a simple explanation for the machine malfunction is not found, a written record should be prepared covering the following information:

1. A statement covering the age and history of the vacuum system, the serial number, what it has been used for, what it is currently being used for, who used it and in what manner, types of materials being used in the vacuum system, available maintenance history and in general, as many details as can be acquired.
2. Note carefully the symptoms observed with the particular machine and what has been done to this point about correcting these problems. When this information is available, do not hesitate to call the service engineer for the equipment and give him all details possible. It is entirely possible that, given useful information, he or she may be able to prescribe, via phone, the course of action needed to cure the vacuum system's troubles.

Also, if thorough information can be acquired via phone, the service engineer will be much better prepared to take care of the problem when he or she arrives at the plant, should that be necessary. The time it takes to repair the system will often be a function of the quality of communication between the plant and the service engineer.

Selecting Service Personnel within User Organization

Whether a service engineer has been called or not, if it is preferred to proceed immediately with troubleshooting a vacuum system, it may be possible to arrange for the services of a qualified individual within the user organization. Generally, the first choice for troubleshooting should be someone within the company who has had previous vacuum system experience whether with the same type of equipment

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or with some other type. A large organization may have a complete department devoted entirely to the maintenance of vacuum equipment. It is also possible that within a company some individual may have responsibility for maintenance of helium leak detection equipment (which has its own vacuum system). If neither a regular vacuum technician nor a leak detector maintenance technician is available, an electronic technician or perhaps a mechanical technician with some electronics knowledge would be desirable.

Recognizing Abnormal Operation of Vacuum Systems

There are really only two basic groups of vacuum systems problems, though each of these may be split into numerous subheadings: (1) vacuum system and/or mechanical problems and (2) automation and/or electrical or electronic problems. One of the most difficult and yet most important questions to answer adequately is just how well the machine would perform under a given operational condition — in other words, when a machine is normal in operation and when it is not.

For example, assume that all automation and normal sequential functions perform properly, but doubt exists that the vacuum performance of the machine is either normal or adequate under the operational conditions existent. It may be that the system is doing as well as can be expected when its actual work load, along with the time elapsed because system cleaning and maintenance, are considered. The best course of action in this case is to discuss the present operations and the previous operational history of the vacuum machine with the service engineer. If the information given him is correct and complete, he or she can evaluate the performance of the machine in the light of his or her field experience.

Performance of Vacuum System during Starting Transients

It is possible that, with extensive auxiliary equipment and heavy gas loads in the vacuum system, pumping times greater than normal may exist. It should also be noted that the rated performance for vacuum systems is for machines that are kept running almost constantly and not for equipment that has just been started up after routine shutdown or recent cleaning. When a machine has been freshly cleaned or simply shut down for some time, it may take 24 h or more before routine operational pumping times are obtained on a predictable basis.

Discriminating between Vacuum System Contamination and Leaks

After it has been determined that the vacuum performance of the system is abnormal, it is important to decide just what degree of malfunction is actually present. This is important because the two main problems will fall under the general headings of system contamination and system leaks. Whether or not a system is leaking or is contaminated is sometimes quite difficult to determine. However, if the vacuum system has been operating normally and has apparently slowly degraded in performance to an unacceptable but not catastrophic level, it is probably subject to contamination problems of one sort of another.

It is also necessary to consider any recent work done on vacuum systems because this, of course, could be a potential cause of system leaks. However, if vacuum performance has degraded rather drastically, especially to the point where only roughing level vacuum can be obtained, a leak is almost certain and troubleshooting procedures should be oriented around that assumption.

Residual gas analysis indicating a high nitrogen peak will often suggest a leak as opposed to contamination.

The most difficult vacuum system problems to solve are those where degradation is definitely moderate by any standard and could thus be caused by either system contamination or system leaks. If such appears to be the case, it is highly advisable that a thorough mass spectrometer leak detection test be performed. This is, as a matter of fact, a procedure that many use immediately on any vacuum system where performance levels have dropped to an unacceptable figure. It is a desirable procedure, because once leaks are eliminated as a source of trouble the only problem left is discovering and remedying the source of system contamination.

Problems Caused by Contamination within Vacuum Systems

As previously mentioned, one of the broad basic causes of poor vacuum performance is system contamination. It is also possible for the mechanical pump oil to become contaminated, which in itself can cause poor pumping characteristics. Before disassembling or cleaning an entire vacuum pump system, one of the first things to check is the condition of the pump oil. Immediately

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flushing and refilling of mechanical pump oil is called for if any indication of discoloration, low operating level or thinning out of the oil itself is evident. Many unnecessary cleaning jobs have been done because the mechanical pump was not routinely flushed and filled first.

It should also be noted that even though the roughing pressure may appear normal, this may be misleading to the extent that the mechanical pump may be just able to hold this pressure with no pumping capacity in reserve. Should this be the case, normal roughing pressures will be produced, but the moment a work leak is encountered, system performance will suffer. It never hurts to change the oil in the roughing pump. Be sure to flush only with specified roughing pump oil. Never under any conditions use acetone or other solvents in any mechanical pump.

Problems Caused by Contamination of Cold Traps in Vacuum Systems

If it is found that no performance improvement is attained after servicing the mechanical pump or pumps and attempting another system pumpdown, the next step before attempting complete disassembling and cleaning of the vacuum pump system is to follow the maintenance manual procedure for complete vacuum system shutdown.

Then remove, inspect and thoroughly clean the cold traps, baffles and cryopanels. After heavy use with dirty work loads, deposits accumulating on these cryopumps may reduce their ability to freeze out moisture due to the insulating effect of the previously trapped compounds. They may also produce a long term slow leak effect due to the outgassing of the materials deposited on their surface. This is why a vacuum system left running without liquid nitrogen after having been exposed for some time to heavy work load will often achieve substantial better vacuum when left running over a weekend. Sooner or later, the contamination on the cold traps, baffles and cryopanels will complete its outgassing and be pumped out of the system. In extreme cases, however, actual removal and cleaning of cold traps, baffles, cryopanels and chamber interior will restore system performance much quicker than attempting to clean only the pumps.

Changing Oil in Diffusion Pumps

A question that arises when the vacuum pump system has been shut down and the cold traps or baffles removed for cleaning is whether or not to remove the diffusion pump for cleaning and an oil change. This may be a very difficult question to answer. One should consider the degree of system malfunction, the length of time since the oil has been replaced and whether or not the vacuum system was ever inadvertently exposed to the atmosphere during operation. This is sometimes caused by improper operation and a hand operated valve or by accidental tripping of the wrong valve when an automatic system is operated in the manual mode. It should be noted that a system may stand a great deal of abuse in this particular area. However, if a system has been in operation for six months to a year and conditions have been moderately adverse, it would be considered good practice to change the diffusion pump oil. If the old oil has been *cracked* due to exposure to the atmosphere, then the pump should be cleaned before the new oil is added.

Preliminary Operation Following Maintenance Work on Vacuum Systems

After mechanical pumps have been cleaned and flushed, their oil changed, belt tension checked and adjusted, hose connections routinely tightened and checked, cold trap and baffles cleaned and the diffusion pump cleaned and the oil replaced, the system should then be put through a normal startup and pumpdown procedure and allowed to run for at least 24 h.

Performance checks should then be made on the system. It is very likely at this time that the system performance will be close to original specifications. If the diffusion pump oil was changed, performance is likely to improve during several initial days of operation as the diffusion pump oil becomes conditioned. This is a common occurrence in all diffusion pump vacuum systems.

If performance does not improve after the above procedures have been accomplished and thorough leak testing with a helium mass spectrometer leak detector has revealed no system leaks, it is then safe to conclude that cleaning of the entire vacuum system is necessary. This, of course, could have been done immediately on noticing the first malfunction symptoms. However, the previous procedure is recommended because total cleaning is frequently unnecessary and takes a much longer time to accomplish than the routine cleaning described.

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Detection and Repair of Leaks in Vacuum Systems

The process of helium mass spectrometer leak testing in high vacuum systems involves procedures and considerations described below. No differentiation is made here between manual and automatic operation because the basic vacuum plumbing system is identical, with the exception of hand operated rather than air operated valves. There are three general headings under which leaks may be classified: (1) gross single or cumulative leaks, (2) small single or multiple leaks and (3) virtual leaks.

Causes and Detection of Single Gross Leaks in Vacuum Systems

The single gross type of leak is usually one wherein a sealing member is or has become totally ineffective. This may occur as the result of an inadvertently pinched O-ring seal or improper welding. Often a gross leak of any type is also defined as one wherein the vacuum system cannot be rough pumped to below 100 Pa (1 torr) in the specified time for the pump system. However, it is usually found that, if roughing pumps cannot reduce pressure to the 100 Pa (1 torr) range, a seriously damaged seal will eventually be discovered.

Testing for a very large single leak with a throttled leak detector requires a slow and thorough operation. If a leak is such that pressure in the vacuum system only reaches the 100 to 50 Pa (1 to 0.5 torr) range, it may be easier to locate the leak by the vacuum gage tracer gas technique.

It should be noted here that many modern leak detectors have gross leak testing capabilities. Refer to each manufacturer's specifications.

Causes and Detection of Gross Cumulative Leaks in Vacuum Systems

Gross cumulative leaks, usually defined as several rather large leaks in vacuum system, give rise to the same lack of performance as that caused by a gross single leak. All the same procedures apply in dealing with gross cumulative leaks with the exception that, although large cumulatively, they may be too small individually to respond to the thermal conductivity gage spray leak test. If it is suspected that several leaks are causing the system failure (and this may indeed be the case, particularly if the system has been cleaned and reassembled by inexperience personnel), it may be advisable to engage a service engineer for assistance in remedying the problem. This is suggested because gross cumulative leaks in any vacuum system usually appear only during construction, after a system has been subjected to physical punishment or after inexperienced personnel have attempted the disassembly, cleaning and refitting of the vacuum plumbing. Inexperience in making vacuum seals may cause sealing surfaces to be damaged. Also, careless handling of parts, such as the overextension of brass bellows while it is removed from a bellows sealed valve, may cause rupture and should always be considered as a possible cause of gross leakage in a vacuum system.

Remember when testing for leaks due to ruptured bellows assemblies that a bellows will give no indication of a leak when the valve is closed unless leak tests are made through the vent on the atmospheric side of the valve to which the bellows is still exposed. Test possibilities may be found by examining drawings of bellows stem sealed valves. One may find that the leak can be located by using a leak detector connected to the pump valve or, in some cases, the vent valve. Judiciously opening and closing the suspected leaky valve while leak testing the dysfunctional bellows may permit its identification as the source of leakage.

Causes and Detection of Small Single or Multiple Leaks in Vacuum Systems

Small single or multiple leaks are readily located with a helium mass spectrometer leak detector. These types of leaks may allow a vacuum system to be evacuated at least into the low pascal range and usually into the high vacuum range. Perhaps the ultimate vacuum system pressure would be only about 0.5 Pa (3.75 mtorr). Under these conditions, a helium mass spectrometer leak detector properly connected to the vacuum system in question will quickly enable these small leaks to be detected. All suspected areas are helium tracer probed or bagged methodically in sequence while using a suitable leak testing procedure.

Causes of small single or multiple leaks are most often: (1) flanges that have been improperly tightened; (2) O-rings that have simply aged and taken a set; (3) undamaged O-rings that are improperly seated; (4) electrical feed-through seals; (5) tiny cracks in ionization gage tubes; (6) improperly fitted gage tubes; (7) poor fitting and/or seating of gaskets; and (8) weld joints that leak after repairs or on completion of new systems.

Any or all of these may contribute to small single or multiple leaks.

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PART 5. Equipment and Techniques for Measuring Pressure in Vacuum Systems

Introduction to Vacuum Gages

As important as the production of vacuum is the ability to gage its results through pressure measurement. Various types of commercial gages are available that cover the pressure range from atmospheric pressure to less than 10 μPa (100 ntorr). In the high pressure region, gages are used that depend on the actual force exerted by a gas. At low pressures, some specific property of gases (such as thermal conductivity or ability to become ionized) is used as the basis for measuring pressures.

Gages are generally calibrated in pressure units such as millipascal or micropascal (or the older units of torr or bar). The various types of common vacuum gages may be summarized as follows.

1. Pneumatic force gages depend on the actual force exerted by the gas. Examples are mercury and oil manometers, McLeod gages, Bourdon gages and diaphragm gages.
2. Thermal conductivity gages depend on the change of the thermal conductivity of a gas with change of pressure. The most common examples are the Pirani and thermocouple gages.
3. Ionization gages depend on the measurement of electrical current resulting from ionization of gas. Examples include thermionic ionization gages (Bayard-Alpert), cold cathode gages (penning or Philips) and alphatron gages.

Bourdon and Diaphragm Vacuum Gages

The Bourdon and diaphragm gages are mechanical gages that are used primarily for giving an indication that a vacuum system is actually below atmospheric pressure. Most of these gages indicate negative gage pressure from atmospheric pressure down to their lower pressure limit in the low pascal range (a fraction of a torr). They can be constructed of noncorrosive materials to make it possible to use them in the presence of corrosive gases and vapors. Because they work on the basis of the force exerted by a gas, they measure the total pressure of a mixture of gases and vapors.

Operation of Bourdon Vacuum Gage

Bourdon gages, shown in Fig. 21a, make use of a tube that is sealed off at one end with the other end leading to the connection to the vacuum system. The tube is usually of elliptical cross section and is bent into an arc. A change of pressure inside the tube makes it change its curvature. This change is transmitted through a series of levers and gears to a needle that gives a reading of the pressure on a circular scale behind the needle. As shown in Fig. 21b, the calibration of the scale in pascal absolute should ideally have 100 000 on top center, 0 at left bottom and 200 000 at right bottom. A few gages in North America are still based on inch of mercury, from 0 to 30 in. Hg, where 0 represents atmospheric pressure and 30 represents a good vacuum. Actually, the accuracies of most Bourdon gages may not be sufficient to read a good vacuum: the smallest reading is about 1 kPa (0.01 atm). However, these gages are occasionally still used to indicate the condition of a vacuum system.

Operation of Diaphragm Vacuum Gage

The operation of the diaphragm gage shown in Fig. 21c is based on transferring the distortion of the diaphragm to a scale reading. Diaphragm distortion is caused by a pressure differential across it. The scale may be calibrated in kilopascal, in torr or in inch of mercury.

Operation of Liquid Level Manometers (McLeod Gages)

Before 1981, the gage used most commonly as a comparison calibration standard by the National Institute of Standards and Technology and industry was the McLeod gage, a mercury barometer. It has since been replaced by the spinning rotor gage and accepted by the National Institute of Standards and Technology as the primary standard. As a

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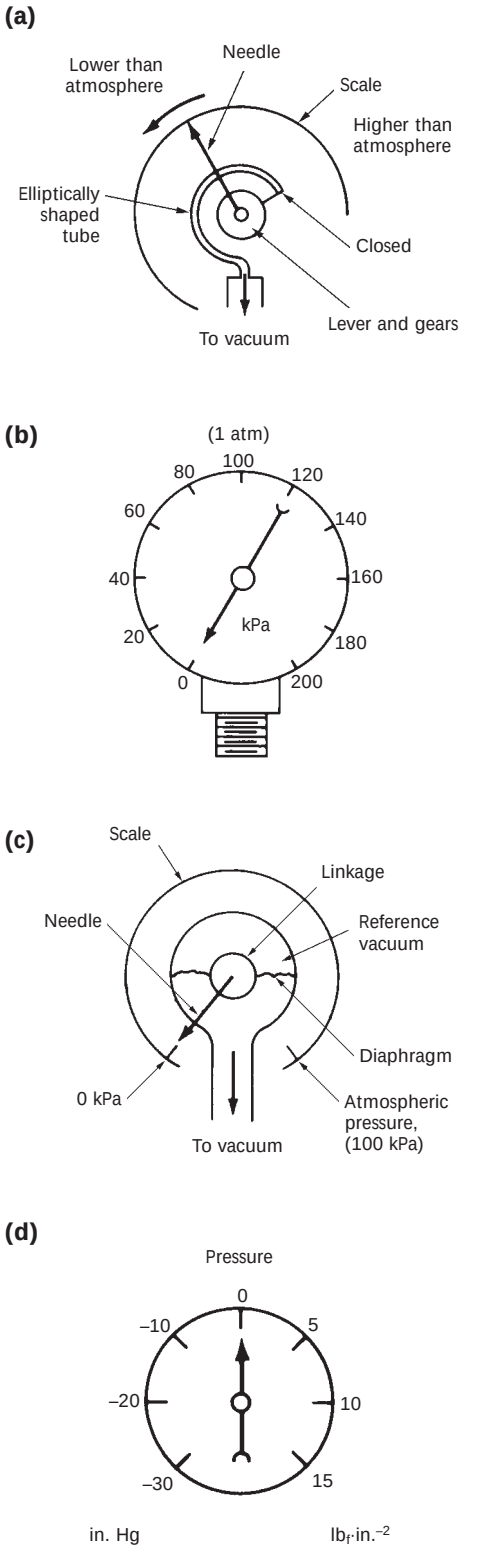
FIGURE 21. Principles of operation of mechanical vacuum gages: (a) elements of Bourdon gage; (b) external appearance of Bourdon gage; (c) elements of diaphragm gage; (d) older English combination gages with inch of mercury calibration on the left and pound per square inch on the right.



result, the following description of the McLeod gage will be abbreviated but sufficient to understand it.

The principle is based on the application of Boyle's law and is quite simple. A known volume of gas, at the pressure that is to be measured, is trapped and compressed by a known ratio to a new pressure that may be determined. By inserting the known values (original volume, final pressure and final volume) into the Boyle's law formula ($P_i V_i = P_f V_f$), the original pressure of the gas may be computed.

Determining Gas Pressure from McLeod Gage Reading

The gage is operated by raising the mercury above the gage head cutoff point indicated in Fig. 22a. A sample of the gas to be measured is trapped by rising mercury in the bulb volume between the cutoff point and the top of the closed capillary tube. This volume may be called V_i and is determined by the manufacturer when the gage is being fabricated. The mercury level is raised until the level in open capillary *B* is directly opposite the top of the closed capillary tube *A*. The mercury level is raised until $h = h'$. Raising the mercury level has compressed the sample volume of gas in the closed capillary so that it occupies the tube length, h. The sample has now been compressed to a new volume V_f equal to the cross sectional area of the capillary tube times the height *h*. The head of mercury, which is compressing it to this volume, is also $h' = h$. Applying Boyle's law, Eq. 28, it follows that:

$$\textbf{(28)} \quad P_i V_i = P_f V_f$$

where P_i is pressure of gas sample to be measured (unknown); V_i is bulb volume (known); P_f is final pressure of compressed gas sample which is indicated by the height of the mercury column, $h' = h$; and V_f is volume of compressed gas sample which equals gas column height *h* multiplied by the cross sectional area *a* of the closed capillary column. Inserting known values in Eq. 28 yields Eq. 29:

$$\textbf{(29)} \quad P_i V_i = h\left(a\,h\right) = a\,h^2$$

Limitations of McLeod Gage Measurements

The McLeod gage does not measure the pressures of condensables in the vacuum system. On the other hand, it is equally sensitive to all gases that follow Boyle's law. Its biggest disadvantage is that it has

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a discontinuous gage reading; continuous readings of pressure variation in a system are not obtainable with a McLeod gage.

Operation of Spinning Rotor Gage

The spinning rotor gage (Fig. 23) has been accepted by the National Institute of Standards and Technology as a transfer standard gage. This is possible because the principle on which the gage works can be related through calculation to basic laws of physics. Its name says exactly what it is — a spinning rotor. Several manufacturers produce them for use in metrology laboratories or for industrial applications where higher accuracy is needed without a mercury manometer and its toxicity related hazards.

A magnetized ball is magnetically suspended in a small chamber to eliminate all sources of friction except air friction. It is made to spin or rotate while suspended. If there are gases present in the chamber, the ball will slow down due to the impacts from molecules in the chamber. The rate at which it slows down is directly proportional to the gas pressure (number of impacts). All that needs to be done then is to very accurately measure the rate at which the ball slows down and calculate the pressure as a result. This is done by measuring the frequency of the magnetic pulses induced in the pickup coils. The calculation is, of course, done electronically by the attached control unit.

One manufacturer of this gage states an accuracy of 1 percent of the reading ±4 µPa (30 ntorr) between 10 µPa to 1 Pa (70 µtorr to 10 mtorr). Although you will not be using this gage as a routing pressure gage, your system gages may be calibrated using the spinning rotor gage.

FIGURE 22. Operating principle of the McLeod gage: (a) head arrangement; (b) quadratic scale measurement system ($h' = h$); (c) linear scale measurement system.

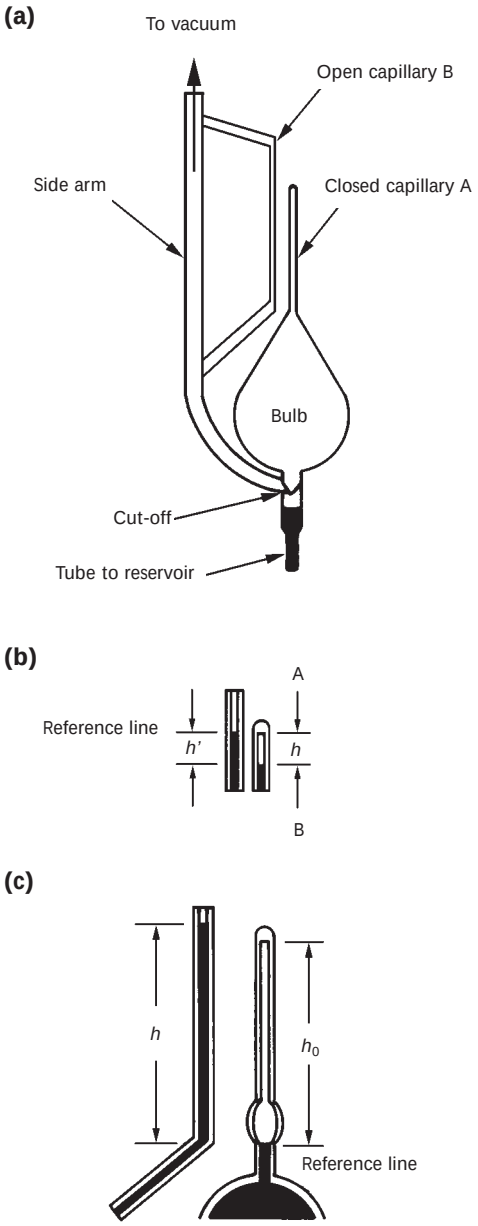
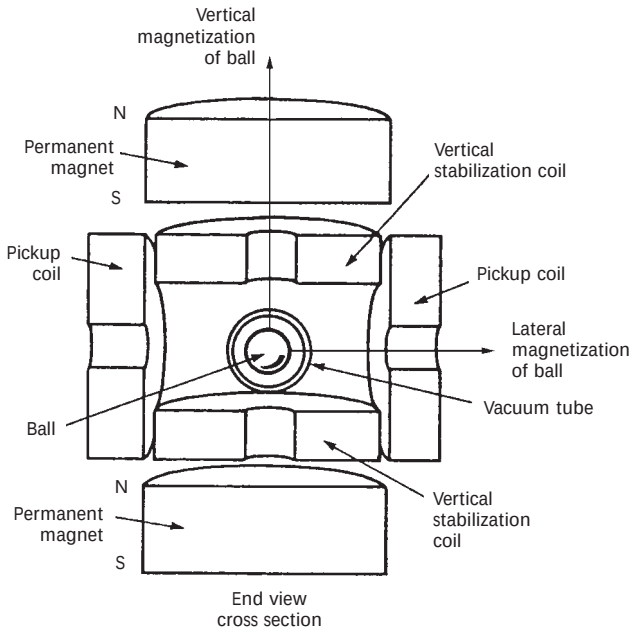


FIGURE 23. Spinning rotor gage.



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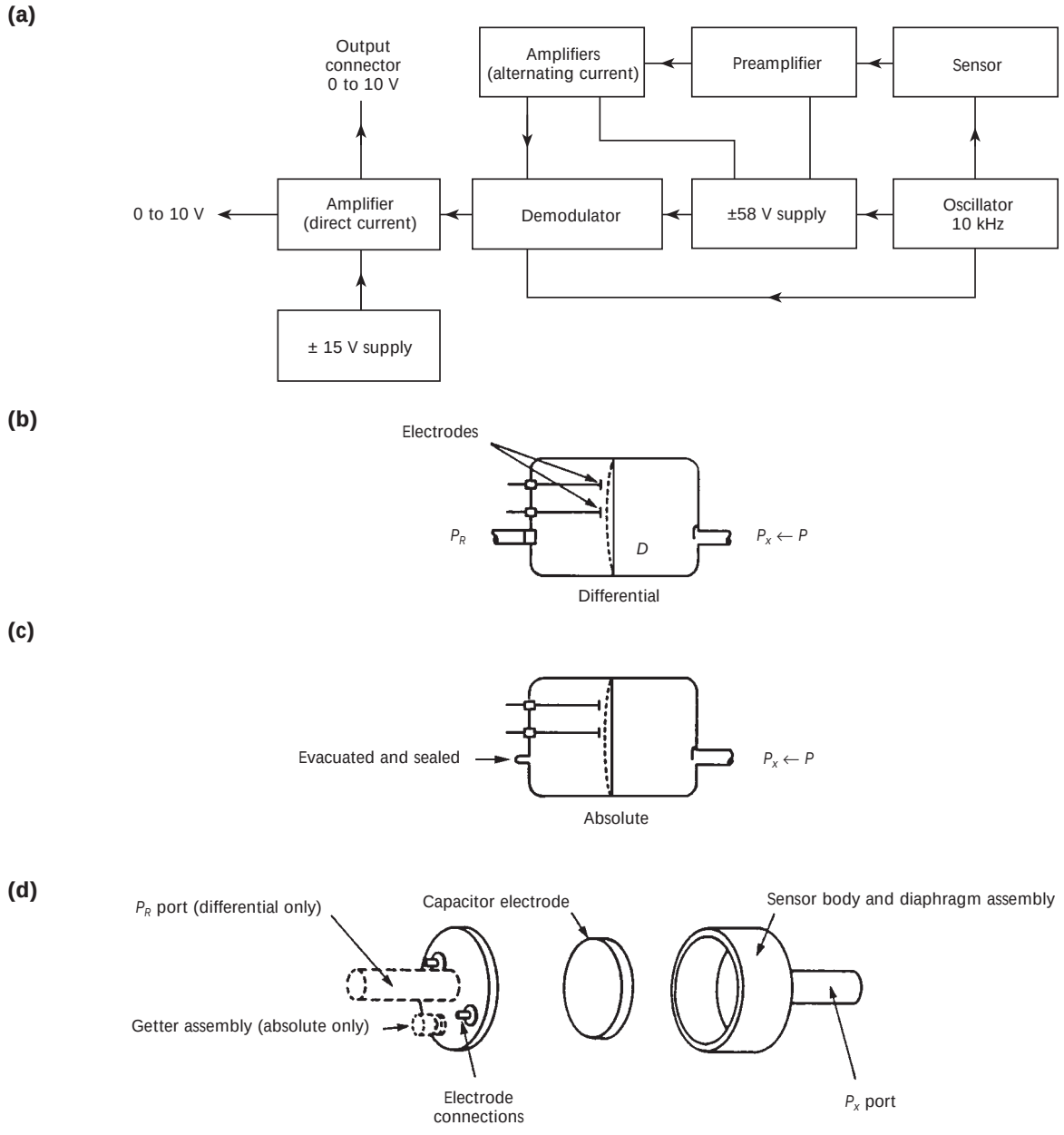
Operation of Capacitance Manometer

The capacitance manometer (Fig. 24) is another pressure gage that can be used in the rough vacuum range. It is capable of measuring the absolute pressure or relative pressure, depending on the gage model used. It does respond to the total pressure. It is not sensitive to changes in gas mixture as are many other gages.

The sensing unit contains a tensioned metal diaphragm, one side of which is exposed to the gas whose pressure is to be measured. For absolute pressure measurement, the other (reference) side contains an electrode assembly placed in a sealed high vacuum reference cavity. Because the electrodes in the absolute pressure gage are not exposed to the gases being measured, this gage is not affected by oil or water vapors or by corrosive or other chemically active process gases.

The diaphragm deflects with changing pressure force per unit area — independent of the composition of the measured gas. This causes a capacitance

FIGURE 24. Manometer gage: (a) schematic of electronic system; (b) differential setting; (c) absolute setting; (d) components.



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change between the diaphragm and the adjacent electrode assembly. The capacitance change is sensed in an oscillator circuit and converted to a frequency change proportional to the diaphragm deflection.

This frequency change, in turn, is converted in the unit to be displayed as the pressure reading. The sensor unit may be constructed of materials such as nickel base alloy and stainless steel, allowing the gage to be used with corrosive gases.

This gage is sufficiently accurate (about 1 percent of reading) and precise that one can worry about the effect of temperature changes (Charles' law) on the pressure readings. The sensor head may be placed in a constant temperature oven as a result. This gage is often used as a flow controller because of its fast response (milliseconds) to pressure changes. If you desire to use a capacitance manometer over a wide range, you may need several units. The gage is constructed to read over three or four orders of magnitude. If you wish to read from atmosphere (760 torr) into the high vacuum range (10 µtorr), that is seven orders of magnitude. Therefore, you need several different gage units. These gages can be constructed so that pressures from 10^5 to 10^{-5} torr may be sensed, but any particular gage is limited to about four orders of magnitude of that range. Below 0.1 Pa (1 mtorr) the accuracy falls dramatically.

The capacitance manometer may receive more maintenance than many gages because of its ability to read accurate and precise pressure values. It may periodically be taken to the calibration lab for a check against some standard gage. When it is used in dirty or corrosive gas systems, the sensing side of the gage head may be flushed with an appropriate solvent.

Overpressuring the gage (20 percent over full scale) may shift the reading or permanently damage it. An isolation valve is often used to prevent this.

Measuring Pressure in Vacuum Systems with Thermal Conductivity Gages

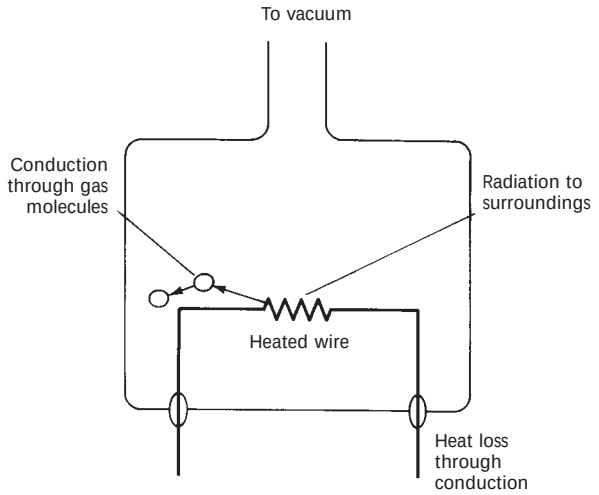
Heat transfer through a gas is related to the molecular density of the gas between surfaces across which a temperature difference exists. As gas molecules are removed from a system, the amount of heat transferred by conduction in the gas is also reduced. Finally, at a sufficiently low pressure, heat transfer within a thermal conductivity gage occurs by radiation and convection losses, while conduction effects are negligible (Fig. 25). The rate of heat transfer in a low pressure gas depends in a complex manner on the specific heats, molecular weight, temperature and pressure of a particular gas. Under suitable conditions it can thus be used as an indication of the pressure.

The useful pressure range of thermal conductivity gages extends from 270 Pa (2 torr) to about 0.1 Pa (1 mtorr), where the rate of heat transferred by radiation begins to predominate over the rate of heat transferred by conduction in the gas. The two most common types of thermal conductivity gages are the Pirani gage and the thermocouple gage. In both gages, conductivity changes of a gas cause a variation in the heat losses from an electrically heated filament. This temperature change is measured by means of a thermocouple in the thermocouple gage. A bridge circuit measures the change of electrical resistance of the heated filament in the Pirani gage.

Construction and Operation of Thermocouple Vacuum Gage

Figure 26 shows a simplified schematic of a thermocouple gage circuit. A thermojunction of two thin dissimilar metals are connected to the midpoint of a tungsten heater wire that is supported inside a metal envelope attached to the vacuum system. A constant current of the order of 30 mA is passed through the heater wire. The thermal electromotive force developed across the thermocouple wires is of the order of 10 to mV and may be read on a simple meter. The temperature attained by the thermocouple

FIGURE 25. Principle of the thermal conductivity (Pirani) gage. Thermal losses from the electrically heated resistance wire vary with heat conduction by gas molecules. Heat losses are reduced as gas pressure is lowered.



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depends on the conductivity of the gas surrounding the junction and thus on the pressure. The gage is calibrated to read on a logarithmic scale whose range may be extended upward by incorporating the convection principle with some reduction in accuracy.

The thermocouple gage, though not as accurate as the Pirani in vacuums near 10 mPa (or 0.1 mtorr), is more than adequate for forepressure measurements. Because of its simplified circuit, it is only about half as expensive as a Pirani gage and can be easily packaged into multistation vacuum leak testing instruments.

Advantages and Limitations of Thermocouple Vacuum Gages

The thermocouple gage has the virtue of simplicity and the disadvantage of a nonlinear scale. The calibration of the thermocouple gage may be changed by changing the heater current. A low value of heater current and a sensitive meter in the thermocouple spread the scale at low pressures. High current and a less sensitive meter spread the scale at higher pressures.

The advantages of the thermal conductivity gages for industrial application are numerous. They respond to vapors, read continuously and remotely, need not be fragile or bulky and may be used in automatic control systems. Their selective response to hydrogen and helium makes them useful for leak hunting. No damage is done to these gages if the vacuum system is exposed to atmospheric pressure while they are on.

Circuit and Operating Principles of the Pirani Vacuum Gage

Pirani gages use a Wheatstone bridge circuit, as shown in Fig. 27, which serves to heat a filament and to balance its resistance against a standard resistor sealed off in high vacuum. A change of pressure causes a change of filament temperature and, consequently, of the filament resistance, thus unbalancing the bridge. The pressure can then be measured in terms of the unbalanced voltage. Alternatively, the power required to maintain the filament temperature at a constant level is a measure of pressure. The temperature in this case is kept constant by means of feedback circuit.

The sensitivity of a Pirani gage decreases rapidly as the pressure is increased, owing to the fact that collisions between gas molecules become more frequent and that the thermal conductivity tends to become independent of the pressure. In the usual Pirani gage, a dummy tube (compensator) just like the one connected to the vacuum is used for one arm of the bridge. This tube is highly exhausted and sealed off. The two tubes are mounted together so that they will have the same ambient temperature. The bridge is balanced while the gage tube is under vacuum. The unbalanced current of the bridge is then taken as an index of pressure.

More recent digital readout Pirani gage designs incorporate compensating networks within the Wheatstone bridge to

FIGURE 26. Simplified thermocouple gage circuit.

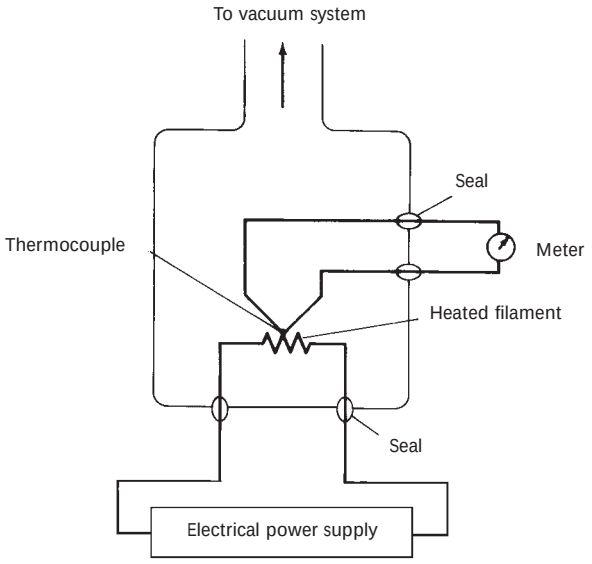
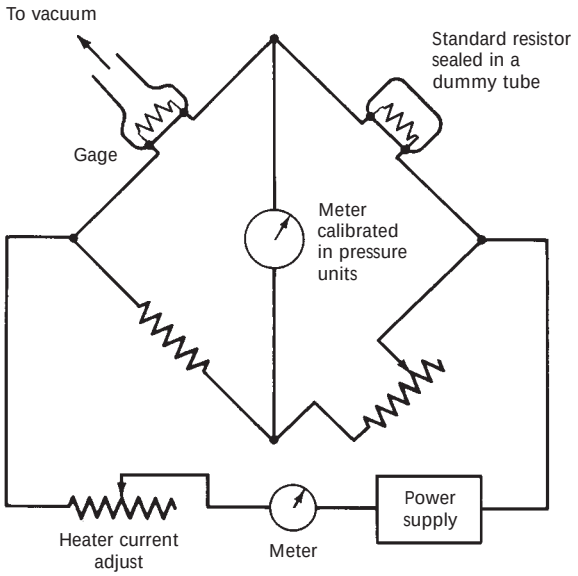


FIGURE 27. Pirani gage circuit.



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produce fairly accurate absolute pressure readings from atmosphere to 0.1 or 0.01 Pa (1.0 or 0.1 mtorr).

Creation of Ions in Ionization Gages Used for Measuring Vacuum

A neutral particle (atom or molecule) contains the same number of positively charged protons in the nucleus as negatively charged electrons in the orbits around the nucleus. By detaching one of the electrons from a neutral particle, a positive molecular or atomic ion is produced. The process is called ionization. This positive ion will be influenced by the same electric and magnetic forces that influence an electron, but in the opposite direction. For example, a negatively charged plate will attract a positive ion.

Ionization is fairly easily accomplished by electron bombardment. Electrons of sufficient energy, directed at a neutral particle, cause an energy transfer whereby the orbital electron attains sufficient energy to overcome the atomic forces that bond it to the nucleus. The orbital electron leaves its orbit as a free electron, leaving behind a positively charged ion. The ability of a gas to become ionized is the basis of ionization gages.

Types of Ionization Gages Used to Measure Vacuum

The different types of ionization gages vary in the manner of forming positive ions and in the manner of collecting them. all require calibration, although variation in sensitivity within a particular model is not great. The two ionization gages most commonly used are (1) the cold cathode or discharge gage (Philips gage) and (2) the thermionic ionization gage (Bayard-Alpert gage).

Of the several types of ionization gages, all have the common feature of measuring an ionization current that is proportional, for any one gas, to the molecular concentration. However, the probability of ionization of a molecule by bombardment by a charged particle is almost independent of the velocity of the molecule. Thus, the gage actually operates by measuring the molecular concentration in its electrode region rather than the pressure there.

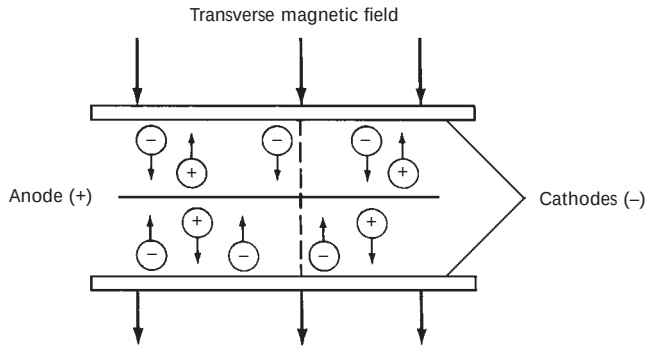
Cold Cathode Vacuum Gages

The cold cathode type of vacuum gage is also known as the Philips discharge gage or Penning gage. In the cold cathode gage (Fig. 28), electrons are drawn from the two plate type cathodes by the application of a high voltage and are attracted to the positive anode. The path of the electrons from cathode to anode is made several hundred times longer by arranging a magnetic field across the tube in the direction shown. The path now traveled by the electrons is a helix rather than a straight line. The increase length results in a proportional increase in the probability that an electron will ionize the molecules of residual gas by collision. An ionization current is produced that is several times greater than that which would be produced if no magnetic field were present. Actually, the total discharge current (the sum of the electron current from the cathode and the positive ion current to the cathode) is used as a measure of pressure in the system. No amplification of the discharge current is necessary and it may be fed directly to a pressure indicating microammeter that responds to the net current.

Performance Characteristics of Cold Cathode Ionization Gages

The range of cold cathode gage pressure measurements extends from 100 Pa to 10 µPa (0.5 torr to 0.1 µtorr). Because of its simplified circuit, this type of ionization gage is relatively inexpensive. Because the resistance changes with pressure, the ionization current output is nonlinear. The most accurate readings are obtained between 100 and 0.1 mPa (1 torr to 1 µtorr) where they can be used for fine pressure measurements. the cold cathode gage is not subject to sensing tube failures as a result of exposure to high pressures or a sudden loss of vacuum. Because of the

FIGURE 28. Principle of cold cathode discharge gage.



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heavy type of construction, the tube is not easily degassed. It is more readily contaminated owing to the high rate of ionization existing within the tube. Therefore, cold cathode gages should not be used for forepressure measurements.

Design and Construction of Cold Cathode Ionization Gages

The most common commercial cold cathode discharge gages do not use separate cathode plates. The trend has been instead toward all-metal construction with the inside wall of the tube acting as the cathode. The anode is usually in the shape of a ring, but also may be round, square or rectangular (Fig. 29). In some cases, use is made of a wire loop anode sufficiently heavy to prevent vibration and sagging. A compact, high strength alloy magnet is used. Usually, the magnet and gage tube are made as a single unit. Stainless steel, aluminum and nickel plated copper are used in commercial gages for the tube body (cathode). Theoretically, the cathode material should not sputter readily so that it will not produce a conducting layer on the insulator through which the anode is connected.

Principle of Operation of Thermionic Ionization Gages

The hot wire ionization gages is most widely used for measuring absolute pressure below 100 µPa (1 µtorr). Its operation depends on ionization of a gas with electrons emitted from a heated filament. The ions thus produced are collected and the resulting current measured. The most common version of the gage (Fig. 30) uses a tungsten or thoria coated iridium hairpin filament to emit an electron current of about 5 mA. The electrons are accelerated outward toward a cylindrical grid operated at about +150 V. The electrons usually do not hit the grid structure when they first reach it, but oscillate through it several times before being collected. An emission regulation circuit is used to keep the electron current at a steady value. Positive ions formed between the gird structure and an outer, cylindrical collector electrode are attracted toward the collector maintained at about –20 V. This positive ion current, flowing to the ion collector electrode, is

FIGURE 29. Commercial cold cathode gage.

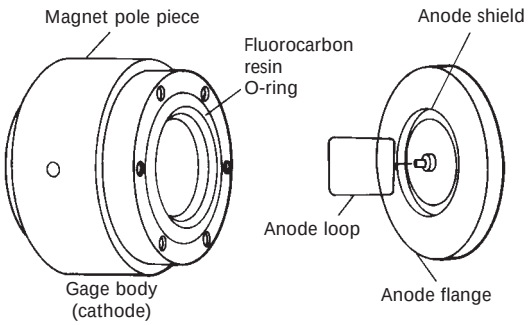
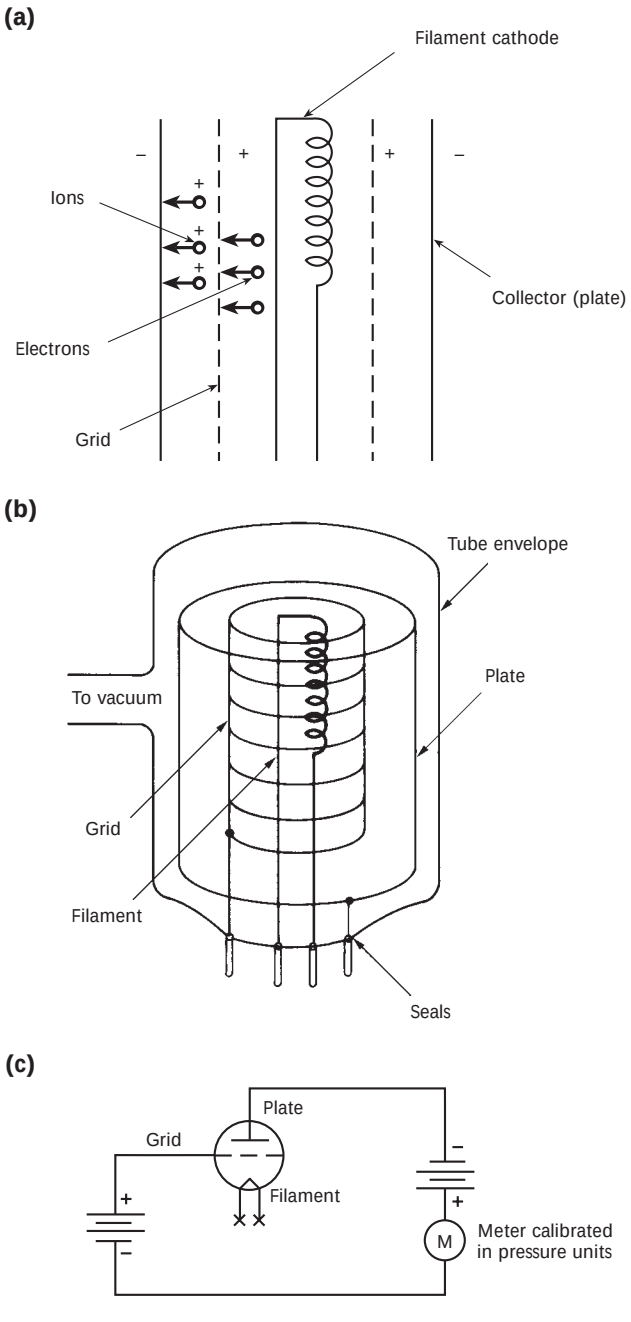


FIGURE 30. Hot filament ionization gage: (a) principle; (b) construction; (c) simplified electrical circuit.



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proportional to gas density over a wide pressure range.

Performance Characteristics of Thermionic Ionization Pressure Gages

The lower pressure limit for the gage configuration of Fig. 30 is 1 μPa (10 ntorr) The limitation is due to an X-ray effect that produces a constant residual collector current irrespective of pressure. Electrons arriving at the positive grid produce x-rays that irradiate the negative ion collector and release from its surface photoelectrons that are attracted to the positive electrode. The current of photoelectrons leaving the ion collector is indistinguishable from a current of positive ions arriving, down to pressures of 1 μPa (10 ntorr). The photoelectron current is roughly proportional to the surface area of the ion collector and surface area of the grid.

Operating Principle of Bayard-Alpert Gage for Pressures down to 1 nPa (75 ptorr)

For accuracy in reading pressures below 1 mPa, the constant residual collector current must be reduced to as low a level as possible. The Bayard-Alpert modification of the thermionic ionization gage accomplishes this by inverting the structure as shown in Fig. 31. The filament is outside the cylindrical grid, which acts as a positive potential to collect the electrons. The ion collector is at a negative potential and consists of a fine wire suspended centrally within the grid.

Because the area of the ion collector exposed to radiation from the grid is about 100 times smaller than that in the conventional gage, the production of photoelectrons and, therefore, of the residual constant background current is reduced proportionally. This makes it possible to measure ion currents corresponding to pressure of the order of 10 nPa (0.1 ntorr). Most of the X-rays are absorbed in the Bayard-Alpert gage by the glass envelope. However, to measure low pressure, it is necessary to thoroughly outgas the tube. Outgassing is usually accomplished by electrically heating the grid.

Performance Characteristics of Bayard-Alpert Vacuum Gages

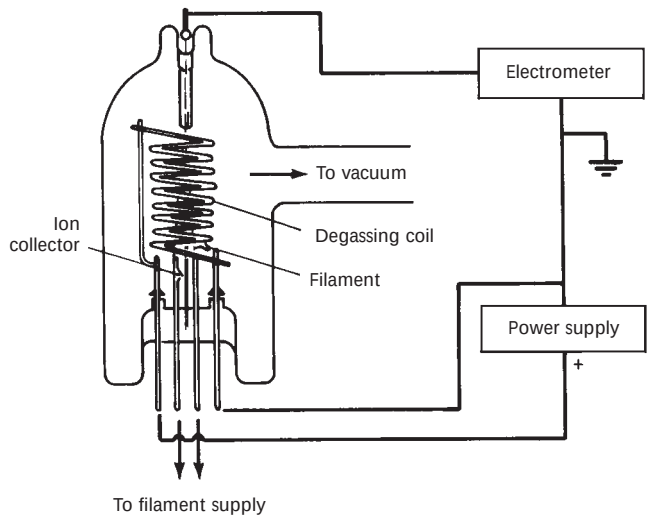
Major sources of error in pressure measurement with the Bayard-Alpert gages are (1) pumping action of the gage, (2) high-frequency oscillations and (3) decomposition of gas. Gage pumping action is a chemical as well as an electrical phenomenon. Chemical pumping at 8 mA electron current and 150 V electron energy is less than 2 $L \cdot s^{-1}$ (4.25 $ft^3 \cdot min^{-1}$) for nitrogen. This pumping action causes the gage to indicate lower system pressure than actually exists. High frequency oscillation in the gage may cause a buildup of potential as much as –150 V on the glass walls. This may have a serious effect on the gage sensitivity, especially between 100 and 10 mPa (1.0 and 0.1 mtorr). Some manufacturers coat the inside of the glass walls with a metallic film to remove this potential, thus increasing its accuracy.

Gas decomposition is encountered when the tungsten filament is operated at 2000 K (3140 °F). The most effective way to reduce this problem is by reducing the filament temperature. Thoria coated iridium filaments have been successfully used, providing high emission at relatively low temperature.

Calibration of Thermionic Ionization Gages for Different Gases

A thermionic ionization gage has different sensitivities for different gases. In reality, the gage measures molecular concentrations rather than true pressures. A gage measuring the pressures of two gas samples at different temperatures, but having the same pressure for both samples though the higher temperature sample really has a higher pressure.

FIGURE 31. Bayard-Alpert gage.



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The actual pressure of a particular gas is dependent in a complex fashion on the mass of the gas molecule and its ionization energy. These factors, though, are constant, so that a gage calibrated for nitrogen may accurately read the pressure of other gases by simply multiplying the indicated pressure by a constant factor.

As an example, consider a gage that is calibrated for nitrogen and reads 0.1 μPa. If the system is evacuated and backfilled with nitrogen, then it can be assumed that the total indicated pressure is almost completely due to nitrogen and therefore an actual pressure of 0.1 μPa (1 ntorr) exists. If, instead, the system was backfilled with helium, the total indicated pressure would be due almost entirely to helium and the actual pressure would be 6.2×0.1 mPa (6.2×1 μtorr) — 6.2 is the correct multiplication factor for helium.

Table 3 lists correction factors for different gases. Figure 32 is a graph of actual pressure versus indicated pressure for three gases, air, helium and argon, for a Bayard-Alpert gage.

Leak Testing with Bayard-Alpert Electronic Gage

Experience indicates that the Bayard-Alpert hot filament pressure gage, when used as an electronic leak detector on small volume systems, provides solutions to some of the problems of system leak detection encountered with the helium mass spectrometer. Unlike the spectrometer, the electronic leak detector uses a system's own vacuum pump, which is proportioned to the size of the system on which it is used. To operate, the detector need only be connected to the controller on the selected detecting device (either pump or gage) and an electrical outlet.

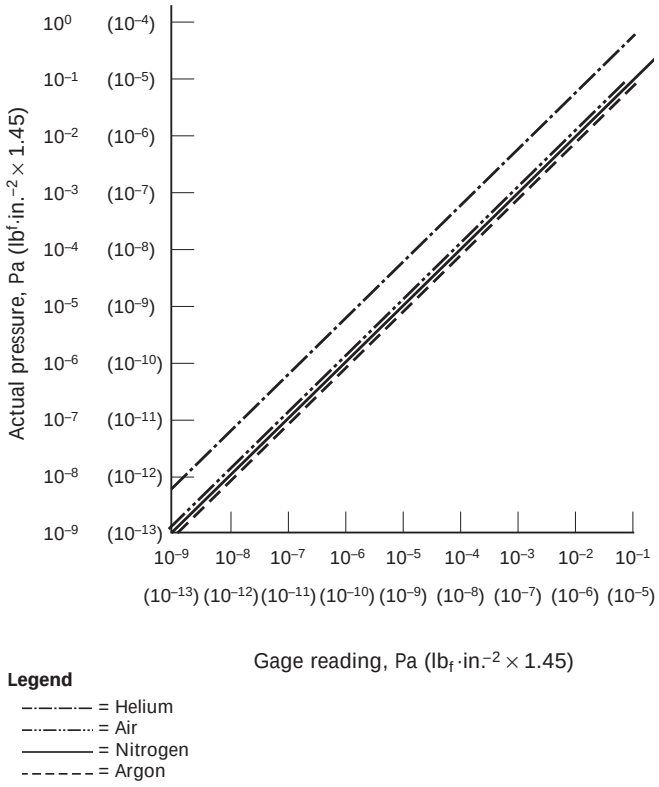
With the electronic detector, a small volume system can be leak tested at virtually any pressure at which it based out. When a leak is found, it can often be temporarily closed with plastic sealant and use of the system can continue until a permanent repair can be effected, thus avoiding wasted runs and down time. With the electronic detector, response to a leak is extremely rapid, regardless of the size of the system. Furthermore, cleanup time (that time required, once the tracer gas has been removed from the leak, for the background of tracer gas to dissipate, restoring a good signal-to-noise ratio) is remarkably short. Finally, the detector does not require liquid nitrogen and does not restrict the user to helium as a tracer gas. Although oxygen and argon give the greatest sensitivity, many other gases can be used effectively.

On the other hand, the measurement of a leak with the electronic detector presents one problem not encountered with the helium mass spectrometer. Unlike the spectrometer, the electronic

TABLE 3. Calibration of Bayard-Alpert ionization gages for different gases. Multiply ion gage reading by factor shown for correct pressure. To get sensitivity in μA·Pa^{-1}, divide 750 by gage factor (or μA per μtorr, divide 100 by gage factor).

Gas or Vapor	Gage Factor	Sensitivity μA·Pa^{-1}	Sensitivity (μA·μtorr^{-1})
Air	1.10	682	(91)
Argon	0.84	892	(119)
Carbon dioxide	0.73	1030	(137)
Carbon monoxide	0.94	800	(106.5)
Helium	6.20	121	(16.4)
Hydrocarbon pump oil	0.20	3750	(500)
Hydrogen	2.00	375	(50)
Krypton	0.53	1420	(189)
Mercury	0.29	2580	(344)
Neon	0.42	1790	(238)
Nitrogen	1.00	750	(100)
Oxygen	1.18	634	(84.5)
Silicone pump oil	0.37	2030	(270)
Water	1.12	670	(89.3)
Xenon	0.37	2030	(270)

FIGURE 32. Actual pressure versus indicated gage pressure for Bayard-Alpert gage.



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detector cannot be calibrated in absolute units. This does not mean that the electronic detector necessarily has a low sensitivity, but rather that its sensitivity varies with the size of the total gas load of the system on which it is used.

The electronic leak detector has the advantage that it is almost impossible for an operator to inadvertently damage it, the system on which it is being used or any instruments on that system. However, getting the best performance out of the instrument requires a reasonable amount of operator skill and experience.

Sensitivity Limitations of Bayard-Alpert Gage Used As a Leak Detector

As with any electronic device, the sensitivity of a Bayard-Alpert pressure gage is limited by the signal-to-noise ratio. The noise encountered comes from many different sources and is found to cover a broad frequency spectrum. The higher frequency noise sources are often the ion gage connections and the amplifier itself. Good connections and shielding should be maintained throughout this part of the ion gage circuit. Effects should be made to reduce the flow of cooling air currents about the gage tube and the movement of the collector cable during leak detection. The amplifier and, particularly, the filament emission regulator circuit should be working correctly to avoid variations in collector current. In the case of the ion pump, pressure changes due to gas bursts or leakage current in the pump can be a source of fluctuation. The pump history may show a cause for these effects and they may be cured by bakeout or high potential electrical testing in certain cases. Noise originating in the alternating current line should be largely eliminated by the filtering system in the leak detector.

Very low frequency noise or drift, having a time constant in the order of minutes, may be caused by a number of conditions. For instance, the system gas load may be changing, as is the case during pumpdowns or when the system is subject to thermal drift. In such cases, it is proper to wait until the system has based out and/or the thermal drift has been eliminated before leak testing. However, electronic detectors are normally supplied with an output connection to which a strip chart recorder can be attached. The deflection on the strip chart is of a definite and characteristic form, which allows it to be separated with reasonable ease from the background noise.

Obviously, the electronic leak detector is not the answer to all leak detection problems. It is impractical for work that requires absolute measurements of leaks and for all work with components. For the engineer interested in low pressures on small to medium systems, however, portability, ease of operation and low price (about one tenth the price of the helium mass spectrometer) make the electronic detector an extremely valuable tool.

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PART 6. Techniques for Detection of Large Leaks in Operating Vacuum Systems

Problems in Locating Gross Leaks in the Coarse Vacuum Range

Large leaks can be the most difficult and exasperating ones to find in vacuum systems. Most of the sensitive techniques and equipment developed for leak detection in vacuum systems are inapplicable at the pressures attainable by vacuum system pumps when large leaks are present (100 to 0.1 kPa or 760 to 1 torr). Consequently, large leaks usually are sought by one or another of a number of relatively crude techniques. Some of these tests are based on pressure testing or bubble leak testing techniques.

Design of Vacuum Systems for Convenience of Leak Testing during Operation

Because almost every (if not every) vacuum system will leak at one time or another during its lifetime, it is well to give some thought to the problem of ease of leak testing during the design of a vacuum system. A great amount of time can be wasted if poor leak hunting techniques must be used because it is too difficult or impossible to use a better technique on the existing system. The lack of forethought in this matter is all the more deplorable because improving vacuum system design to get better leak hunting efficiency usually requires only simple and relatively inexpensive measures, such as proper location of a valve or gage that will be in the system anyway.

It should be possible to isolate the roughing pumps from the system with a valve that can also be used to throttle the pumping speed of these pumps. A thermal conductivity gage should be placed in the fore vacuum line between this valve and the diffusion, turbomolecular or ion pump, for use in rate-of-rise measurements as well as to monitor the fore pressure. A stub into the foreline should also be inserted at this point for connection of a vacuum leak detector. The stub should have a valve and connection fitting (a flange that mates with the leak detector, a quick disconnect fitting or the like). This connection for a leak detector should be of fairly high conductance in order that response time not be impaired. If only the basic version of leak detector (without roughing pump) is available, it will help to have another valve between the stub and the turbomolecular pump or diffusion pump, so that the roughing pump for the system can be used to evacuate the line to the leak detector.

If possible, it is desirable to have a valve between the high vacuum chamber and the diffusion pumps. It need not be possible to throttle the pump with this valve, its main purpose being to isolate the chamber for either isolation or rate-of-rise tests. The chamber itself should have one or more ionization gages (even if an ion pump is used) in addition to any ultrahigh vacuum gage that may be used.

Leakage Rates Tolerable in Operating Vacuum Systems

Leaks can be tolerated in an operating vacuum system if the mass flow rate of the leak plus any outgassing load does not exceed the capacity of the pump at the operating pressure. For example, a system that must be maintained at 10 µPa (0.1 µtorr) with a 0.1 $m^3 \cdot s^{-1}$ (200 $ft^3 \cdot min^{-1}$) pump can handle $10 \times 10^{-6} \times 100 \times 10^{-3} = 10^{-6}$ $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} std $cm^3 \cdot s^{-1}$) of gas. So long as the sum of all leakage and outgassing is less than this value, the vacuum system operating pressure of 10 µPa (0.1 µtorr) will be obtained and there is no need to search for leaks smaller than about 10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-6} std $cm^3 \cdot s^{-1}$) in this system. If there are leaks larger than can be handled by the vacuum pumps, one of the techniques to be described can be used to locate the leak. In most cases the actual value of the leakage rate is not desired, although it can be obtained by using calibrated leaks with the leak detector on smaller volume systems or by using system calibrated leaks on very large volume systems.

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Leaks, Outgassing and Trapped Gas (Virtual Leaks) in Operating Vacuum Systems

When a vacuum system fails to reach the ultimate pressure that has previously been obtained with the system or which is expected for other reasons, air leakage into the vacuum system is to be suspected. However, a vacuum system that takes an unusually long time to reach its ultimate pressure (and for practical purposes fails to reach this pressure) often has internal sources of gas and vapor rather than leakage from outside the system. Before embarking on extensive leak hunting, the possibility of internal gas sources should be examined, as should the possibility of dysfunctional vacuum pumps or gages.

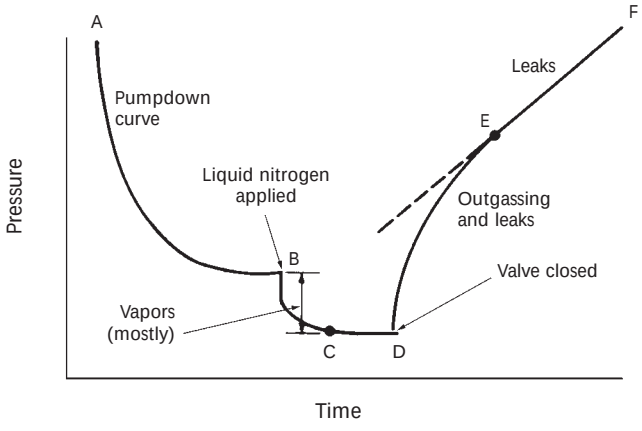
Gases and vapor can be released inside the system from the chamber walls and other materials inside the system (outgassing) or from small volumes with very low conductance paths for pumping (virtual leaks). Outgassing results from the evaporation of materials in the vacuum system (e.g., organic materials, ice on the exposed surfaces of cold traps and elsewhere, oil or grease etc.) as well as from permeation through the walls of the vessel and desorption of gas and vapor from interior surfaces. Outgassing is best controlled by careful attention to the properties of materials permitted in the system, cleanliness in construction and use of the system and the use of bakeout and cold trap techniques. Virtual leaks commonly arise from double welds, double gasket design, blind stud holes that are not vented etc. and can be avoided by proper design and fabrication. The various considerations and techniques used to minimize outgassing and virtual leaks are described earlier in this chapter.

Analysis of Vacuum System Pressure Transients during Pumpdown and without Pumping

Some degree of outgassing will be present in any vacuum system and will constitute a larger proportion of the gas pumped out as the vacuum decreases. An indication of the amount of condensable vapor present can be obtained from vacuum gage readings made with and without a cold trap. A marked reduction in pressure when the cold trap is filled indicates the presence of condensable vapors arising from outgassing surfaces and virtual leaks. Measurement of the rate of pressure rise can be used to verify the presence of leaks and can also provide an estimate of their size if the volume of the system is known.

Figure 33 shows a typical pressure time curve for a vacuum system with a liquid nitrogen cold trap. The curve shows the pressure variations during the pumpdown cycle and during a rate-of-rise measurement. The characteristic exponential decrease in pressure occurs from A to B, during pumpdown. The pressure levels off as the system approaches equilibrium between pumping speed and the gas load from leaks and outgassing. At B the liquid nitrogen trap is filled and the pressure falls rapidly as condensable vapors are captured by the trap. Again an equilibrium pressure is reached, limited by noncondensable gas from leaks. At point D the vacuum chamber is valved off from the pumps and cold trap and the pressure begins to rise. The rate of pressure rise will decrease in the region from D to E as the contribution from outgassing becomes negligible in comparison with any leaks present. Finally, the pressure-time curve becomes nearly a straight line in region E-F. If slope dP/dt approximates Q_L/V, where Q_L is the leakage rate and V is the volume of the vessel.

FIGURE 33. Pressure versus time curve of vacuum system pumpdown and subsequent measurement of rise rate.



Legend

A = Pressure before pumpdown
B = Liquid nitrogen trap is filled
C = Trap captures condensable vapors
D = Vacuum chamber is valved off
E = Pressure rise curve is no longer influenced by outgassing
F = Final reading

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Sensitivities of Leak Tests for Operating Vacuum Systems

The choice of leak testing technique for use on operating vacuum systems depends on such factors as (1) magnitude of leakage, (2) pressure within vacuum chamber during leakage detection and/or measurement, (3) pressure external to vacuum chamber during leakage detection and/or measurement, (4) capacity of vacuum pumps at operating pressure with leakage occurring, (5) tracer gas type and ease of detection (if tracer is other than air), (6) internal volume of vacuum system, (7) virtual leakage and effects of outgassing and (8) sensitivity of vacuum gage or tracer leak detector used in leak testing.

Table 4 lists the pressure ranges and leakage rate sensitivities of various techniques of leak testing of operating vacuum systems. Of course, when vacuum systems are not operating and can be pressurized or when components of vacuum systems can be removed and tested separately for leaks, the many other leak testing techniques described in this volume may be applicable.

Auditory Aids to Detection of Large Leaks in Operating Vacuum Systems

The first indication of the existence of a large leak in a continuously pumped vacuum system is usually an audible one—the distinctive sound of a mechanical pump that is pumping large quantities of air long after the system should be in the initial vacuum range (see curve *AB* in Fig. 33). Gross leaks correspond to openings with diameters of about 10 µm (4×10^{-4} in.) and larger. Hence, the hissing of air through large leaks can sometimes be heard and used to locate them. An improvised stethoscope or listening tube improves both the sensitivity of the technique and the ability to pinpoint the location of the leak. Advanced ultrasonic leak detectors can also be used to locate large leaks. Sensitivity may also be improved (and the pump spared) if pressure testing is used instead of vacuum testing.

TABLE 4. Sensitivities of some techniques of leak testing in vacuum systems.

	Smallest Detectable				
	Pressure Range		Leakage		
Technique	kPa	(torr)	Pa·m³·s⁻¹	(std cm³·s⁻¹)	Remarks
Hissing of air	10 to 200 kPa	(100 to 2000)	3×10^{-3}	(3×10^{-2})	quiet room
Wavering flame	100 to 400 kPa	(1000 to 4000)	4×10^{-3}	(4×10^{-2})	draft-free room
Halide torch	>100 kPa	(1000)	1×10^{-5}	(1×10^{-4})	used with refrigerant-12
Bubble techniques					
air and water immersion	0.01 to 400 kPa	(1 to 4000)	1.5×10^{-5}	(1.5×10^{-4})	
water and alcohol immersion	0.01 to 400 kPa	(1 to 4000)	5×10^{-8}	(5.0×10^{-7})	good ventilation
air and soap film	0.01 to 400 kPa	(1 to 4000)	5×10^{-6}	(5.0×10^{-5})	good light; ≥ 5 min observation
Spark coil or discharge tube	0.1 to 100 Pa	(0.001 to 1.0)	~0.001	$(\sim 1 \times 10^{-2})$	leakage dependent on voltage; glass system; residual gases cause confusion
Pirani and thermocouple gages	$<1 \times 10^{1}$ Pa	(0.1)	1×10^{-6} to 1×10^{-7}	$(1 \times 10^{-5}$ to $1 \times 10^{-6})$	used with acetone, hydrogen methanol
Halogen detector	<10 Pa	(0.1)	1×10^{-7}	(1×10^{-6})	
Ionization gage	<0.07 Pa	(0.0008)	dependent on pressure		used with hydrogen, helium, oxygen, butane
Ion pump leak detector	<0.01 Pa	(0.0001)	dependent on pressure		used with argon, oxygen, helium
Mass spectrometer leak detector					
direct flow	<0.01 Pa	(0.0001)	5×10^{12}	(5×10^{11})	used with helium
counterflow	40 Pa	(0.3)	1×10^{11}	(1×10^{10})	used with helium
residual gas analyzer			10^{-10} to 10^{-11}	$(1 \times 10^{-9}$ to $1 \times 10^{-10})$	used with any gas

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Pressure Gage Leakage Tests of Small Vacuum Systems in Operation

A simple technique can be used for preliminary leakage testing of small high vacuum systems in operation. This technique makes use of the vacuum gage that already exists in most vacuum systems. The most common gages are of the thermal conductivity type for pressures as low as about 0.1 Pa (1 mtorr) and some variation of the ionization gage for pressures below the 0.1 Pa (1 mtorr) range. Both gage types can be used for leakage detection, but the ionization gage is preferable because of its faster reaction time. However, if a very large leak makes it impossible for the pump to reach the working range of the ionization gage, the thermocouple gage may be used in essentially the same way but at a slower pace.

High Pressure Air Jet Tracer Technique for Locating Leaks in Operating Vacuum Systems

A simple leak locating tracer technique involves blowing a jet of high-pressure air onto the outside of the vacuum chamber wall. This raises the air pressure differential across a small area of the chamber wall. If a leak is within this area it will now conduct more air into the chamber. The higher leakage rate can immediately be detected on the vacuum gage as a slight increase in chamber pressure.

In practice, a sharp air jet from a small nozzle is moved over all suspected areas; the common shop air supply system will do very well. The scanning can be rapid, because reaction and recovery times are of only a few seconds duration. This technique is most useful for quickly testing for leaks in a weld or an O-ring sealed flange.

Vacuum Hose Technique for Locating Leaks in Small Operating Vacuum Systems

Another simple technique of locating leaks in operating vacuum systems is based on the same idea, to change the pressure differential across the leak and to observe the change in leakage rate with help of the gage. This time, however, the pressure on the air side of the leak is reduced rather than increased. For this procedure, a source of vacuum is required. The vacuum line available in many laboratories, a small vane pump or even a water injection pump are all adequate. A hose of appropriate diameter is connected to the vacuum pump; its other end is then simply pressed against the chamber wall to create a small area of reduced pressure. If the leak is within this area, an almost immediate improvement in the chamber vacuum will result.

The vacuum hose technique works best on flat, smooth wall sections. Its special merit, besides being very fast, is that the area under investigation is sharply limited and very well defined. In cases where there are several potential leaks in a small area, it has proven to be superior to any tracer gas technique. The vacuum hose can be applied to one small zone after another until the leak is positively localized, whereas it is difficult to confine any tracer gas to equally small zones without diffusing some into adjacent areas.

Helium Mass Spectrometry

The helium mass spectrometer leak detector (usually referred to simply as a helium leak detector) is adjusted to respond only to helium gas (atomic mass = 4). Although several types of mass spectrometer are used in these devices, by far the most common is the simple magnetic analyzer.

By choosing the suitable magnetic field strength and acceleration voltage, the mass spectrometer can be tuned to any mass of gaseous particle. Hence, any gas could be used as a tracer gas for leak detection. Helium has often been chosen for the following reasons. It is present in the atmosphere at a concentration of about 5 $\mu L \cdot L^{-1}$. Thus, air leaks cause very little helium background in the detector. Helium is inert and readily available in most countries. Because it is the lightest gas except hydrogen, helium's diffusion and molecular flow rates are the highest available with a nonhazardous gas. These properties are highly desirable in a tracer gas.

Helium Tracer Gas for Large Leaks in Vacuum Systems

The helium mass spectrometer leak detector can sometimes be used to find even large leaks, although its main use is in finding small and very small leaks. Because the pressure in the conventional helium mass spectrometer leak detector cannot exceed 10 mPa (0.1 mtorr), the leaking vacuum system is pumped at the greatest attainable pumping speed and the opening to the leak detector is then throttled until the operating pressure is achieved.

It is particularly important that the helium probing procedure be observed when testing for large leaks. Otherwise,

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the detector can easily become saturated long before the leak is reached. If a counterflow leak detector is being used, testing pressures maybe as high as 40 Pa (0.3 torr) can be tolerated without the need for throttling the leak detector, without loss of sensitivity. Some models of leak detectors have built-in capabilities of testing at pressures as high as 400 Pa (3.0 torr). The pressure testing technique on large leaks is virtually impossible because it saturates the mass spectrometer detector chamber with helium tracer gas.

Leak Testing of Vacuum Systems of from 100 to 0.1 Pa (100 to 1 mtorr)

Most of the above techniques for detecting large leaks have sufficient sensitivity to be useful with leaks that limit the pressure to the vacuum range of 100 to 0.1 Pa (100 to 1 mtorr) with the pumping speeds commonly used in this range ($S \geq$ ~1 $L \cdot s^{-1}$ or ~2 $ft^3 \cdot min^{-1}$). However, when vacuum system pressures lower than 100 Pa (1 torr) can be obtained, several additional vacuum leak testing techniques avoid the inconvenience of pressure testing and can be used on systems that cannot be pressure tested.

Tesla coils and high voltage discharge devices, which were among the earliest leak detection tools used on vacuum systems, provide a rather qualitative indication of the pressure and type of gas in the system. They can be used only on glass systems or in glass walled sections of metal systems. For example, they can be used along the glass tube leading to an ion gage only if the ion gage is turned off. Commercial spark coils (Tesla coils) for vacuum testing produce a high frequency potential of several thousand volts at a pointed electrode. When the tip of this electrode is held near (about 1 cm from) a glass system whose pressure is in the vacuum range of 100 to 0.1 Pa (1.0 to 0.001 torr), a gaseous electrical discharge is produced in the vicinity of the electrode. The color and appearance of this gaseous discharge depend on the composition of the residual gas in the system and on its pressure.

Sensitivity and Limitations of Spark Coil Leak Location

The white spark technique of high voltage discharge leak location is qualitative, but will probably detect leakage as small as 10^{-5} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-4} std $cm^3 \cdot s^{-1}$). The size of the smallest detectable leak depends on leak geometry. The leak testing technique consists of evacuating the system to a pressure between 1 kPa and 1 Pa (10 and 0.01 torr) and scanning over the suspected areas with a probe connected to a high voltage induction coil.

The white spark technique is only applicable where no metal exists because the spark from such a coil will ground through metal parts. If the spark tip is brought closer than several centimeters from metal parts, the spark will jump to the metal. Thus, spark coils cannot be used on all-metal systems. However, they can be quite useful on all-glass systems or even on metal systems containing glass parts. On continual exposure, the high voltage spark may puncture thin glass walls. Therefore, the probe should be moved slowly rather than held in one place. In the same manner, a high voltage spark might score the barrel of a fluorocarbon resin stopcock and rupture plastic or rubber gaskets.

Location of Vacuum System Leaks by Glow Discharge Color

The color differentiation technique of high voltage discharge leak testing is primarily a technique for leak location and is applicable to evacuated systems. It is always used in the tracer probe mode. The color differential technique involves observing changes in color of high voltage glow discharges within the evacuated space produced by probe gases or vapors entering the leak. A spark coil can be used to excite a visible glow discharge if the pressure in the system is within the range of 1 Pa to 1 kPa (0.01 to 10 torr). A tracer gas such as carbon dioxide or a volatile liquid such as benzene, acetone or methyl alcohol is applied to the exposed outer surface of the vacuum system under test. When the tracer gas or vapor enters the system through a leak, the color of the discharge changes from the reddish purple of air to a color characteristic of the tracer material. For liquids such as benzene, acetone or alcohol, the color of the glow discharge would be grayish blue. Carbon dioxide gives a bluish green glow to the electrical discharge.

During glow discharge leak testing of vacuum systems, the spark coil tip is kept on one glass section of the system under test. Preferably this section will be between the diffusion pump and the forepump to have a pressure sufficient to maintain a glow discharge. The nature of the glow discharge will depend on the pressure and on the gases in the system. The glow discharge color is characteristic of the gases present. For air, this color is reddish or purplish. The exact color (as for other gases) depends to some extent on the glass used in the system. Soda glass will show a yellow-green fluorescence whereas lead glass shows a blue

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fluorescence. The probing fluid used can be a gas or a liquid. Some tracer materials that are commonly used are illuminating gas, ether and carbon dioxide. With the first two materials the discharge takes on a grayish blue appearance. This is similar to the characteristic color or carbon dioxide (see Table 5) but, possibly because of fluorescence of the glass, the color is often reported as bluish green.

Leak Location by Isolation in Operating Vacuum Systems

The principle of fault isolation applies particularly strongly to leak detection in operating vacuum systems. Because leak hunting is usually a tedious and time consuming job at best, any steps taken to isolate the leak to a particular part of the system can shorten the process considerably. Often various parts of the system can be valved off and pressure gages used to indicate when the leak has been isolated. A system that has a history of achieving adequately low pressure may leak after being opened. In this case, the obvious initial candidates for leak testing are the gaskets on any flanges removed and possible the valves used to vent or seal off the system. For many systems there is a high probability that the leak will be found in these mechanical seal areas rather than elsewhere, but in some cases, such as when temperature cycling of the system is involved, the new leaks may be far removed from the openings used. If the leak can be positively isolated to a given area, it should be.

TABLE 5. Discharge colors in gases and vapors at low pressures.

Gas	Negative Glow	Positive Column
Air	blue	reddish
Nitrogen	blue	yellow or red gold
Oxygen	yellowish white	lemon
Hydrogen	bluish pink or bright blue	pink or rose
Helium	pale green	violet-red
Argon	bluish	deep red or violet
Neon	red-orange	red-orange or blood red
Krypton	green	no distinctive color
Xenon	bluish white	
Carbon monoxide	greenish white	white
Carbon dioxide	blue	white
Methane	reddish violet	
Ammonia	yellow-green	
Chlorine	greenish	light green
Bromine	yellowish green	reddish
Iodine	orange-yellow	peach blossom colored
Lithium	bright red	
Sodium	yellowish green (whitish)	yellow
Potassium	green	green
Mercury	green or goldish white	greenish blue or greenish

Sealing Technique for Determining Leak Location

The sealing technique involves gradually covering outside parts of a system being evacuated with some material that will seal the leak. Once the leak has been covered, the pressure will drop. In this way, leaks can be located and permanent repairs made. The procedure is to paint, brush or spray the sealing substance over various parts of the system until a change in pressure is noted. Either a thermal conductivity or an ionization gage may be used, the choice being dictated by the pressure.

The sealing substance may temporarily or permanently seal the leaks. some semipermanent sealants are insulator lacquers, shellac in alcohol, epoxy and vacuum cements that are liquid at room temperature such as cellulose acetate. Some temporary sealants are water, acetone and alcohol.

Two effects result from a liquid sealant. First, after the initial closing of the leak, the pressure will drop. Second, as the vapor enters the system, the gage will show a change in pressure, which will depend on the nature of the vapor and on the type of gage. The vapors from solvents such as water, acetone or alcohol are readily condensable. Consequently, all gages used with a cold trap will show a pressure change when a leak is covered by a liquid. The particular liquid used (no cold trap) will determine whether the gage shows an increase or decrease in pressure. Alcohol, acetone and ether — commonly used probe liquids — all show an initial increased pressure reading with an ionization gage or thermal conductivity gage but may then change to a decrease in pressure due to the temporary plugging of the leak.

Effect of Sealant Material with Very Small Leaks

For very small leaks, a permanent sealing material works satisfactorily. The temporary sealing substances are quite effective for all sizes of leaks except the very smallest. If a very small leak is sealed with a temporary sealant, it will open again at some inopportune time; therefore, this technique is not recommended if the small leaks have to be located and permanently repaired.

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Effect of Sealant Material with Large Leaks

In repairing large leaks, the sealant material is drawn into the vacuum system and a seal cannot be obtained. Although the permanent sealing substances give fairly satisfactory results with leaks in metal plates, in soldered, brazed and welded joints and in glass systems, they are not as satisfactory as a final repair obtained by reworking the material of the vacuum system by soldering or welding. The permanent sealing substances make further reworking of the glass or metal very difficult.

Temporary Sealants to Locate Large Leaks in Vacuum Systems

Despite its drawbacks, the traditional technique of sealing suspected leak areas can sometimes succeed where other techniques fail It involves the application of a low vapor pressure sealant (usually vacuum putty or duct seal) to the suspected leak. The process is time consuming. It can cause difficulty in making a permanent leak repair unless the sealant is all removed with solvent before repairs are made. In no event should vacuum putty or other sealants be relied on for a permanent seal.

A leak can in effect be sealed by applying a forevacuum to the region external to the suspected leak. For example, a flange joint can be sealed with tape except for a gap at one point. A vacuum hose can then be pressed against this gap to evacuate the volume around the flange gasket. Although obviously limited in scope, this *overvacuum* technique can be useful in leak isolation.

Repairs of Large Leaks in Operating Vacuum Systems

If any general advice can be given about the repair of leaks, design can help considerably in reducing exposed areas.

Because the outgassing rate of elastomers increases as the temperature is raised, the ultimate pressure can be reached more rapidly if the elastomer can be heated. However, all elastomers are damaged when heated too much. Also, the compression set increases more rapidly with temperature.

Because of these properties, elastomeric gaskets are not normally used in ultrahigh vacuum systems. Such systems are baked at temperatures well above the damage point of insulator lacquers, sealing waxes, fast setting adhesives, epoxy coatings, vinyl plastic coatings, solder (and glycerine at liquid helium temperatures). In short, temporary leak seals are made with almost anything handy. Some techniques, e.g., epoxy, come close to being permanent repairs, but most temporary seals can be expected to give trouble at some further time. They can be a constant source of worry if not properly repaired when it first becomes possible to make a permanent seal.

The simplest leak to repair properly is a leaking flange gasket that can be sealed either by tightening the flange bolts a little more or by replacing the gasket. Most other leaks require reworking of the part. Leaking welds should be ground down to a smooth, clean surface before rewelding to help prevent the formation of a virtual leak under the new weld. In all cases, all vestiges of any temporary sealants used must be removed before starting a repair.

Sensitivity of Glow Discharge Color Leak Testing

The color differentiation technique will detect a gas pressure change of about 1 Pa (10 mtorr). The sensitivity of the technique is dependent on the pumping speed of the vacuum system as measured in the glow discharge area.

Limitations of Glow Discharge Color Leak Testing Technique

Part of the vacuum envelope of the system under test has to be transparent so that the change in color of the discharge can be seen when leaks exist. Because the procedure depends on detecting total tracer gas pressure buildup, the time that the test object has to be left standing before testing increases with an increase in desired leakage sensitivity. Any gas or liquid whose glow discharge color is different from the background discharge color may be used as a tracer. However, gasoline, benzene, pyridine and solutions containing nitrogen compounds should not be used as tracers because they adhere to glass.

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PART 7. Leak Testing of Vacuum Systems by Vacuum Gage Response Technique

Principles of Leak Testing by Vacuum Gage Response to Tracer Gases

The procedure of leak testing by vacuum gage response is based on the principle that most vacuum gages have a pressure response dependent on gas composition. If the composition of gas in a system changes, the reading on the gage reflect this change. Leak location therefore consists of spraying a tracer gas on the suspected leak and observing any response by the vacuum gage to the tracer gas that enters the system through the leak.

Most stainless steels used in vacuum work are called 18-8 stainless steels because they contain about 18 percent chromium and 8 percent nickel. These steels are nonmagnetic and the melting points of austenitic stainless steels are over 1400 °C (2550 °F). Surfaces of stainless steels remain smooth because oxides and hydroxides do not occur as in other types of metals. This means that the effective surface area is less and vapors are adsorbed in smaller quantities. This leads to much easier degassing and quicker pumpdown.

The vacuum gage leak test depends on maintaining a constant gas pressure in the system. If the system pressure varies for reasons unrelated to testing, leak location using pressure gages is impossible. The sensitivity of vacuum gage leak testing is relatively low (10^{-5} Pa·m^3·s^{-1} or 10^{-4} std cm^3·s^{-1}). The necessary instruments cannot be used in a contaminated atmosphere because they will respond to other gases present in the air. Therefore, these instruments are not widely used where welding (inert gases), cleaning (solvent fumes), brazing (combustion products) or painting (paint solvents) operations are performed.

Rubber and grease should be minimized, particularly in the connection link to the leak test gage being used as the detector, because they tend to absorb tracer gas (helium, halogens etc.) in the early phases of leak testing and outgas them later when high sensitivity is needed.

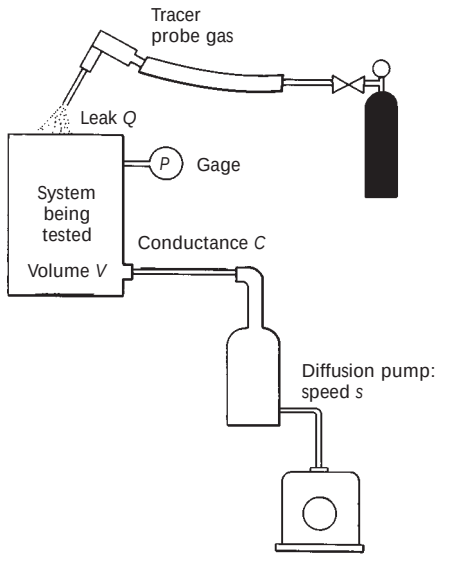
Procedures for Locating Leaks by Vacuum Gage Tests

In the evacuation mode, the system under test is evacuated and the suspected leak is sprayed with tracer gas (see Fig. 34). Pressure gage response to the tracer gas indicates that a leak has been located. The procedure is to expose small areas of the external pressure boundary surfaces of an evacuated system to a tracer gas. If a leak is present, this gas enters the evacuated system and displaces or mixes with any residual gas in the neighborhood of the gage. There are several variations of this procedure, depending on the vacuum gage used and the technique of increasing specificity, but the various techniques have a number of feature in common.

Application of Vacuum Gage Leak Testing

The vacuum gage leak testing procedure is extremely popular for leak location on vacuum systems because a pressure gage is usually built into the system. The only other requirement for the test is tracer gas. This procedure was once widely used for leak testing of components, but with the advent of more specific and more

FIGURE 34. Idealized system for vacuum gage response testing.



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sensitive leak detectors, it has fallen into disuse.

It is possible to use the vacuum gage response leak testing procedure for approximating leakage measurement on vacuum system. This is done by stabilizing the system, hooding it and introducing tracer gas into the hood. However, the response is not generally quantitative and is too nonspecific to be of much value. It is always questionable whether the pressure age response is due to increased concentration of the tracer gas or to some other factor.

Sensitivity of Vacuum Gage Leak Testing

The sensitivity of vacuum gage leak testing is dependent on the sensitivity of the absolute pressure gages being used and on the pumping system on which they are mounted. The leakage sensitivity is ordinarily in the range of 10^{-5} to 10^{-7} Pa·m^3·s^{-1} (10^{-4} to 10^{-6} std cm^3·s^{-1}). This can be increased by modifications that increase specificity of the gage response to the tracer gas.

In the tracer probe leak testing technique, the size of the leak that can be detected by a vacuum gage is dependent on the pumping speed of the system. As a first approximation, this procedure can detect a pressure change of one fiftieth of the pressure in the system. Smaller leaks, i.e., leaks that do not contribute more to system pressure or composition, will not be detected by this procedure.

Characteristics of Typical Vacuum Gages Used in Leak Testing

Many gages such as the Pirani and thermocouple gages use the thermal conductivity principle to measure pressure. These gages usually have a leak checking position on their meter scale. In this position, the pointer is in the center of the meter scale and operates at high sensitivity. Any movement of the pointer indicates a leak. Some instruments amplify the change of pressure indication of gages, which simplifies leak location procedures. Ionization gages are specifically modified for leak testing of evacuated systems.

Advantages of Leak Testing with Vacuum Gages

The major advantage of leak testing with vacuum gages on existing vacuum systems is that no additional leak testing equipment is necessary. Leak location may be performed using gages already on the system. The procedure is inexpensive and does not require highly trained test personnel. In the pressurizing mode, leak testing by thermal conductivity gage response is also an inexpensive technique of leak location. The equipment is portable and may be used on a variety of gases in the system.

Maximum Sensitivity of Leak Testing by Vacuum Gage Response

Maximum sensitivity will be obtained when the test includes (1) complete coverage of the leak by the tracer gas; (2) high sensitivity of the gage to the tracer gas; (3) low value of viscosity of the tracer gas; (4) a small effective pumping speed for the tracer gas; and (5) tracer gas with a high molecular weight.

Effect of Selection of Vacuum Pump

It is possible to use a small pump to evacuate the system being tested, but pressure fluctuation will be created. The pumping speed is more effectively reduced by using a large pump and a small conductance connection to the system. In practice, a turbomolecular or diffusion pump is preferable to a mechanical pump, because these pumps produce less pressure fluctuation. Of course, on a system with built-in pumps the pumping speed can not be altered for leak location, so the sensitivity is fixed by system design.

Effect of Molecular Flow In-Leakage on Vacuum Gage Response

For gage response for large leaks, it can be assumed that flow through the leak is laminar. In small leaks (10^{-7} Pa·m^3·s^{-1} or 10^{-6} std cm^3·s^{-1}), the flow will be molecular. In molecular flow, the leakage is inversely proportional to the square root of the molecular weight of the leaking gas. The same relationship applies to the conductance that determines the pumping speed of tubulation (see Eq. 27). If the leakage into the system is molecular and the pumping speed is determined by the tubulation leading to the pump, the pressure in the system is independent of the property of the leaking gas. The gage response is then dependent only on the relative sensitivity of the gage to the tracer gas as compared to air.

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Tracer Gas Pressure Sensitivity Factor for Vacuum Gages

Because there are a variety of factors involved in choosing a combination of proper tracer gas and vacuum gage, it is often easier to determine the sensitivity factor experimentally:

$$\phi = \frac{\text{Pressure caused by tracer gas on the leak}}{\text{Pressure on system with air on leak}} \quad (30)$$

The experimental values of this tracer gas sensitivity factor are listed in Table 6. The minimum detectable leakage can be determined from tracer gas sensitivity factor and leak testing conditions:

$$Q_{\min} = \frac{\Delta P_2 S_a}{\phi} \quad (31)$$

where ΔP_2 is smallest measurable air pressure variation, $Q_{\min}$ is smallest measurable leakage, S_a is pumping speed for air at the gage and ϕ is ratio defined by Eq. 30.

It is apparent from the above discussion that the minimum measurable leakage will be within a decade of the minimum measurable pressure change, multiplied by the pumping speed at the pressure measurement site. In designing this type of leakage measurement, the response time of the system must also be taken into account. The response time constant T_c of the system is the time for the leak indication to fall to $1/\epsilon$ or 36.4 percent to its maximum value.

$$T_c = \frac{V}{K S} \quad (32)$$

where V is the volume of the evacuated system, K is the ratio of effective pumping speed for tracer gas to pumping speed for air and S is pumping speed.

The testing techniques can be divided into three categories: (1) sealing techniques, (2) thermal conductivity techniques and (3) ionization efficiency techniques. These in turn have various subdivisions.

TABLE 6. Tracer gas sensitivity factor.

Tracer Gas	Hot Cathode Ionization Gage	Pirani Gage
Butane	10.0	1.0
Diethyl ether	5.0	0.7
Carbon dioxide	1.0	0.3
Carbon tetrachloride	1.0	0.05
Benzene	0.3	0.1
Hydrogen	0.4	0.4
Coal gas	0.25	0.25

Factors Affecting Sensitivity and Response Time of Vacuum Gage Leak Testing

The pumping speed S_a used in Eq. 31 is the pumping speed at the site of the gage. Thus, the location of the gage affects the sensitivity. If the gage is connected by way of a restriction, it will be difficult to detect small leaks anywhere except near the gage itself.

The response time depends primarily on the volume V of the system and on the effective pumping speeds at the test chamber, i.e., on the speeds S for air and KS for the tracer gas. The pumping speed of a turbomolecular or diffusion pump varies inversely as the square root of the molecular mass. The vacuum gage response will depend on the ratio of the leak detector response for air to its response for the tracer gas. The gage response will also depend on the ratio of the leakage rate for tracer gas to the leakage rate for air.

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PART 8. Leak Testing of Systems by Thermal Conductivity Techniques

Thermal Conductivity Technique for Leak Testing of Vacuum Systems

The thermal conductivity leak testing technique can be used with either the pressurized system (detector probe) technique or the evacuated system (tracer probe) technique. In the evacuated system mode of leak testing, gages normally found on the system are used. In the pressurized system mode, special leak detectors are necessary.

Tracer Probe Technique of Thermal Conductivity Leak Testing

The tracer gas detector for the tracer probe technique all evolved from thermal conductivity gages present on vacuum systems. Either thermocouple or Pirani gages normally mounted on the vacuum system are used for thermal conductivity leak testing by the tracer probe technique. Because these gages best respond to a pressure between 100 Pa and 10 mPa (1 torr and 0.1 mtorr), they are used on systems with low pumping speed. Alternatively, these gages can be placed between the turbomolecular or diffusion pump and the fore pump on a vacuum system. The thermal conductivity technique is very old, yet it is continually used in leak location on vacuum systems. New tracer fluids are used to enhance the technique and modifications are made on the pumping equipment to increase the leakage sensitivity.

Because the response of a thermal conductivity gage depends on the mass of the gas molecules, these gages can be used with a tracer gas to find leaks. When a leak is covered with a light gas such as helium, the gage will read higher than for an air leak. Conversely, a heavy gas such as argon will cause the gage reading to decrease. Volatile liquids such as acetone or alcohol can also be used but the response will depend on whether the vapors enter the leak or the liquid freezes in the leak, temporarily sealing it. One must keep in mind that (because of the fairly long response time of thermal gages) the leak may have been covered some time before the gage gives any indication. Hence, the leak may have to be located by successive approximations — a characteristic of most leak detection techniques. Because most vacuum systems will have either a thermocouple or Pirani gage to monitor fore pressure, these gages in the pressure range from 0.1 to 30 Pa (1 to 300 mtorr) are both simple and convenient.

Thermal Conductivity Leak Testing with Hydrogen Tracer Gas and Charcoal Trap

For example, if probing with hydrogen gas, an increase of tracer gas partial pressure may be obtained by reducing the turbomolecular pump speed with an in-bleed or reducing the diffusion pump speed by reducing the heater voltage. This decrease of hydrogen gas pumping speed is obtained without materially reducing the pumping speed for other gases. Modifications of this simple leak location technique are similar to those described later in this chapter for ionization gages. For example, in a Pirani leak detector using hydrogen gas, the gage is isolated from the system by a cooled charcoal trap. With this device it is possible to locate leaks as small as 10^{-7} Pa·m^3·s^{-1} (10^{-6} std·cm^3·s^{-1}).

Thermal Conductivity Leak Testing with Butane Tracer Gas

A differential leak detector for butane tracer gas uses two vacuum gages in a Wheatstone bridge circuit. One of the gages is in series with a charcoal trap. This arrangement has stability because any random pressure changes will be detected by both gages while the butane tracer gas will be absorbed by the charcoal. In this technique, the charcoal does not have to be heated during detection. The sensitivity of this differential system is reported to be 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}). Some thermal conductivity leak detectors are specifically designed for the detector probe technique.

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Thermal Conductivities of Different Tracer Gases

In principle, any tracer gas having a thermal conductivity different from that of air could be used with thermal conductivity leak detectors. The leakage sensitivity depends on relative differences of the thermal conductivities of the gases that are compared in Table 7. It is apparent that both hydrogen and helium show large relative differences and are therefore the most sensitive tracer gases with this technique. For special applications, it is sometimes desirable to use one of the other tracer gases. Table 7 gives some indication of results expected. It is clear that either gases with a thermal conductivity greater than air (such as helium, methane etc.) or those with thermal conductivities less than air (such as halogenated hydrocarbons, argon, carbon dioxide etc.) would be suitable.

Effect of Detector Probe Pumping Speed

The tracer gas emerging from leaks is drawn into sampling probes by the action of a small pump. The pump can be run at two speeds: a maximum speed for fast response and a slower speed to give an increased detection sensitivity at some sacrifice in response time. To obtain a good response, the thermal conductivity sensing elements must be small enough to fit in chambers of small volume. Because it is intended to detect changes in gas concentration rather than rates of flow, the gas should be made to flow past the entrance of the element chambers rather than through them.

Thermal Conductivity Leak Detector with Hot Wire Bridge Sensor

The thermal conductivity leak detector of Fig. 35 is based on a hot-wire bridge in

TABLE 7. Thermal conductivities of tracer gases for a temperature 20 °C (70 °F) in units of $W \cdot m^{-1} \cdot K^{-1}$ ($BTU \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1} \cdot ft$).

Gas	Chemical Formula	Molecular Mass (atomic mass units)	Thermal Conductivity[a] $W \cdot m^{-1} \cdot K^{-1}$	Thermal Conductivity[a] $BTU \cdot h^{-1} / ft^{2} \cdot °F \cdot ft^{-1}$
Air	mixture	29.9	0.025 57	0.014 78
Acetylene	C_2H_2	26.0	0.019 51	0.011 28
Ammonia	NH_3	17.0	0.023 06	0.013 33
Argon	A	39.9	0.017 58	0.010 16
Benzene	C_6H_6	78.0	0.009 31	0.005 38
Butane	C_4H_{10}	58.0	0.014 22	0.008 22
Carbon dioxide	CO_2	44.0	0.015 10	0.008 73
Carbon disulfide	CS_2	76.0	0.007 10	0.004 10
Carbon monoxide	CO	28.0	0.023 53	0.013 60
Ethane	C_2H_6	30.0	0.019 06	0.011 02
Ethylene	C_2H_4	28.0	0.017 73	0.010 25
Halogenated hydrocarbon F-11	CCl_3F	137.4	0.008 13	0.004 70
Halogenated hydrocarbon F-12	CCl_2F_2	120.9	0.009 58	0.005 42
Halogenated hydrocarbon F-21	$CHCl_2F$	102.9	0.011 42	0.005 54
Halogenated hydrocarbon F-22	$CHClF_2$	86.5	0.007 58	0.006 60
Halogenated hydrocarbon F-113	$CClF\text{-}CClF_2$	187.4	0.010 88	0.004 38
Halogenated hydrocarbon F-114	$CClF_2\text{-}CClF_2$	170.9	0.151 20	0.006 29
Halogenated hydrocarbon F-132				
Helium	He	4.0	0.186 32	0.087 40
Hydrogen	H_2	2.0	0.013 32	0.107 70
Hydrogen sulfide	H_2S	34.0	0.009 34	0.007 70
Krypton	Kr	83.8	0.032 39	0.005 40
Methane	CH_4	16.0	0.046 02	0.018 72
Neon	Ne	20.2	0.020 41	0.026 60
Nitric oxide	NO	30.0	0.025 29	0.011 80
Nitrogen	N_2	28.0	0.016 00	0.014 62
Nitrous oxide	N_2O	44.0	0.025 78	0.009 25
Oxygen	O_2	32.0	0.016 00	0.014 90
Propane	C_3H_8	44.0	0.025 78	0.009 25
Sulfur dioxide	SO_2	64.0	0.016 00	0.005 14
Water vapor	H_2O	18.0	0.018 81	0.010 87
Xenon	Xe	131.3	0.051 90	0.030 00

a. Thermal conductivity values for a temperature of 20 °C (70 °F) in units of $W \cdot m^{-1} \cdot K$ ($BTU \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1} \cdot ft$).

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which two resistance elements form two arms of the bridge network. One element is exposed to air containing tracer gas, while the other is exposed only to air and serves as a reference to compensate for changes in ambient conditions. As shown in Fig. 35, the sensing elements are mounted in a metal block inside a hand-held probe unit. Gas samples are drawn up through a narrow-bore tube. The sensing elements consist of coils of thin tungsten wire mounted on glass-metal seals in a compact assembly, into which the pump connects. The sensing probe is also fitted with a small meter to repeat the leak indication of the amplifier unit. Operators find this assembly to be convenient, particularly when testing awkwardly shaped equipment.

The electronic circuitry can be transistorized and thereby made compact enough for the unit to be hand held. The electronic components consist mainly of a stabilized power supply for the thermal conductivity bridge and an amplifier to increase and measure the amount of bridge unbalance. The electrical power source can be either batteries or line current. A four-step attenuator makes it possible to vary the sensitivity of the meter response by two decades.

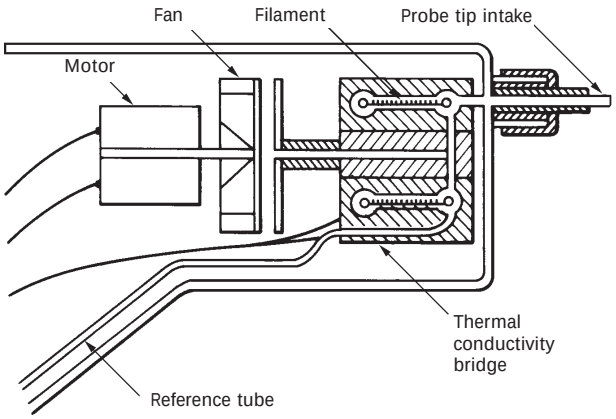
Leakage Sensitivity of Hot Wire Bridge Thermal Conductivity Tester

The minimum detectable leak, in terms of quantity of tracer gas per unit time, depends on the rate of flow of the gas through the leak detector and the minimum concentration to which the hot wire bridge detector will respond. By reducing the rate of flow, smaller leaks can be detected. However, there is a practical limit, because it is important in leak location that the detector should respond quickly when the probe traverses the position of the leak. Reducing the rate of flow of tracer gas lengthens the response time and beyond a certain point the indications from the leak detector become meaningless.

The detector shown in Fig. 35 can detect a 60 $\mu L \cdot L^{-1}$ concentration of hydrogen gas. This gives a response at one tenth of full scale, with a pumping speed for the probe of 0.13 $cm^3 \cdot s^{-1}$ (0.5 $in^3 \cdot min^{-1}$). The instrument will detect an 8×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (8×10^{-6} std $cm^3 \cdot s^{-1}$) hydrogen leak. With argon, which has a much lower thermal conductivity difference from air, only a 1.3×10^{-5} $Pa \cdot m^3 \cdot s^{-1}$ (1.3×10^{-4} std $cm^3 \cdot s^{-1}$) leak can be detected.

When testing with the hot wire bridge thermal conductivity detector, the atmosphere must be free from tracer gas. If a system with very large leaks is being tested, the local atmosphere may become contaminated with tracer gas. Although this will be inherently balanced out by the reference circuit, ultimate leakage sensitivity is bound to decline.

FIGURE 35. Thermal conductivity leak detector using two hot wire detectors in a Wheatstone bridge arrangement.



Advantages and Limitations of Hot Wire Bridge Leak Detector

The relatively low operating temperature of the filaments makes the hot wire bridge leak detector quite safe to use under most industrial conditions. The functional life and long-term stability of the sensing elements are good. The only effect that has been noted after long periods of operation under industrial conditions was the accumulation of a dust deposit in the intake line, which was easily removed. Unfortunately, this versatility is also a disadvantage. Because of a lack of selectivity, this instrument can not be operated at high sensitivity in atmospheres contaminated with other gases.

The thermal conductivity bridges used in these detectors do not actually measure thermal conductivity. Because of their structure, the readings obtained with these detectors are dependent on tracer gas thermal conductivity combined with density, accommodation coefficient and viscosity. Therefore, the values of sensitivity inferred from thermal conductivities of Table 7 are not absolute, but merely an indication of the expected general trend in the results.

A thermal conductivity detector, similar to that of Fig. 35, uses a four-element wire bridge. This bridge was also found useful for vacuum leak detection. The sensitivity of this type of leak detector was improved by use of thermistors, with their higher thermal coefficient of resistance, instead of wire elements. These detectors were tested in submarine service, where they were found useful in detecting leaks of a variety of gases.

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PART 9. Leak Testing of Vacuum Systems by Ionization Gage or Pump Techniques

Ionization Gage Technique of Leak Testing of Vacuum Systems

The ionization gage technique of leak testing of pumped vacuum systems involves spraying the suspected leak area with tracer gas and observing any pressure change indicated on an ionization gage. Any gage that measures ionization of the gas may be used; this can be either a hot cathode gage, a cold cathode gage or even an ion pump.

In ionization gages, ionization current depends on the probability of ionizing collisions. With all other variables held constant, this probability of ionization varies from one gas to another. When the tracer gas is applied to the leak, some of the gas in the gage is replaced by tracer gas that causes an ionization current either lower or higher than the steady ionization current due to the prevailing pressure in the system in the absence of tracer gases.

As long as the leaks being located are the ones that limit the system pressure, the ionization gage technique may be applicable to very low pressures and/or very low leakage rates. It has been used for location of leaks in ultrahigh-vacuum systems. On very small volume systems, this technique is reported to be more sensitive than the mass spectrometer leak detector.

Use of Ionization Gages As Leak Detectors for Vacuum Systems

As described above, ionization gages respond differently to different gases. For example, if first air and secondly helium are admitted through a small (molecular flow) leak into a system using diffusion pumps, then the ionization gage response to the helium will be about 15 to 20 percent of the response to air. In this case the actual pressure in the system will be virtually unchanged. This follows because both the leakage rate and the pumping speed vary in the same way. Both are inversely proportional to the square root of molecular mass. The decreased response for helium is due to the fact that the ionization potential of helium is much higher than the ionization potentials of nitrogen, oxygen or air. On the other hand, the ionization potentials of hydrogen and carbon are somewhat lower than that of air and indeed the response of an ion gage to hydrogen and hydrocarbon compounds such as acetone, alcohol or butane is greater than that of air. The application of this behavior to leak detection is obvious. In practice, one usually adjusts the grid-current control until the ion gage reads near full scale to obtain maximum sensitivity. Then the system is probed with one of the tracer gages or vapors mentioned while monitoring the reading of the ionization gage.

Effect of Tracer Gas Properties on Ionization Leak Test Sensitivity

It is desirable that the ionization efficiency of the tracer gas be as different as possible from that of the background gas (air). In general, gage sensitivity increases with the number of electrons in the molecule. Examination of ion gage sensitivities suggests that the best gases for this technique are either the low molecular weight gases such as hydrogen, helium and neon or the high molecular weight vapors such as acetone, ether and alcohol. In using the vapors, care must be taken that they do not plug the leak. In some cases, response may be delayed because of adsorption of vapors on the interior surface of the leak.

Care must be taken that the tracer gas does not permanently react and change the gage sensitivity. For example, applying carbon dioxide for a time can change the sensitivity of a Penning gage. The discharge current decreases about 30 to 40 percent probably because of a film of carbonates on the electrodes. This general technique can be modified in several ways. Instead of an ionization gage, an ion pump may be used. Selectivity of the gage to the tracer gas may be increased by use of a double gage setup, where a gage is positioned so that it is selective only to the tracer gas. Another modification of this technique is to use the poisoning effect of oxygen on the emission of electrons from a tungsten filament.

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Sensitivity of Ion Gage Leak Detection in Vacuum Systems

Several conditions can reduce the sensitivity of the ion vacuum gage leak detection technique. If several leaks are present in the system, the differential response of the gage will be smaller than for a single leak. The response time of a large systems may be comparable with the fluctuations or drift that may be present on the normal gage reading. In such cases it is difficult to tell when a leak has actually been encountered. If a leak is definitely suspected in one location, the signal-to-noise ratio can be improved somewhat by alternately probing with helium and acetone.

The sensitivity of the ionization gage technique can be greatly improved by commercially available leak detection devices that attach to the recorder terminals of most ionization gage and ion pump circuits.

Leak Detector with Magnetron Ionization Gages

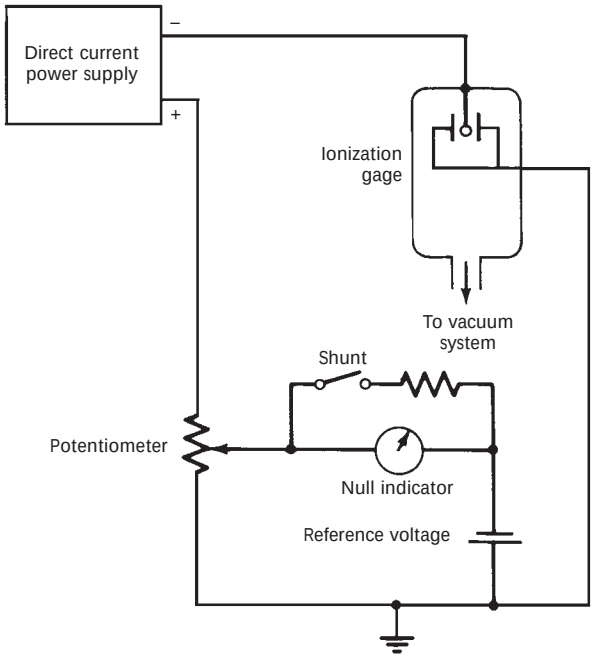
Another leak detector uses two magnetron ionization gages enclosed as a unit of the same general dimensions as the mass spectrometer leak detector analyzer section. The two ionization gages are connected in series, with the second gage cryogenically trapped. The two gages are balanced on a bridge circuit. Tracer gas changes the current of the first gage, but is condensed and therefore does not affect the second gage. With two gages, background pressure variations do not affect the detector. The leakage sensitivity of this magnetron ionization detector is reported to be 10^{-11} Pa·m^3·s^{-1} (10^{-10} std cm^3·s^{-1}).

Differential Ionization Gage Leak Detection Instrumentation

To obtain adequate leakage sensitivity with the ionization gage technique, the background ionization current may be nulled using a sensitive difference amplifier or a galvanometer with backing off voltage control, so that very small changes in ionization current are detected. An example of a circuit for such testing is shown in Fig. 36. The indicating instrument has been replaced with a potentiometer. The null-balance instrument can be a galvanometer or a sensitive microammeter, either of which is provided with a suitable shunting circuit. In a stable vacuum, constant current flows through the gage tube and the potentiometer, creating a steady voltage drop across the potentiometer. The battery provides a reference voltage and the potentiometer can be adjusted to give a null indication on the galvanometer. The shunting switch is left closed until this adjustment is made.

As shown in Fig. 36, the null set potentiometer devices can compensate the gage current due to the air leak (i.e., provide a counter current adjusted to give a null reading) and then amplify any variations from null. The result is a great magnification of pressure variations too small to be detected on the meter of the ion gage. Noise and drift variations, which are magnified as well, set the practical limit to the sensitivity obtained by using these devices. Small leaks can sometimes be located in the presence of a pressure drift if the output of the ion gage leak detector is monitored with a strip chart recorder. The location of the leak is indicated by the change in slope of the drift curve. For stable systems, the ion gage leak detector can detect a 1 percent change in the pressure reading of the gage circuit. Because this sensitivity approaches or exceeds that of the helium mass spectrometer leak detector for pressures below 1 μPa (10 ntorr), the ionization gage technique is often used with vacuum systems operating in the ultrahigh

FIGURE 36. Null balance circuit for leak location with an ionization gage leak detector.



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vacuum range. But if very small leaks must be found at moderate vacua, about 100 µPa (1 µtorr), as for example in the leak testing of an ultrahigh vacuum system bakeout, then a mass spectrometer detector must be used.

Selective Tracer Gas Transmission Leak Testing with Ionization Gage

The sensitivity of an ion gage to tracer gas can be increased if air is excluded and the tracer gas only is selectively brought to the ion gage. If this is done, the gage will not respond to extraneous pressure changes. Selectivity can be increased by use of a selective membrane or a cryogenic trap in front of the gage. For example, palladium metal passes only hydrogen gas. On the other hand, silica gel passes not only hydrogen but the noble gases (helium, neon and argon). Neither palladium nor silica gel will pass air through the barrier wall. A cryogenic cold trap can collect hydrocarbon vapors that condense with it, so they cannot form interfering carbon layers on barriers of ionization gage components.

Palladium Barrier Ionization Gage for Detecting Leaks in Vacuum Systems

The palladium barrier gage is typical of several that have the property of selective allowing hydrogen to pass into a vacuum gage, to the exclusion of all other gases. It uses the fact that hot (about 800 °C or 1470 °F) palladium metal is permeable to hydrogen but not to other gases. As shown in Fig. 37, the palladium barrier gage is in essence an ionization gage with a palladium barrier between it and the vacuum system. The palladium is heated either by electron bombardment or by conduction from a hot filament. The gage is evacuated, sealed off and gettered to achieve a very low pressure in the gage itself. The gage can be placed in the foreline of the system; because only the hydrogen passes through the barrier, the pressure in the gage is just the partial pressure of this hydrogen tracer gas alone. It is claimed that this device can detect changes as small as 3 µPa (20 ntorr) in the partial pressure of hydrogen and some claim to have detected leaks as small as 5×10^{-11} Pa·m^3·s^{-1} (5×10^{-10} std cm^3·s^{-1}). However, sensitivities corresponding to leakage rates in the range 10^{-7} to 10^{-8} Pa·m^3·s^{-1} (10^{-6} to 10^{-7} std cm^3·s^{-1}) are more normal in actual practice.

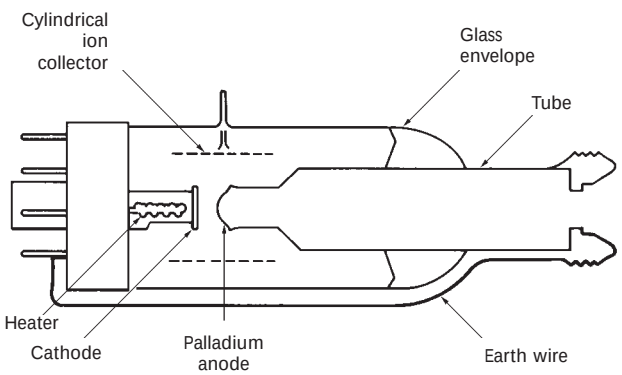
Leakage Sensitivity of Palladium Barrier Ionization Gage

The direction and rate at which hydrogen passes through the palladium barrier depends on the hydrogen pressure differential across the barrier. Thus, hydrogen in the gage volume can be removed by reducing the external hydrogen pressure below the internal value. The gage can detect a pressure change of about 3 µPa (20 ntorr), but must be operated under carefully controlled conditions to achieve this sensitivity. Use has been made of a hydrogen generator consisting of a hot tungsten filament that decomposes oil vapors present in the vacuum system. To obtain maximum leak detection sensitivity, it is sometimes found necessary to maintain a hydrogen partial pressure in the system of about 40 µPa (0.3 µtorr) by glowing the tungsten filament at temperature of about 800 °C (1470 °F).

Precautions with Palladium Barrier Ionization Gage

It is necessary to place a liquid nitrogen trap between the palladium barrier ionization gage leak detector and the rest of the system to exclude hydrocarbons and water vapor from the gage. These vapors dissociate at the hot palladium surface to give hydrogen, which produces a spurious response. In addition, the cracked hydrocarbons build up a carbide layer on the palladium, which reduces its permeability. It is also desirable to use a turbomolecular pump with oil free bearings rather than an oil diffusion pump in the vacuum system; otherwise, the hydrogen that results from the decomposition of diffusion pump oil gives rise to an unstable background ion current in the gage. In a system containing multiple leaks, oxygen in the air entering the undetected leaks

FIGURE 37. Palladium barrier ionization gage.



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combines at the hot palladium surface with the hydrogen entering through a leak that is being probed. If there is an excess of oxygen, all hydrogen will react with the oxygen before it can pass through the barrier and will therefore be undetected. Under these circumstances, a controlled leak of hydrogen could be admitted to the system to take up the oxygen.

If air is admitted to the ion gage, the palladium becomes oxidized even if it is cold. Whenever this occurs, 2 to 3 h of run-in time is required to obtain reproducible results on duplicate runs. Therefore, even if the gage is not in use, the forepumps should be operated continuously to prevent air contact with the palladium. If the gas is left exposed to the atmosphere, several warm-up runs should be made to allow hydrogen to pass through the calibrated leaks and be pumped down between successive runs.

Vacuum Leak Testing with Cryogenically Trapped Gage with Silica Gel Absorbent instead of Palladium

It is possible to use an absorbent to pass the tracer gas and block air. Silica gel, outgassed at 300 °C (570 °F) and then cooled to liquid nitrogen temperatures, is commonly used for this purpose. Under these circumstances, silica gel readily passes hydrogen and the noble gases (helium, neon, argon), but not air. The system uses a cold cathode gage and hydrogen. The gage is separated from the system by a liquid nitrogen cold trap filled with silica gel.

Sensitivity of Silica Gel Absorbent Leak Testing

When silica gel is used in the cold trap, the ionization gage leakage sensitivity is claimed to be about a hundred times greater than that of the palladium hydrogen system. However, several hours are required to measure leakage rates of the order of 10^{-13} Pa·m^3·s^{-1} (10^{-12} std cm^3·s^{-1}). Careful degassing of the leak detector and the tube to be tested is necessary. One advantage claimed for silica gel is a long usage time before it has to be degassed again. The increased sensitivity of silica gel is claimed to be due to less gas evolution from the gel than from heated palladium, which results in lower pressures. This detector, although very sensitive, is limited by long pump down times.

Ion Pump Technique of Vacuum System Leak Detection

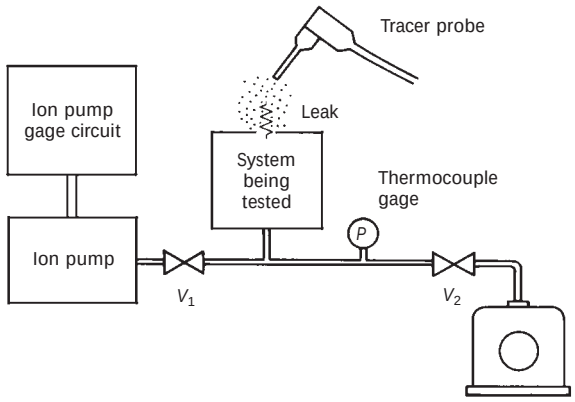
Cold cathode, gas discharge ion pumps are convenient instruments for leak location. An ion pump acts not only as a pump but also as an effective pressure gage, because the pump current is proportional to the number of molecules being pumped. The pump current is also dependent on the ionization efficiency of the gas molecules being pumped. The pumping speed is dependent on the ionization efficiency of the gas molecules being pumped. The pumping speed is dependent on the molecular chemical reactivity rather than the molecular weight, so the response of an ion pump to a tracer gas will be different from the response of an ionization gage. A typical arrangement for ion pump leak testing of evacuated systems is shown in Fig. 38.

Effect of Tracer Gas on Leakage Response of Ion Pump

The response of an ion pump to various probe gases is shown in Fig. 39. As may be seen from those curves, the response differs with time, not only in magnitude, but also occasionally in sign. The best gases for leak location using an ion pump seem to be argon, oxygen and carbon dioxide.

The pumping speed of an ion pump depends strongly on the chemical activity of the gas being pumped. Unlike a turbomolecular pump or a diffusion pump, the pumping speed of an ion pump varies with chemical species rather than with molecular mass. The actual pressure in an ion pumped vacuum system will thus vary as different gases are introduced via a molecular flow leak.

FIGURE 38. Ion pump leak detector arrangement.



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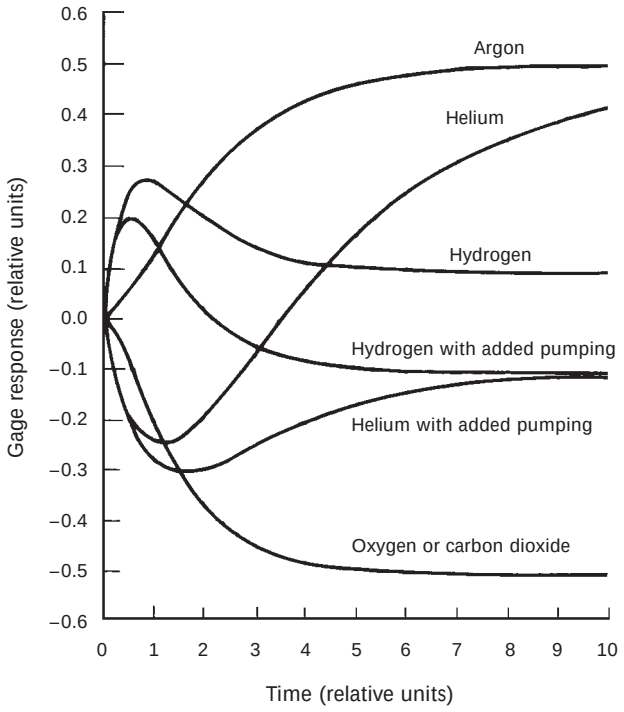
Therefore, if the ion gage is mounted on an ion pumped system, the change of its indicated pressure in response to a change in gas composition will be markedly different from that of the same gage on a diffusion pumped system. It is customary to use the ionization current in an ion pump as a measure of the pressure in the pump. The response of such an ion pump pressure gage is similar to that of an external thermionic ionization gage. Both types of gage show an increase in pressure when either argon or helium enters the vacuum system. A pressure decrease is indicated when oxygen or carbon dioxide enter. Thus, these gases can be used to detect leaks in ion pumped systems in the same manner as the ionization gage described just previously. However, note that the two types of pumps give opposite responses for helium.

Null Set Circuit for Ion Pump Leak Detector

The response amplifier type of ion gage leak detector circuit sketched in Fig. 40 can be used with the recorder output of the circuit associated with either an ion pump or a thermionic ion gage. The pressure fluctuations (noise) or an ion pumped system are usually somewhat less than for a turbomolecular or diffusion pumped system, unless the ion pump is experiencing argon instability (burping).

FIGURE 39. Response of an ion pump gage indication to leaks of various gases.



The sensitivity of an ion gage leak detector on a system using ion pumping is shown in Fig. 41 as a function of pressure.

FIGURE 40. Schematic circuit diagram of an ion pump leak detector.

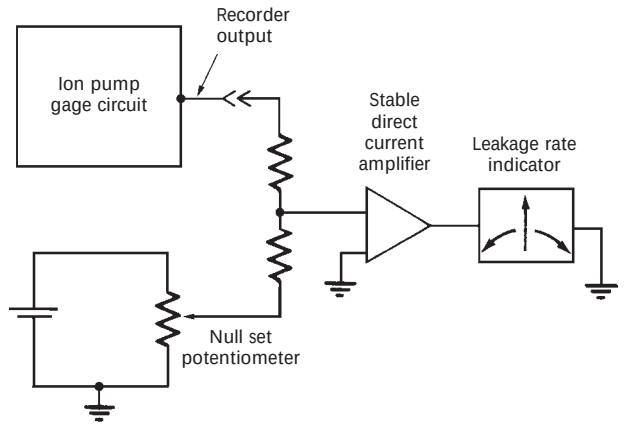
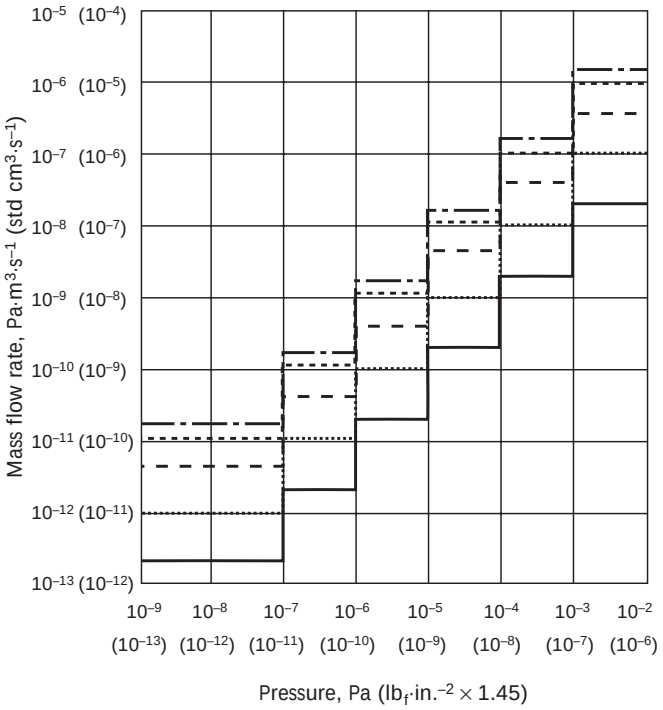


FIGURE 41. Minimum detectable leakage rate as a function of pressure for vacuum systems with ion pumping.



Legend

—·—·— = 400 L·s⁻¹ (850 ft³·min⁻¹)
····· = 125 L·s⁻¹ (265 ft³·min⁻¹)
– – – = 75 L·s⁻¹ (160 ft³·min⁻¹)
····· = 40 L·s⁻¹ (85 ft³·min⁻¹)
——— = 8 L·s⁻¹ (17 ft³·min⁻¹)

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Leakage Sensitivity of Ion Pump Technique

Leaks in the 10^{-12} Pa·m^3·s^{-1} range may be located with an ion pump. This is a conservative estimate of the sensitivity; the current changes being measured are several orders of magnitude greater than the corresponding mass spectrometer ion currents. With the ion pump leak detector system shown in Fig. 38, the procedure is to evacuate an ion pump and keep it operating at low pressure with the valve V_1 closed. The system to be leak tested is first evacuated by a mechanical pump to a pressure of about 1 Pa (7 mtorr). Valve V_1 is then opened and V_2 closed until an equilibrium pressure is reached (a few minutes). When the leak is probed with argon, the ion pump current should increase rapidly, presumably due to the low speed of the pump for argon. Probing with hydrogen and oxygen causes a reduction in pressure, because these gases are pumped more rapidly than air. With helium used as the search gas, the sensitivity is lower than for argon.

Leaks as small as 10^{-11} Pa·m^3·s^{-1} (or 10^{-10} std cm^3·s^{-1}) are located using the ion pump technique. Leaks between 10^{-4} and 10^{-6} Pa·m^3·s^{-1} (10^{-3} and 10^{-5} std cm^3·s^{-1}) could be located by partial opening V_1 and by having V_2 opened sufficiently to avoid a pressure increase in the system during the leak testing procedure. Leaks of 10^{-6} to 10^{-7} Pa·m^3·s^{-1} (10^{-5} to 10^{-6} std cm^3·s^{-1}) could be determined a few minutes after opening V_1 and closing V_2. Leaks smaller than 10^{-9} Pa·m^3·s^{-1} (10^{-8} std cm^3·s^{-1}) required a longer time, depending on the volume and outgassing properties of the item under test.

Leak Detection by Reduction of Thermionic Electron Emission by Oxygen Tracer Gas

A very sensitive means of locating leaks in vacuum systems is to observe the temperature limited emission of electrons from a heated tungsten filament in a vacuum. When a stream of oxygen tracer gas is blown over the outside of a leak, the resulting increase in oxygen pressure within the vacuum system causes the filament's emission to drop. Although the principle has been known for a long time and various circuits have been developed for its use, this technique has not been extensively used. An instrument in which the grid of a triode ionization gage is connected externally to the collector to form a diode is used to detect oxygen admitted to the apparatus under controlled conditions. A leak that gives rise to a partial air pressure of 10 µPa (0.1 µtorr) is readily detected when probed with oxygen. The detection circuit used is a modified ionization gage control unit. The filament is heated by a regulated power supply, but is not emission regulated. For stable operation of this type of detector, using thoria coated tungsten filaments, it is best to reduce the thoria to thorium at the beginning of the test by heating the filament for a few seconds to a temperature of 2400 K (3860 °F).

Sensitivity Characteristics of Thermionic Electron Emission Oxygen Leak Detector

The greatest sensitivity to oxygen tracer leakage is at an operating temperature just below 1900 K (2960 °F), when the tungsten surface is partly covered with thorium. This can be obtained only when leaks of 10^{-10} Pa·m^3·s^{-1} (10^{-9} std cm^3·s^{-1}) or less are remaining in a well baked system pumped at a speed of 10 L·s^{-1} (21 ft^3·min^{-1}). The filament can become desensitized when it becomes carburized. It is because of the danger of carburization in the presence of hydrocarbon vapors and because of the influence of residual water vapor on the emission of electrons from the thoriated tungsten, that the detector is not very suitable for use in leak testing of unbaked vacuum apparatus. If a filament becomes carburized accidentally it must be replaced; no thermal treatment cycle will bring it to a sensitive state again. But in a well baked system, thoriated tungsten filaments can, if necessary, always be restored to a desired state of sensitivity again by a short period of running at a temperature of about 2400 K (3860 °F).

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CHAPTER 7

Bubble Testing

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PART 1. Introduction to Bubble Emission Techniques of Leak Testing

Principles of Bubble Testing for Leaks

In leak testing by the bubble test technique, a gas pressure differential is first established across a pressure boundary to be tested. A test liquid is then placed in contact with the lower pressure side of the pressure boundary. (This sequence prevents the entry and clogging of leaks by the test liquid.) Gas leakage through the pressure boundary can then be detected by observation of bubbles formed in the detection liquid at the exit points of leakage through the pressure boundary. This technique provides immediate indications of the existence and location of large leaks, 10^{-3} to 10^{-5} Pa·m^3·s^{-1} (10^{-2} to 10^{-4} std cm^3·s^{-1}). Longer inspection time periods may be needed for detection of small leaks, 10^{-5} to 10^{-6} Pa·m^3·s^{-1} (10^{-4} to 10^{-5} std cm^3·s^{-1}), whose bubble indications form slowly.

In bubble tests, the probing medium is the gas that flows through the leak due to the pressure differential. The test indication is the formation of visible bubbles in the detection liquid at the exit point of the leak. Rate of bubble formation, size of bubbles formed and rate of growth in size of individual bubbles provide means for estimating the size of leaks (the rate of gas flow through leaks).

Classification of Bubble Test Techniques According to Test Liquids

Bubble test techniques for detecting or locating leaks can be divided into three major classifications related to the technique of using the test liquid:

1. In the liquid immersion technique, the pressurized test object or system is submerged in the test liquid. Bubbles are then formed at the exit point of gas leakage and tend to rise toward the surface of the immersion bath.
2. In the liquid film application technique, a thin layer of test liquid is flowed over the low pressure surface of the test object. An example of this solution film leak test is the well known soap bubble technique used by plumbers to detect gas leaks. Films of detection liquid can be readily applied to many components and structures that cannot be conveniently immersed in a detection liquid. For detection of small leaks, this liquid should form a thin, continuous, wetted film covering all areas to be examined.
3. The foam application technique is used for detection of large leaks in which the applied liquid forms thick suds or foam. When large leaks are encountered, the rapid escape of gas blows a hole through the foam blanket, revealing the leak location.

Classification of Bubble Test by Pressure Control

Subclassifications of these basic techniques of bubble testing refer to different techniques for controlling the pressure differential acting across the pressure boundary. Several techniques are used to raise the pressure differential and so to increase the rate of gas leakage and the rate of formation of bubbles.

1. Pressurize the interior volume of the test object or system before and during the leak test. Internal gas pressure should be applied across the pressure boundary before test liquid contacts the external surface. This tends to prevent entry of liquid into leaks, which might possibly clog the leaks to gas flow. Protection against hazards of overpressure must be provided.
2. Control the heating of sealed test objects and small components to cause internal gas expansion. This increases the pressure differential and causes outward gas flow through possible leaks in the pressure boundary.
3. Apply a partial vacuum above the surface of the test liquid (immersion liquid or solution film). This reduces external pressure to the pressure boundary. The resultant increase in pressure differential across the system boundary acts to cause gas flow through any leaks that are present.

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Advantages of Bubble Testing

Bubble testing has the obvious advantages of being relatively simple, rapid and inexpensive. It is a fairly sensitive leak detection technique and enables the observer to locate the exit points of leaks very accurately. (The point of exit may not be directly opposite the entry point of the leak, especially in welds or castings.) Another major advantage of bubble testing is that very large leaks can be detected readily. Bubble test techniques also provide very rapid responses even for small leaks. (Some more sensitive leak testing techniques often have responses so slow that a leak may be missed while probing.) With bubble tests, it is not necessary to move a tracer probe or detector probe from point to point. In immersion bubble tests, the entire pressurized component can often be examined simultaneously for leaks on exposed surfaces visible to the observer. In some cases, test components may have to be turned over to expose the underside to view, so that leaks from this area can be seen. All leaks are revealed independently in immersion bubble testing. If desired, large leaks can be first detected with rapid bubble test techniques. These leaks can then to sealed before refined leak testing apparatus is used to detect smaller leaks.

The bubble testing technique lets the observer distinguish real from virtual leaks. (Virtual leakage is a primary problem in leak testing of vacuum systems but may also be encountered when bubble testing.) In addition, during bubble tests it is not necessary that all connection pipes and valves be free from leaks. However, detection of small leaks requires operator patience and additional test time for bubble or foam indications to form. Care is required to ensure that all detectable bubble indications present are observed. Bubble testing is satisfactory for detecting gross leakage. With inert probing gases and test liquids, bubble tests are fairly safe in a combustible atmosphere. However, this depends on selection of proper tracer gas and test liquids. The required level of operator training and skill is minimal, compared with some more complex techniques of leak testing.

Limitations of Bubble Techniques of Leak Testing

Conditions that interfere with bubble emission techniques of leak testing or limit their effectiveness include the following: (1) contamination of test specimen surfaces; (2) improper temperatures of test specimen surfaces; (3) contaminated or foaming test liquids; (4) improper viscosities of test liquids; (5) excessive vacuum over surface of test liquid; (6) low surface tension of test liquids leading to clogging of leaks; (7) prior use of cleaning liquids that clog leaks; (8) air dissolved in test liquids or outgassing from corroded test surfaces, causing spurious bubble formations; and (9) leaks with directional flow characteristics, intermittent or very slow leakage or porosity leaks.

Prior bubble testing or contamination may clog leaks and lower the sensitivity of subsequent leak testing by more sensitive techniques.

Effects of Test Surface Contamination, Porosity or Temperature

Surface contamination of the test specimen can occur with small immersed test parts or on scaled, dirty or greasy surfaces of large vessels or components. Grease, rust, weld slag, oxide films or other surface films, as well as weld porosity open to a surface may be sources of bubbles giving false indications of leakage. Temporary plugging of leaks might also occur because of some common manufacturing techniques such as peening or metal smearing that closes the openings to leaks at metal surfaces. Leak testing must be done before painting, galvanizing, coating or plating of surfaces, which may plug leaks temporarily. Difficulties can also result when tests are performed with test specimen surface temperatures either too high or too low for inspection procedure requirements.

Effects of Properties and Contamination of Bubble Test Liquid

Contaminated test liquids or test liquids that foam on application can cause formation of spurious bubbles on test specimens, which is not related to leakage through the pressure boundary. Incorrect viscosity of the test fluid can also affect formation of visible streams of bubbles at leaks. Formation of spurious bubbles caused by air dissolved in water or other immersion liquids hinders detection of bubble emission from real leaks. When bubble tests are conducted on metallic vessels, some bubbles can evolve from outgassing from patches of corrosion.

Effects of Excessive Vacuum over Bubble Test Liquid

Excessive vacuum on the low pressure side of the pressure boundary of test objects

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could occur when using the vacuum box pressure differential technique of bubble testing. Excessive vacuum (absolute pressure too low over the test liquid) can lead to boiling of the detection liquid. When the immersion liquid is boiling, bubbles of vapor form throughout the solution and typically rise to the liquid surface. These could interfere with operator detection and observation of bubble formation caused by leakage. The amount of vacuum allowed in immersion bubble testing depends on the immersion test liquid. It should be the maximum vacuum attainable without causing the test liquid to boil.

Effects of Low Surface Tension of Bubble Test Liquid

Clogging of small leaks with leakage rates less than 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) can result from premature application of the test liquid, either by immersion or film solution. Most bubble testing solutions have a low surface tension. Detection solutions with low surface tension promote surface wetting. This increases the tendency of the test liquid to enter and block very small leaks. This tendency can be reduced, however, if the vessel or test component is always pressurized before covering the surface under test with any liquid. Clogging of existing leaks could also occur if the test liquid used in bubble emission tests enter the leaks after an external vacuum is released.

Effects of Prior Surface Cleaning of Test Objects

Prior use of cleaning liquids on test object surfaces can also result in clogging of leaks. Thus, all test objects must be thoroughly dried by heat or vacuum or both, after cleaning with liquid solutions before leak testing with gaseous tracers.

Effects of Porosity, Intermittent Leaks and Check Valve Leaks

Leaks with special characteristics may react in ways such that they cannot always be found reliably by bubble tests. For example, porosity leaks cannot be detected by bubble tests if the pores are very small. Some types of leaks may pass gas in only one direction; if this direction is inward, bubble tests of outside surfaces will not detect them. With intermittent or very slow leaks, close operator surveillance of the test surface is often necessary to detect bubbles.

Importance of Cleaning Test Surfaces after Bubble Testing

Cleaning of test object surfaces and drying of test objects to remove all bubble test liquids from within leaks is essential when these same test objects are subsequently subjected to more sensitive leak tests with gas tracers (such as halogen vapor or helium leak tests). The later gas tracer leak tests could be invalidated if prior bubble testing had clogged the leaks with water or other liquids.

Factors Influencing the Sensitivity of Bubble Testing

As noted earlier in this chapter, the basic principle of the bubble test consists of creating a pressure differential across a leak and observing bubbles formed in a liquid medium located on the low pressure side of the leak or pressure boundary. The sensitivity of the bubble test technique can be influenced by factors such as (1) pressure differential acting across the leak; (2) viscosity of pressurizing tracer gas; (3) test liquid used for bubble formation; (4) contamination on surfaces being tested (i.e., paint, dirt, oil etc. on inside or outside surface of object being tested); (5) ambient weather conditions (such as rain, temperature, humidity or wind); (6) lighting in test area; (7) test equipment; and (8) test personnel technique and attitude.

Properties Affecting Leak Detector Solution Performance

1. *Surface tension* affects the speed and size of bubble formation. Lower surface tension solutions form many small bubbles and the reforming of new bubbles. Higher surface tension solutions slowly form very large bubbles that are slower to break, but usually do not reform new bubbles. Water softener is used to reduce surface tension.
2. Good *wetting action* and a large *contact angle* are the result of lower surface tension. Poor wetting action and a small contact angle are the result of higher surface tension.
3. *Viscosity* affects the size of bubble growth. Lower viscosity solutions produce smaller bubbles. Higher viscosity solutions produce larger bubbles. Glycerine may be used to control viscosity.
4. *Evaporation rate* controls the amount of test area that may be covered with leak detector solution before the final inspection. It is desirable therefore to

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use a solution that has a slow evaporation rate to be able to cover a larger test area. Evaporation rate is also temperature dependent with an increase in temperature causing an increase in evaporation rate and vice versa.

Techniques for Attaining Required Bubble Test Sensitivities

As long as the pressure differential can be maintained, the bubble test technique can be used. However, the sensitivity of a leak testing procedure must be adequate to permit detection of all leaks of a certain size and larger so that all detected leaks can be repaired. The hole or crack that constitutes the physical leak is usually characterized for size of leak by the amount of gas passing through it as leakage. The sensitivity of a bubble test can be increased by (1) increasing the time allowed for bubble formation and observation, (2) improving conditions for observing bubble emission and (3) increasing the amount of gas passing through the leak.

Improving Bubble Test Sensitivity by Better Observational Capabilities

The actual sensitivity of a specific leak test procedure can be improved by an increase in observational ability. An increase in observational ability could be attained by the following means.

1. Position test surfaces optimally for visual inspection.
2. Improve lighting to highlight bubble emission clearly and use clean translucent immersion liquids.
3. Increase time for bubble formation and observation by test operators.
4. Eliminate false bubble indications (caused by boiling, entrained air or contamination of inspection liquids, for example).
5. Decrease surface tension of the detection liquid that causes more and smaller bubbles to appear.
6. Reduce pressure above the inspection liquid, which makes the individual bubbles larger.
7. Select test site and time to provide optimum ambient conditions, such as temperature, wind and lighting conditions.
8. Use leak detector solutions that are fluorescent and colored for increased contrast with different test surfaces.

Factors affecting operator comfort and ability to see bubble indications must also be considered. Tests might be postponed until proper test conditions can be attained.

Each of these aids to sensitivity enables the test operator to detect the bubble emissions from smaller leaks or to separate the indications for closely adjacent leaks more readily and so improve the reliability of leak detection.

Increasing Bubble Test Sensitivity by Raising Tracer Gas Flow Rate

Increase in sensitivity resulting from improvements in leak test procedures are typically attained by raising the rate of flow of tracer gas through the existing leaks. The increased amount of gas flow through the leak passageway may be attained by a change in the properties of the gas (lower gas viscosity or lower mass). Alternatively, the quantity of gas passing through the leak could be increased by applying a higher pressure differential across the leak. This higher differential pressure could be achieved by a higher level of internal gas pressurization of the vessel or component under test, by heating the gas within a sealed component to increase its pressure or by reduction of the pressure acting through the test liquid on the low pressure side of the pressure boundary. These techniques increase the sensitivity of the test procedure to which the components are subjected. They may also result in more easily observed bubble indications that improve the reliability and speed of bubble testing.

Sensitivities Attainable with Liquid Film Bubble Testing

The actual sensitivity attained in bubble testing depends on the control and selection of leak test conditions that influence factors affecting sensitivity. Sensitivity also depends on the selection of the test technique. The liquid application technique (solution film technique), in which a thin film of liquid is applied and bubbles form in air (like soap bubbles floating on water), is typically used only for leak detection and location. A leak is a physical hole; the gas passing through it is leakage. Service requirements or specifications for testing may require that any detectable leakage be taken as cause for rejection or for repair of leaks. In this case, it is not necessary to measure actual leakage rates to determine the disposition of the test items. The sensitivity of the liquid application technique of bubble testing is adequate for locating leaks with leakage

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rates in excess of 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}). The solution film procedure is widely used on large pressurized systems that cannot be immersed in detection liquid. The technique is ideal for quick detection of large to moderate size leaks (10^{-2} to 10^{-4} Pa·m^3·s^{-1} or 10^{-1} to 10^{-3} std cm^3·s^{-1}) at very low costs.

Sensitivities Attainable with Immersion Bubble Testing

In bubble testing by the immersion technique, test sensitivity depends on operating conditions and selection of both the tracer gas and the test liquids. Other factors can also change the test sensitivity actually attained. With certain combinations of tracer gases and detection liquids, sensitivities of 10^{-8} Pa·m^3·s^{-1} (10^{-7} std cm^3·s^{-1}) have been attained with calibrated leaks operating under laboratory conditions. Under excellent industrial immersion bubble testing conditions, maximum sensitivity of bubble testing is in the range of 10^{-5} to 10^{-6} Pa·m^3·s^{-1} (10^{-4} to 10^{-5} std cm^3·s^{-1}).

Operator Training and Motivation to Maintain Bubble Test Sensitivity

The sensitivity of bubble testing is hard to define because it also depends on the observation and alertness of the leak test operator. Practically, under excellent industrial test conditions, there is no question that leakage of 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}) can be observed by the immersion bubble testing procedure. However, it is a different matter when operators do not know that a leak exists and have to examine a long weld seam for a possible bubble. Conceivably, they might not wait long enough for the bubbles to form or they might fail to look carefully after sufficient time at every portion of every area where a potential leak might exist. Thus, optimum bubble observation conditions and continuing training and motivation of bubble test operators to achieve and maintain their best observational capabilities are essential if the reliability and sensitivity of bubble testing are to be ensured.

Effects of Test Pressures on Bubble Formation

Because a minimum pressure is required to form a bubble in a liquid, bubble testing sensitivity depends on the pressure differential acting across a leak. Bubble testing sensitivity increases with an increase of pressure across a leak. Sometimes, it is possible for the operator to estimate that a certain rate of leakage is observed because a bubble of a particular volume is being observed. However, this type of leakage rate estimation can be inaccurate on very small leaks because of the finite solubility of the tracer gas in the bubble test liquid. It is theoretically possible for a small leak to exist where the tracer gas from a capillary leak dissolves in the test liquid so fast that no leakage bubble indication is visible. Special techniques that serve to increase the pressure differential across the leaks can be used to increase bubble testing sensitivity. Sensitivity improvements resulting from such special techniques are described in the discussions of each individual technique in this chapter.

Preparation of Test Objects for Bubble Testing

Before bubble testing, test objects must be prepared to ensure that surface contamination, liquid blockage of leaks, protective coatings, sources of gas emission, uncovered openings and other conditions that could interfere with effective leak testing have been properly corrected or controlled. In addition, safety precautions are required when pressurizing vessels, components and systems for leak testing. Otherwise, excessive pressure may destroy the test object or injure the test operator. Typical requirements for precision leak tests in aerospace and general industry specifications may serve as illustrative examples of factors to be considered in various applications.

Precleaning of Test Object Surfaces before Bubble Testing

Before leak testing by bubble techniques, the test object surface areas to be tested must be free of oil, dirt, grease, paint and other contaminants that might mask a leak. Surface contamination of the test item in the form of grease, loose paint, rust, weld slag or chemicals may become a source of bubbles, giving false indications of a leak. Temporary plugging of leaks might also occur because of common manufacturing techniques. Leak testing must be done before painting or plating of test objects or else such coverings must be removed to expose leak openings and ensure absence of leak blockage. Tests must not be performed on grease filled components. Any test object condition that could lead to contamination of the bubble test detection fluid or that could cause foaming of the inspection liquid should not be permitted. Foaming creates

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spurious surface bubbles on the test specimen.

Whenever feasible, bubble tests should be performed before any other tests where gas is the pressurizing medium. Any possible clogging of leaks by prior exposure to liquids (as by prior hydrostatic pressure tests, surface cleaning with liquid agents or storage in damp environments subject to condensation of water vapor) must be avoided. When test surfaces have been previously exposed to liquids such as hydrostatic tested castings, this surface condition must be corrected by careful drying (with heat or vacuum or both) to remove liquid that may be clogging the leaks.

In addition, castings to be coated after hydrostatic testing with synthetic rubber or rubbery coatings that require vulcanizing after application with heat must be dried carefully to remove any moisture that may have penetrated into porosity or other casting defects. Failure to remove from these openings the water that did not leak on hydrostatic testing will cause the coating to blister and fail when moisture in cavities tries to escape during the vulcanizing of the coating.

Sealing of Openings in Vessels and Test Objects before Leak Testing

Leak tests must often be performed on vessels, pipe sections, valves and other components or system elements that have intentional openings such as at flanges, threaded holes, instrument connections and points of attachment to other elements of fluid containment systems. All such openings must be sealed using plugs, covers, sealing wax, pipe caps or other components or materials that can be readily and completely removed following completion of leak testing. Except when using back pressurizing techniques, a gas inlet should be provided by attaching a valve to one of the test covers on all items pressurized or subjected to vacuum during leak testing. For the back pressuring techniques, a calibrated pressure gage and valve should be provided on the pressurizing chamber.

Check of Test Object and Equipment before Applying Pressure or Vacuum

The test equipment and sealed test objects should be carefully examined before applying pressure or vacuum to ensure they are properly sealed. It is also vital to establish that all appurtenances that should not be subject to pressure or vacuum have been disconnected or isolated from the test system by valves or other suitable means. Test parts to be immersed must be examined visually for possible leakage paths that should be marked, sealed or repaired (where possible) before immersion in leak testing fluid for bubble tests.

Pressurization of Test Specimens for Bubble Tests

It is necessary to create a pressure differential between the inside of a component and its surroundings if a bubble test is to be used. One technique used in bubble testing is to connect a high pressure gas source to the component through pressure reducing valves with pressure indicating gages. Gases suitable for pressurizing test objects include clean air, nitrogen, helium, argon, refrigerant gases, ammonia and other tracer gases (usually specified for specific leak testing applications).

Compressed air can be used for pressurizing and as a tracer gas, provided it is obtained from a gas cylinder or provided by oilfree compressors and oil filters. Compressed air from shop air lines or local air pumps is not recommended because such air lines and pumps often introduce oil, water and rust into the air. Dirt, oil or water carried in the compressed air supply could act to block small leaks temporarily and may contaminate the item being tested.

Gas pressure should be applied to the unit under test before liquid application or immersion so that the detection liquid will not enter small leaks. Once a leak has been clogged, a much higher pressure differential is required to reopen and detect that leak.

Technique for First Application of Pressure for Proof Testing or Leak Testing in Industry

Typical pressurizing specifications in industry require that the test pressure be gradually increased in the test part or system to about half of the final test pressure and then increased to the final test pressure in steps equal to 0.1 of the final (maximum) test pressure. Unless otherwise specified, the minimum pressure difference between the gas pressure within the test object and the pressure at the greatest depth of the test part in an immersion test liquid should be 100 kPa (15 $lb_f \cdot in.^{-2}$). The maximum test pressure should not exceed the maximum allowable working pressure for the component or system under test, unless special safety precautions are taken to protect personnel and to avoid rupture of the test part. Also, a stress analysis should

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be performed to demonstrate that the part will not be damaged by test pressures.

Unless otherwise specified, the soak time should be at least 3 $s \cdot m^{-3}$ (0.1 $s \cdot ft^{-3}$) of internal volume of the test part or system or 15 min, whichever is longer. Soak time is the time allowed for dispersion of the tracer gas (test gas) throughout the volume of the test part or system, before performing the visual inspection for bubbles.

Special Technique for Application of Test Pressure in Industry

An interesting technique of applying a large pressure differential for leak tests of small cryogenically compatible parts is to first immerse the parts in a liquefied gas such as liquid nitrogen. The liquid nitrogen enters the test part through any existing leaks. Then the part is immersed in a room temperature liquid such as alcohol. On warming test parts in the alcohol, liquid nitrogen gasifies and builds up a pressure. Gaseous nitrogen escaping from the leak is detected by the rising stream of bubbles when the part is immersed in the room temperature liquid.

Controlling Temperature of Test Object, Pressurizing Gas and Test Liquid

For components constructed of steels whose resistance to brittle fracture at low temperature has not been enhanced, controls to maintain test temperatures above 0 ˚C (32 ˚F) are recommended. Maintaining the test object temperature well above the nonductility temperature of the steel reduces the risk of brittle fracture during the bubble emission test. The test pressure should not be applied until the temperatures of the test part and the pressurizing gas are within ±15 percent of the same temperature in celsius degrees (10 percent in fahrenheit degrees). The temperature of the test part, components, pressurizing gas and test liquid must not be at a level that would be injurious to test personnel or to the test equipment, the test object or its components.

Conditions for Visual Inspection of Bubbles

When performing the visual inspection to detect bubble leaks in systems at safe, low pressures, access to the test object area being viewed should permit the placing of the observer's eyes within 0.60 m (2 ft) of the surface to be examined. Where test pressures are higher than is safe for test personnel, electrical or optical apparatus may be used to transmit data to the observer. The angle of view should be no less than 30 degrees with the plane of the surface to be examined. Mirrors can be used to improve the angle of vision and aids such as magnifying lenses may be used to assist examination. Natural or artificial lighting can be used to illuminate the area being examined. The light intensity in the area being examined should be a minimum of 1 klx (100 ftc).

Whenever possible, the bubble leak inspection should generally be performed on test object surfaces in the horizontal position. Where possible, surfaces to be inspected should be up. In immersion bubble tests, the surface to be tested should lie at least 25 mm (1 in.) below the surface of the test liquid at all points. In liquid film tests, the test object surface area of interest should be (where possible) at an angle that allows the inspection film liquid to lie on the surface without dripping off. Excess liquid may be permitted to run off the surface as long as sufficient liquid remains to provide a continuous wet film on the surface being tested. Surfaces of large pressure vessels and components must be tested at all angles because they are not moved during tests.

Speed of Visual Inspection during Bubble Tests

The speed of visual inspection of the test surface during bubble tests should not exceed a maximum rate of 12 $mm \cdot s^{-1}$ (30 $in. \cdot min^{-1}$) for fusion weldments. Small cylindrically shaped parts or semiflat parts that are presented in layers (one deep) for inspection shall have a minimum observation time of 35 $min \cdot m^{-2}$ (3 $min \cdot ft^{-2}$) per observable side. For all other test parts, the parts should be examined individually at a maximum rate of 0.1 m^2 (1 ft^2) per minute per part. However, other speeds of visual inspection may be required during bubble testing of large vessels outdoors.

Aids to Vision Used in Bubble Testing

If the leak is small, the bubbles may be difficult to see unless the observer's eyes are adapted to the specific lighting levels. A reading glass may be found to be of great assistance. A 75 mm (3.0 in.) diameter glass provides a magnification of 2× to 3× when held at a distance of 100 to 120 mm (4.0 to 5.0 in.) from the test object. Because there exists a minimum size of bubble (for a specific inspection fluid and test condition), the reading glass does not introduce any new eyestrain by revealing smaller bubbles. Good lighting is essential. Side lighting of bubbles and

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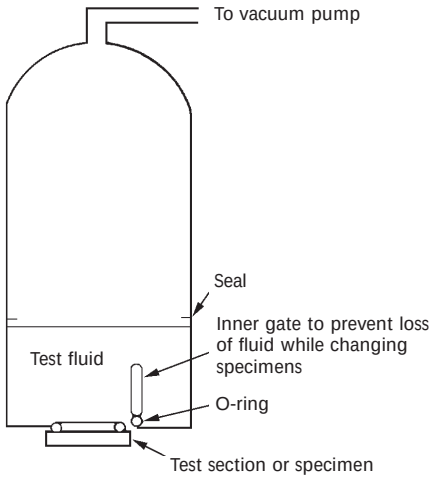
use of a dark background are often helpful. In some cases, a small stream of bubbles may be detected more easily from above than when observed from the side. In other cases, a large hole or crack with high differential pressure may blow test liquid clear of the test surface with no bubbles being formed. Test inspectors should be alert to detect this condition.

Vacuum Technique for Bubble Testing by Immersion

A minimum pressure differential of 100 kPa (1 atm) is typically required for bubble testing of sealed components. Parts that have atmospheric pressure inside can meet this requirement by placing the component within an enclosure and then evacuating the enclosure. This technique can give pressure differentials up to 100 kPa (1 atm). In the vacuum technique, small specimens can be immersed in the test liquid; the test liquid container is then placed within the vacuum chamber (see Fig. 1).[1] The pressure within the vacuum chamber is then reduced to a point that does not allow the test liquid to boil but creates nearly 100 kPa (1 atm) of pressure differential. The amount of vacuum used will depend on the choice of test liquid. It should be the maximum vacuum attainable without making the test liquid boil. Viewing ports in the vacuum chamber (or bell jar) permit observation for a stream of bubbles originating from a single point or of two or more bubbles that grow and then are released from a single point, as the pressure in the vacuum chamber is reduced. This technique is also applicable to unsealed components or specimen sections by use of the vacuum box apparatus of Fig. 2.[1]

FIGURE 1. Vacuum chamber technique for providing pressure differential across leaks during bubble tests.



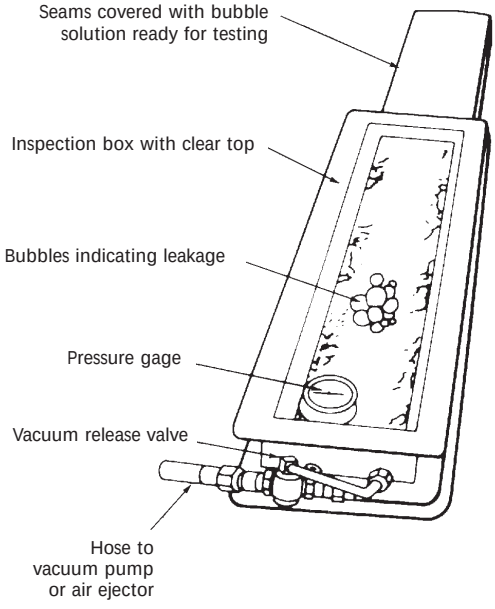
Solution Film Technique for Bubble Testing without Immersion

A relatively simple procedure for bubble testing with films of test liquid consists of three basic steps.

1. Pressurize the system under test.
2. Apply a test liquid in the form of a thin, continuous, wet film to the test object surface.
3. Observe a bubble formation that indicates a leak. A bubblefree solution should be applied gently to preclude bubble formation during liquid film application. The detection solution should be flowed or applied by a fine orifice sprayer, but not brushed, onto the test surface. The sensitivity of the film application bubble test technique is highly dependent on the time and care taken by the operator in applying the test liquid and observing the bubble formation.

Numerous commercial leak testing solutions can be used as solution film bubble testing liquids. One film solution for leak indication consists of 1 part liquid

FIGURE 2. Vacuum box technique for providing pressure difference across leaks in local areas of large test objects.



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soap or detergent, 1 part glycerine and 4.5 parts of water. This solution should be prepared no more than 24 h before the test. Its bubble formation properties should be checked with a sample leak periodically during the period of leak testing. Homemade test solutions left over at the end of each test period should be discarded. Commercial leak testing solutions kept in closed containers or pressure spray cans may be used intermittently and stored for later use, as recommended by their manufacturers.

Precautions in Applying Solution Film Leak Test Liquids

Two cautions apply to solution film techniques of bubble testing. When testing flanges, threads or any joint that has a large exposure area, it is absolutely necessary that the film solution bridge the entire joint. Gas will invariably slip out through the smallest pinhole that is not covered. The second caution applies to the choice of a film solution. For high leak testing sensitivity, it is necessary that the solution film not break away from the joint. Leak indicating bubbles formed should not break due to air drying or weak surface tension of the solution film. Dilution of original film test solutions with added water must be avoided.

Applications of Solution Film Bubble Testing Techniques

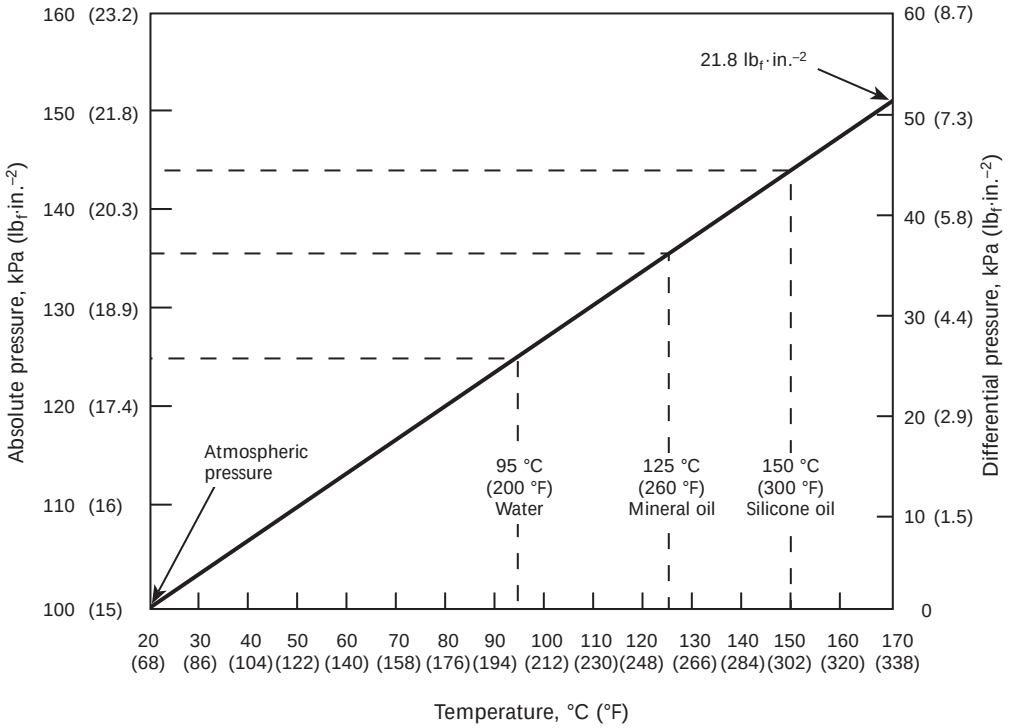
The solution film bubble testing technique can be applied to any test specimen on which a pressure differential can be created across the area to be examined. An example of this technique is the application of leak test solutions to pressurized gas line joints. It is most useful on piping systems, pressure vessels, tanks, spheres, compressors, pumps or other large apparatus on which immersion techniques of bubble testing are impractical.

The system or section being leak tested can be pressurized for film solution bubble tests in various ways. Considerable ingenuity may be required in making up special clamps and fittings for sealing the test component and attaching the pressurizing gas hose. Rubber gaskets or sheets must have an entry hole for the test gas and connection to a pressure gage when used for pressurizing for leak tests.

Bubble Testing of Small Components in Heated Immersion Bath

With small sealed components such as semiconductor and electronic devices in

FIGURE 3. Variation of gas pressure within a component, sealed at atmospheric pressure of 100 kPa (15 $lb_f \cdot in.^{-2}$), as a function of temperature.



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hermetically sealed housings, one widely used technique for creating the pressure differential necessary for performing a bubble test is by preheating the immersion bath of detection liquid and submerging the components to be tested in this bath. As the temperature within the components rises, the gas or air inside the sealed enclosure will expand and the internal pressure will rise. The pressure differential created by placing sealed components in a heated bath will be in the range of 10 to 34 kPa as shown in Fig. 3. Charles law as shown in Eq. 1 relates pressure to temperature:

$$\text{(1)} \quad \frac{P_1}{T_1} = \frac{P_{20}}{T_{20}} = \frac{100}{20 + 273} = \frac{P_2}{T_2}$$

where P_{20} is pressure (in this case, 100 kPa or 1 atm) at temperature T_{20} (293 K or 20 °C); P_1 is initial pressure, which is equivalent to P_{20}; T_1 is initial temperature (in same unit as for T_{20}); and P_2 is pressure at higher temperature T_2.

Once the immersion bath reaches the desired temperature, no further adjustments are necessary, except for minor changes required to maintain a constant bath temperature. If the immersion bath of detection liquid is large enough, specimens to be tested can be mounted on a rack and several components can be tested at the same time. This technique is conducive to the testing of mass produced items such as resistors, semiconductors, integrated circuits and hermetically sealed components.

Comparison of Heated Bath and Vacuum Bubble Testing of Sealed Components

Most electronic component manufacturers use the vacuum technique or the heated bath technique when conducting their bubble tests. The evacuated chamber test is more sensitive than the heated immersion bath type of bubble test. A pressure differential of almost atmospheric pressure (100 kPa, 15 $lb_f \cdot in.^{-2}$ or 760 torr) exists across the pressure boundary in vacuum leak tests of objects with internal room temperature gas pressure of 100 kPa (1 atm). On the other hand, the pressure differential may be about 40 kPa (6 $lb_f \cdot in.^{-2}$) for sealed components in the heated oil bubble test. The heated bath type of test is simpler to perform than the vacuum test.

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PART 2. Theory of Bubble Testing by Liquid Immersion Technique

Principle of Immersion Technique of Bubble Testing

The immersion technique of bubble testing for leaks is applicable for specimens whose physical size allows their immersion into a container of liquid. The test objects could be hermetically sealed or sealed off during the test. This technique involves pressurizing the system or component under test with a gas, before and during the period the component is immersed in an inspection liquid. The source of the leak is indicated by the bubbles of gas formed when the gas under pressure emerges from a leak into the surrounding liquid. The test object and leak test apparatus should be designed to avoid concealed or trapped leaks.

The appearance of a bubble gives an immediate indication of the opening through which the gas passes. The bubble or stream of bubbles, issuing from a leak opening, locates the exit point of leakage. The immersion procedure of bubble testing serves to locate the leak as well as to indicate that a leak exists. The major attributes of bubble testing are its simplicity and its ability to locate leaks very accurately. When large vessels must be tested, immersion may be impossible or impractical. However, channels built around suspected leak areas can be used to contain the immersion test fluid and allow bubbles into subsurface regions of the test fluid.

Conditions Influencing Formation of Submerged Bubbles during Leak Tests

The process of forming bubbles that result from gas flow through a given leak into an immersion liquid depends not only on the pressure conditions but also on the physical properties of test liquids in which bubbles form. It also depends on the properties of the tracer gas that flows through the leak to form the bubble indication. Thus, by a suitable combination of the liquid and the gas selected for testing, the sizes of the bubbles and the rate of formation of bubbles can be modified. For purposes of leak detection and location, it is desirable that the bubbles be clearly visible to the human eye. The sensitivity of the immersion bubble test technique is determined by the operator's ability to observe bubbles formed at the outlet end of small holes. Because of surface tension, these passages often may set up a high resistance to the passages of tracer gas. High test liquid surface tension may restrict the formation of bubble indications. The more readily the bubbles are evolved, the more easily they are observed. This necessity for visibility of test indications is an important consideration when choosing the particular combination of tracer gas and test liquid to be used in immersion bubble testing for leaks. It is possible to change the sensitivity of the bubble test by changing either the tracer gas or immersion liquid. The rate of leakage of the test gas can be increased by selecting a tracer gas with better flow characteristics, without requiring any change in the gas conductance of the leak.

Factors Influencing Diameter and Rate of Formation of Submerged Bubbles

When the test liquid does not wet the solid surface around the orifice of a leak, the bubble rim tends to spread away from the leak orifice. This results in formation of larger bubbles. Larger bubbles are also formed in the presence of traces of grease or other conditions that tend to inhibit surface wetting. For a given gas flow rate, the production of larger bubbles reduces the frequency of bubble formation. With a specific rate of gas leakage, the frequency of bubble formation (number of bubbles formed per unit time) varies inversely with the bubble volume. Thus, the frequency varies inversely with the cube of the bubble radius. As a result, for a given leak, the bubble frequency in organic liquids can be as much as 100 times higher than the frequency of bubble formation in water. Both ethyl and methyl alcohol tend to wet most solids more readily than water and the bubbles will be smaller. When water is used as the immersion liquid for bubble tests, it must be treated to reduce the surface tension. Detergents and wetting agents can lower the surface tension of water. Reducing the

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surface tension reduces the bubble size and the tendency of bubbles to cling to the test object surface. The characteristics of other immersion liquids for bubble tests are described later in this chapter.

Mathematical Equation for Bubble Formation in Liquids

Bubbles are emitted from a leak immersed in a liquid when the pressure of the escaping gas exceeds the sum of the hydrostatic head and the maximum surface tension restraint. Equation 2 applies to the pressure balance in the case of a cylindrical leak hole:

$$\textbf{(2)} \quad P = P_a + \rho g h + \frac{2\sigma}{r}$$

where P is the pressure of gas within the leak capillary and forming bubble, in kilopascal (or dyne per centimeter); P_a is pressure above the test liquid (atmosphere or vacuum), kilopascal (or dyne per centimeter); ρ is density of immersion liquid, kilogram (or gram) per cubic meter; g is acceleration of gravity, meter (or centimeter) per second per second; h is depth of liquid immersion at leak location, meter (or centimeter); r is radius of (cylindrical) capillary leak hole, meter (or centimeter); σ is surface tension of liquid, newton per meter (or dyne per centimeter). Equation 2 is used with all variables expressed in SI units only (or instead in the centimeter-gram-second system).

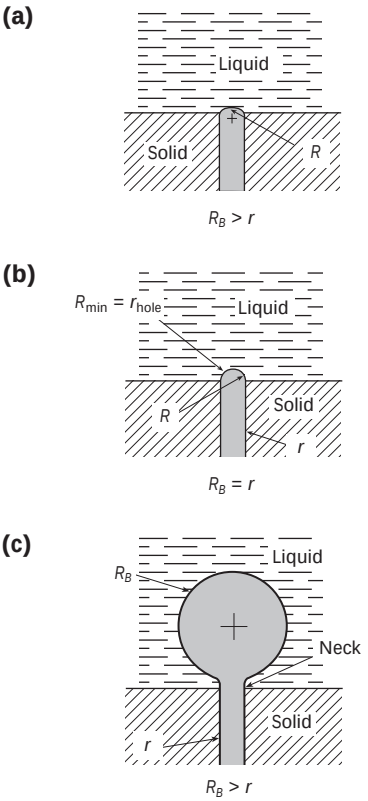
Mechanisms of Bubble Formation in Immersion Test Liquid

As tracer gas exits the leak, each bubble forms and expands, as sketched in Fig. 4. Ultimately, the bubble is attached to the rim of the leak by a neck (Fig. 4c). Now, assume that the bubble formed at the end of a tube is shaped like a part of a sphere. Then as the bubble is being generated, its radius R first decreases from that sketched in Fig. 4a. The minimum bubble radius R_{min} is reached as the bubble shape approximates a half sphere whose radius is identical to the capillary tube radius r as illustrated by the sketch of Fig. 4b. This variation implies that the term $2\sigma/r$ in Eq. 2 reaches a maximum value when the condition of Fig. 4b is reached. This corresponds to a maximum value of excess pressure. Thereafter, the bubble radius R_B increases to form the expanding spherical bubble of volume $V = (4\pi/3)R_B^3$ of Fig. 4c. When the buoyant force $V_{\rho g}$ of the bubble exceeds the surface tension restraint force ($2\pi r\sigma$) at the neck of the bubble, the bubble detaches and rises to the liquid surface. This condition for bubble detachment from the site of the cylindrical capillary leak is suggested as a theoretical approximation in Eq. 3:

$$\textbf{(3)} \quad F = \frac{4\pi}{3} R^3 \rho g - 2\pi r\sigma = 0$$

In Eq. 3, R is bubble radius at the detachment stage and r is the radius of the cylindrical hole and neck from which the bubble detaches, when both r and R are given in identical units. Equations 2 and 3 present an elementary picture of bubble formation and growth. In more rigorous equations, the liquid viscosity affects the bubble size; however, this effect is considered to be negligible for most leaks. With an increase in viscosity, there will be only a small increase in bubble size.

FIGURE 4. Bubble formation at a leak site in immersion detection liquid: (a) bubble with radius less than capillary radius; (b) hemispherical bubble; (c) spherical bubble.



Legend

R	=	radius
R_B	=	bubble radius
R_{min}	=	hemispherical minimum bubble radius
r	=	capillary radius
r_{hole}	=	hole radius

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Critical Pressure Required for Formation of Large Stable Bubbles

The external atmospheric and hydrostatic pressure involved in immersion bubble testing can often be regarded as constant if the hydrostatic head is low. Therefore, there is a maximum pressure inside the incipient bubble that must be reached if the bubble is to expand beyond its hemispherical stage where the minimum bubble radius R_{min} is equated to the leak hole radius r (see Fig. 4b).

This condition imposes a limit on the applications of bubble testing, so its effect should be considered briefly. Suppose, for example, that the end of the capillary is submerged in water at atmospheric pressure of 100 kPa (1×10^6 dyne·cm^{-2}). Suppose also that the other end of the capillary is connected to an internal gas pressure of twice atmospheric pressure (200 kPa). If the surface tension of the water were 0.073 N·m^{-1} (73 dyne·cm^{-2}) and the pressure differential were atmospheric, the capillary leak radius r would be determined by Eq. 4:

$$\textbf{(4)} \qquad r = \frac{2\sigma}{\Delta P} = 2\,\frac{0.073}{10^5} = 1.46\ \mu\text{m} = \left(5.75 \times 10^{-5}\ \text{in.}\right)$$

This radius r represents the smallest capillary radius detectable by bubble testing with a pressure differential of 100 kPa (1 atm) and with unmodified water as the immersion bubble testing fluid.

Advantages of Low Surface Tension Immersion Liquids in Bubble Testing

If bubble tests were made with an immersion liquid of lower surface tension such as methyl or ethyl alcohol, then the same excess pressure of 100 kPa (1 atm) used in the relation $r = 2s/WP$ would allow the formation of a bubble at the end of a capillary with the much reduced radius of $2(0.023/100\ 000) = 0.46 \times 10^{-6}$ m = 0.46 µm. This radius is less than one third of the least radius of a capillary leak detectable by bubble testing when using water as the immersion test liquid. Low surface tension liquids might theoretically reveal leakage rates in the range of 0.02 to 0.01 of the lowest leakage rates detectable when the bubbles are formed in water. Thus, theory indicates that the sensitivity of bubble immersion leak tests could be increased appreciably by the bubble testing detection liquids with low surface tension.

Additional Factors Influencing Bubble Size during Immersion Leak Testing

In bubble immersion leak testing, it is desirable to use a bubble forming testing liquid with low surface tension and low viscosity. The pressure differential acting across the leak should be made higher for detection of capillary leaks of small diameter. In addition, the use of a low viscosity, low mass tracer gas will increase the gas flow rate through the capillary leak.

Another factor that affects the sensitivity of bubble testing is the size of the bubbles involved. The size of bubbles increases with an increase in the surface tension of the immersion testing liquid. To generate large numbers of small bubbles, it is desirable to use liquids with small surface tension. Bubble size can also be affected by vibration. If the test object is subjected to increased levels of vibration, the bubbles break off before they would have been released with a stationary test object. This could be useful because vibration increases the bubble emission rate for a given gaseous leakage rate.

If the pressure acting on the surface of the immersion liquid is reduced below atmospheric pressure until bubbles just emerge from the end of the leakage path, limitations are imposed by the tendency of the liquid to degas and boil under conditions of reduced pressure.

Immersion liquid with a high boiling point (low vapor pressure) allows reasonably low pressures to be used within the detection liquid without boiling. To enable detection of smaller leaks, it is desirable to use immersion liquids having low values of surface tension. However, such low tension liquids also have correspondingly lower boiling points. These liquids may boil spontaneously before the pressure over the liquid could be reduced sufficiently (as by pulling a vacuum over the liquid) to significantly improve gas flow through the leaks or to enlarge the bubbles to increase their visibility. Therefore, the choice of immersion liquid for bubble tests should be made very carefully.

Several different techniques can be used to establish the pressure differential across pressure boundaries that may contain leaks. Whenever convenient, the system, vessel or component under test should be pressurized with gas.

However, if gas pressurization proves impractical, an alternative procedure is to draw a vacuum over the surface of the test liquid. A third alternative is to heat the immersion liquid, thus creating a pressure

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differential by thermal expansion of the air or gas within the test object. A fourth technique is to inject an evaporatable liquid through a leak in an item and permit it to gasify, thus pressurizing the interior. This might be achieved by using a liquid refrigerant and permitting it to gasify at normal temperatures.

Advantages and Limitations of Immersion Bubble Testing in Water Baths

Water, mineral oil or a silicone oil may be used as an immersion test liquid in bubble testing. If water is used, it must be treated to reduce the surface tension. This reduces the bubble size and reduces the tendency of bubbles to cling to the surface of test objects. Bubbles in a water bath cling to the surface of the test component and enlarge before breaking loose and rising to the liquid surface. This means that small leaks forming bubbles in water would require a long response time to produce a bubble that would be visible on the surface. Under these test conditions, a component might erroneously be passed as acceptably sealed because insufficient time was allowed for bubbles to form a conclusive test indication.

Example of Commercial Additive for Water Baths Used in Immersion Tests

A typical additive for water immersion bubble testing will produce bubble indications of small leaks that are impossible to obtain with normal water. These types of additive do not cause foaming, do not support bacteria growth and are inhibited to prevent corrosion of test objects or immersion tanks. One type includes a chromate inhibitor to prevent rust formation on iron or carbon steel. Other inhibitors are used for protection of test parts made of aluminum, titanium or stainless steels. Although the water additives are recommended for use with deionized water, chelating agents can be used to make them compatible with hard water. The additive is added to the deionized water in the immersion tank in typical ratios varying from 1 to 25 percent of additive agent in the water. The additive agents go into solution immediately. The user should use rubber gloves when handling the chromate inhibited additive product or test objects being immersed in solutions with this additive, to avoid possible skin irritation. Still other additive formulations are recommended when the parts to be tested are made of plastic or fiberglass composites.

The minimum leakage rate detectable in immersion bubble tests can be varied by varying the pressure differential applied across the pressure boundaries of the test objects and also by varying the concentration of the additive agent in the water filled immersion tank. Table 1 indicates the typical minimum detectable leakage rates for various concentrations of the additive in immersion tank water, when nitrogen at 25 ˚C (77 °F) is used as the pressurizing gas.

TABLE 1. Typical minimum detectable leakage rates at 25 °C (77 °F) with nitrogen gas at 120 kPa (18 $lb_f \cdot in.^{-2}$).

Additive in Immersion Water (percent)	Rate	
	$Pa \cdot m^3 \cdot s^{-1}$	(std $cm^3 \cdot s^{-1}$)
0	1×10^{-4}	1×10^{-3}
1	1×10^{-5}	1×10^{-4}
5	1×10^{-6}	1×10^{-5}
25	1×10^{-7}	1×10^{-6}

Advantages and Limitations of Immersion Bubble Testing in Oil Baths

A steady stream of extremely fine bubbles appears when objects with leaks are submerged in an oil bath. This provides highly visible bubble indications with short response times. However, a disadvantage of immersion bubble tests using an oil bath is the fact that test components must be degreased after being tested to remove the oil that adheres to the surfaces. A silicone oil bath is particularly expensive to use because the oil clings to the components after immersion and cannot be completely recovered. A mineral oil bath is the most satisfactory immersion test fluid for bubble testing with the immersion liquid under vacuum. Although the degreasing of components after leak testing adds expense to oil bath leak testing procedures, it is often worthwhile because of the improved leak test sensitivity attainable with an oil bath immersion technique of bubble testing.

Advantages and Limitations of Immersion Bubble Tests in Alcohols

Methyl, ethyl or isopropyl alcohol can be used as an immersion detection liquid for bubble testing. One advantage of using alcohol as an immersion bath that is not found with any of the other immersion test liquids is alcohol's cleaning properties. This eliminates the degreasing process. The alcohol also cleans foreign matter introduced by production processes from surface of test objects. After the test pieces are removed from the

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alcohol bath, no alcohol remains on them because alcohol evaporates rapidly. On the other hand, this rapid evaporation could lead to rapid loss of alcohol from uncovered alcohol immersion baths and to fumes and potential fire or explosion hazards.

Alcohol cannot be used as the immersion test liquid in the heated bath technique. In addition, the rapid evaporation of alcohol can be a detriment in the evacuated chamber technique of immersion bubble testing. The amount of fluid in the bath tends to decrease rather rapidly so that repeated replacement is necessary. A bath that can accommodate a tray with numerous test components on it would have a large area that is exposed to the atmosphere and the alcohol evaporation rate would tend to be excessive.

Specific hazards associated with alcohol include the fact that methyl alcohol could be extremely harmful to operating personnel if it were to get into the body or eyes. Severe poisoning or damage to vision can result to the test operator or anyone in the test vicinity unless adequate ventilation is provided to remove harmful vapors. The alcohol fluids are flammable and so present a fire hazard.

Immersion Inspection Liquids for Bubble Testing

Typical bubble tests liquids used in immersion leak tests in industry include the following.

1. Water treated with a liquid wetting agent to reduce surface tension and promote the frequency of bubble emissions; certain solid wetting agents are also very effective in small weight percentages, with water baths.
2. Ethylene glycol (technical grade) undiluted.
3. Mineral oil. Degreasing of test specimens following immersion leak tests may be necessary. If mineral oil having a kinematic viscosity of 3.77×10^{-5} to 4.11×10^{-5} $m^2 \cdot s^{-1}$ (37.7 to 41.1 centistoke) at 25 °C (77 °F) is used as the test liquid, it will meet the material requirements of MIL-STD-202F (April 1980).[2] Mineral oil is the most suitable test liquid for the vacuum technique of immersion bubble testing.
4. Fluorocarbons of glycerine. Fluorocarbons are not recommended for stainless steel or materials for nuclear applications. Glycerine is a relatively poor detection liquid with low sensitivity to bubble emissions (see Fig. 5).
5. Silicone oil having kinematic viscosity of 2×10^{-5} $m^2 \cdot s^{-1}$ (20 centistoke) at 25 °C (77 °F). This liquid will meet the requirements of MIL-STD-202F (April 1980)[2] for electronic components. However, silicone oil should not be used for leak testing of parts to be subsequently painted.

Selection of Tracer Gases and Test Liquids for Immersion Bubble Tests

In general, the sensitivity of bubble testing can be increased in three basic ways: (1) by increasing the pressure differential across the leak, (2) by using immersion test liquids with low surface tension and (3) by using tracer gases with low viscosities.

Figure 5 through 13 present graphic data to illustrate these three techniques and provide quantitative indications of immersion bubble test sensitivity with various combinations of pressure, tracer gases and detection liquids.

Relative Sensitivities of Bubble Test Immersion Liquids with Air Leaks at 310 kPa (45 $lb_f \cdot in.^{-2}$ gage)

The relative sensitivities of immersion bubble testing with various immersion liquids when air at 310 kPa gage pressure (45 $lb_f \cdot in.^{-2}$ gage) is the pressurizing gas are indicated by the curves of Fig. 5. The minimum detectable leakage rate is shown along the logarithmic vertical scale. The time interval between bubble emissions from the leak is shown along the logarithmic horizontal scale. Decreasing the bubble interval (movement to the left in Fig. 5) corresponds to increasing the bubble frequency. The curves farthest to the left are those emission for a specific leak size. The insert in Fig. 5 lists several immersion test liquids. The sequence progresses from best sensitivity (at the top) to the worst leak sensitivity (at the bottom). Deionized water with 20 percent wetting agent appears first among the most sensitive immersion bubble test liquids. Water without addition of wetting agent appears last in this listing at the least sensitive bubble test liquid. Mineral oil, silicone oil and deionized water with two percent wetting agent appear second on the listing with high detection sensitivities. Glycols and glycerine appear very low in this listing and are relatively poor bubble testing liquids. To some degree, this relative sensitivity performance is related to surface tension, as discussed next.

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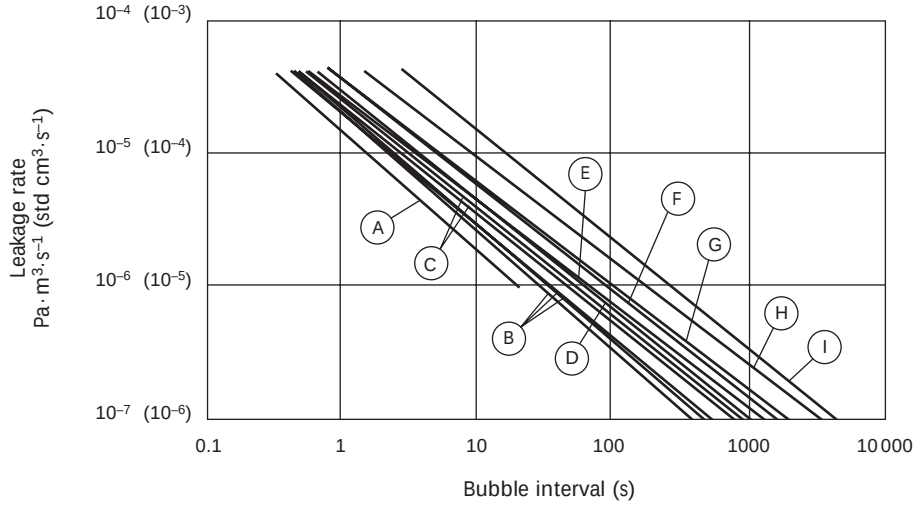
FIGURE 5. Sensitivity of immersion bubble detection liquids at 310 kPa (45 $lb_f \cdot in.^{-2}$ gage) air pressurization.



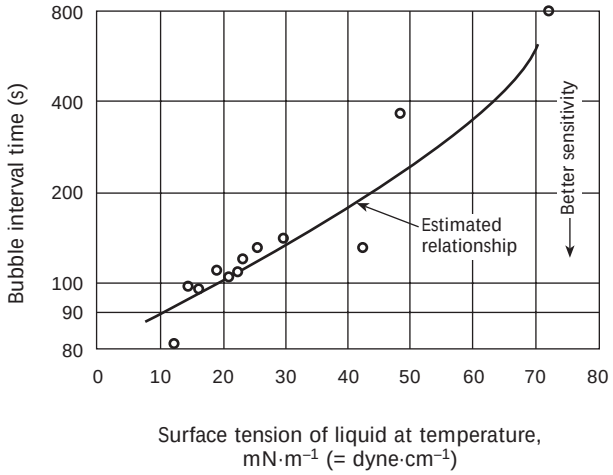
Legend

A. Deionized water with 20 percent wetting agent at 80 °C (176 °F).
B. Mineral oil number 1 at 120 °C (248 °F).
Silicone oil at 120 °C (248 °F).
Deionized water with 1 to 2 percent wetting agent at 25 °C (77 °F).
Fluorocarbon 43 at 25 °C (77 °F).
C. Silicone oil at 25 °C (77 °F).
Denatured alcohol at 25 °C (77 °F).
D. Mineral oil number 2 at 25 °C (77 °F).
E. Mineral oil number 1 at 25 °C (77 °F).
F. Glycol at 120 °C (248 °F).
G. Glycol at 25 °C (77 °F).
H. Glycerine at 25 and 120 °C (77 and 245 °F).
Deionized water at 80 °C (176 °F).
I. Deionized water at 25 °C (77 °F).

Effect of Surface Tension of Immersion Liquid for Helium Leaks at 200 kPa Gage

Figure 6 graphically shows the effect of surface tension of immersion liquids on the sensitivity (frequency of bubble emission) of leak testing with helium tracer gas at a pressure of 200 kPa (30 $lb_f \cdot in.^{-2}$ gage). The vertical logarithmic scale indicates the time interval between successive bubbles. Low values for this bubble interval correspond to high leak test sensitivities with high frequencies of bubble emissions. The horizontal linear scale of Fig. 6 corresponds to the values of surface tension of leak testing liquids, in millinewton per meter ($mN \cdot m^{-1}$) or dyne per centimeter. As liquid surface tension increases, the time between release of successive bubble increases almost exponentially. Highest leak test sensitivity results with the lowest surface tension values along the lower lest extremity of the curve of Fig. 6. The surface tensions of liquids depend on their temperatures. The curve of Fig. 6 indicates surface tension values for detection liquid at the room temperature used in the immersion bubble test.

FIGURE 6. The effect of surface tension on leak detectability in immersion bubble tests with helium tracer gas at a pressure of 200 kPa (30 $lb_f \cdot in.^{-2}$ gage).



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Effect of Helium Pressure Differential across Leak on Bubble Emission Rate

Table 2 and Figure 7 graphically show the effect of varying the pressure differential across the leak on the sensitivity of immersion bubble tests with different test liquids, for the case of helium tracer gas at pressures of 100, 200 and 300 kPa (15, 30 and 45 $lb_f \cdot in.^{-2}$ gage). Helium pressure within the component under test is shown along the linear vertical scale of this graph. The bubble interval is shown along the logarithmic horizontal scale. Highest leak test sensitivity corresponds to high frequencies of bubble emission, equivalent to short bubble intervals. Thus, sensitivity increases as the test points move to the left in the diagram of Fig. 7. Silicone oil heated to 120 ˚C (248 °F) shows the highest sensitivity of the immersion liquids listed in Fig. 7 for each pressure differential. Water with no wetting agent added shows the poorest sensitivity (longest time intervals between bubbles). Water with a wetting agent shows almost an order of magnitude greater frequency of bubbles than water with no wetting agent.

Pressurizing with helium to 200 kPa (30 $lb_f \cdot in.^{-2}$ gage) increases the bubble frequency by a factor of 3 to 5 times, in typical cases, compared to bubble rates with internal pressures of 100 kPa (15 $lb_f \cdot in.^{-2}$ gage). Pressurizing to 300 kPa (45 $lb_f \cdot in.^{-2}$ gage) increases bubble frequency by 5 to 10 times that attainable with internal component pressures of only 100 kPa (15 $lb_f \cdot in.^{-2}$ gage) of helium.

Comparison of Helium, Air and Ammonia As Bubble Test Tracer Gases

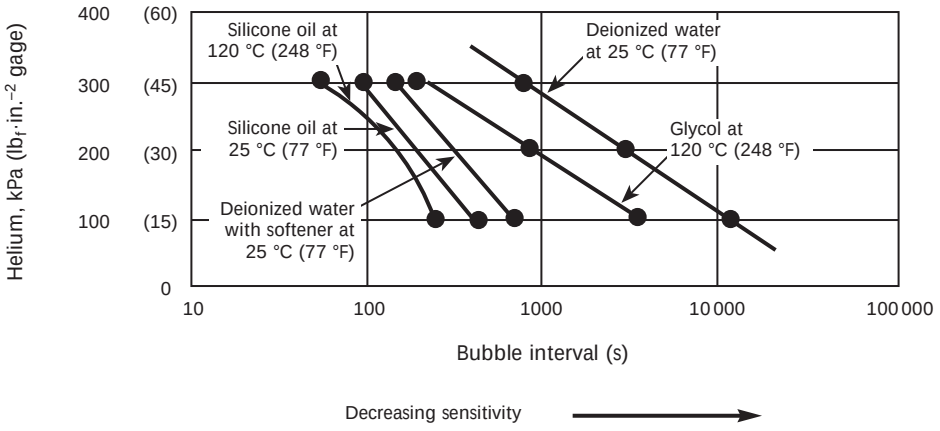
At very low leakage, helium would be expected to be the most sensitive tracer gas for leak testing because its flow is mainly molecular. With leakages between 10^{-6} and 10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} and 10^{-6} std $cm^3 \cdot s^{-1}$), helium tends to be more sensitive than air or nitrogen because a

TABLE 2. Effect of temperature and pressure on interval between bubbles.

			Gage Pressure		
			At 300 kPa (45 $lb_f \cdot in.^{-2}$)	At 200 kPa (30 $lb_f \cdot in.^{-2}$)	At 100 kPa (15 $lb_f \cdot in.^{-2}$)
Test Fluid	°C	(°F)	s	s	s
Silicone oil	120	248	50	135	245
Silicone oil	2	36	95	170	455
Deionized water[a]	25	77	138	265	675
Glycol	120	248	180	855	3470
Deionized water	25	77	800	3210	10 960

a. With water softener.

FIGURE 7. Effect of helium pressure on bubble interval in various immersion test fluids.



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combination of molecular and laminar flow exists in this range. In the laminar flow region, the vapor of ammonia with its low gas viscosity would theoretically be more sensitive than tracer gases such as helium, air or nitrogen. However, for rapid bubble evolution, ammonia does not have high sensitivity in immersion bubble tests. Also, as the leakage rate decreases, the bubble test sensitivity with ammonia tracer gas falls off markedly because of ammonia's solubility in mineral oil immersion liquid (see Fig. 8). However, ammonia is a hazardous tracer gas that should be avoided from the standpoint of safety of test personnel.

Effects of Air or Helium Pressure on Bubble Emission in Various Liquids

Figures 9 to 13 show graphs similar to that of Fig. 8 for both air and helium tracer gases with pressure differentials of 100, 200 and 300 kPa (15, 30 and 45 $lb_f \cdot in.^{-2}$) gage with various test liquids used as immersion baths for bubble tests. Figure 9 depicts deionized water at 25 ˚C (77 °F) without additives. Figure 10 shows corresponding curves for deionized water containing 1 to 2 percent wetting agent at the same test temperature. The shift of the curves to the left in Fig. 10 illustrates the increases in sensitivity provided by adding the wetting agent. Figures 11 and 12 show comparable test sensitivities attained with silicone oil immersion fluid at 25 ˚C (77 °F) and at 120 ˚C (248 °F). Elevated immersion bath temperatures generally reduce the surface tension of the liquid. This gives some improvement in bubble emission test sensitivity. However, immersion of sealed test components into

FIGURE 8. Effect of pressure and gas on bubble leak detectability using mineral oil number 2 at 25 °C (77 °F).

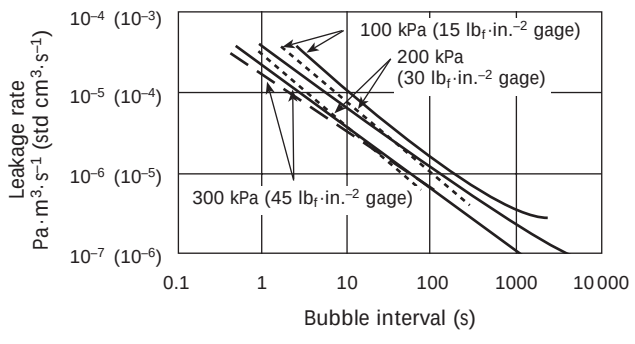


FIGURE 9. Effect of pressure and gas on detectability using deionized water at 25 °C (77 °F).

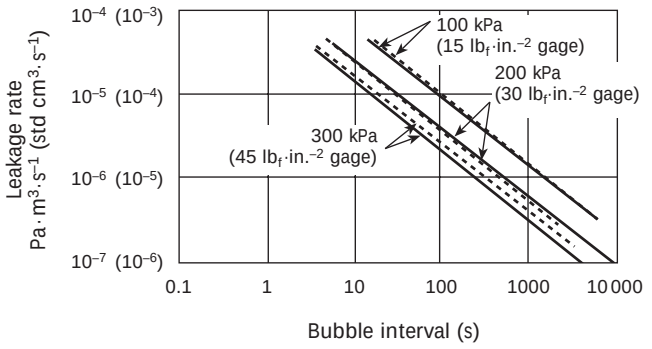


FIGURE 10. Effect of pressure and gas on bubble leak detectability using deionized water (1 to 2 percent softener) at 25 °C (77 °F).

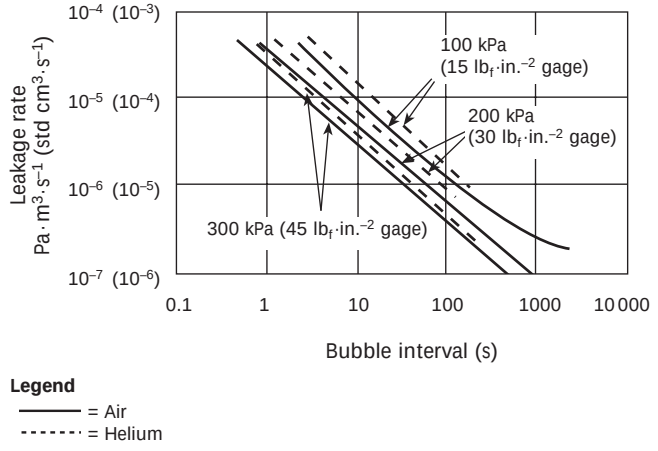
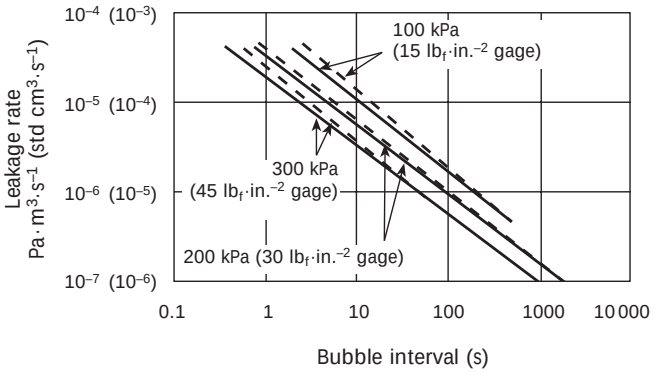


FIGURE 11. Effect of pressure and gas on detectability using silicone oil at 25 °C (77 °F).



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heated baths of detection liquid increases the internal gas pressure and thus raises the pressure differential across the enclosure. For example, by changing from a bath temperature of 25 to 125 ˚C (about 80 to 260 °F), the internal gas pressure of sealed components is increased by about 35 percent. Finally, Figs. 13a and 13b show similar curves for mineral oil and glycol baths of leak test liquid both at 25 ˚C (77 °F).

Limitations of Immersion Techniques of Bubble Testing

Many practical limitations must be considered when selecting the conditions for bubble test in immersion baths with test liquid. The major point is that the bubble test method is limited in application to detection and location of individual leaks. However, many soldered, brazed or welded and fused joints often contain long, fine cracks and numerous small leaks. These adjacent small leaks may have a high collective leakage rate, yet these individual small leaks may not generate bubbles. The bubble test is critically dependent on the operator time and care in observation of bubble indications. Operator training, adequate procedural specifications and maintenance of adequate test records can be vital.

Precautions in Bubble Testing of Sealed Electronic Components in Heated Baths

Many semiconductor manufacturers do not use heated silicone oil immersion bath during bubble testing because oil has been found inside accepted parts and because the oil is difficult to remove from the components. It has been observed that various heated oils have actually traveled back into leaks, particularly with small leaks in the range from 10^{-8} to 10^{-9} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-7} to 10^{-8} std $cm \cdot s^{-1}$) Thus, heated oils may not only act to conceal leaks, but they may also constitute a reliability risk in otherwise acceptable products.

Physical Hazards Associated with Bubble Tests in Immersion Baths

Safety problems must be carefully considered when leak tests involve handling hazardous gases such as hydrogen, ammonia, acetylene, oxygen and natural (fuel) gas. Similarly, care is required when using immersion baths of volatile, flammable or toxic liquids. Solvents such as ether, alcohol, acetone and mineral oils constitute hazards,

FIGURE 12. Effect of pressure and gas on detectability using silicone oil at 120 °C (248 °F).

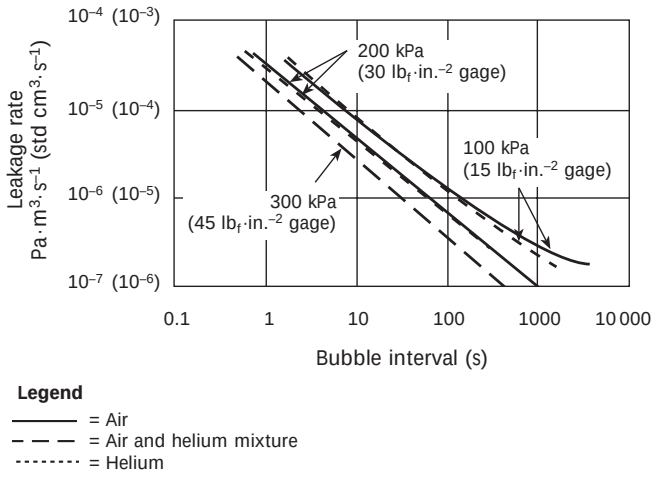
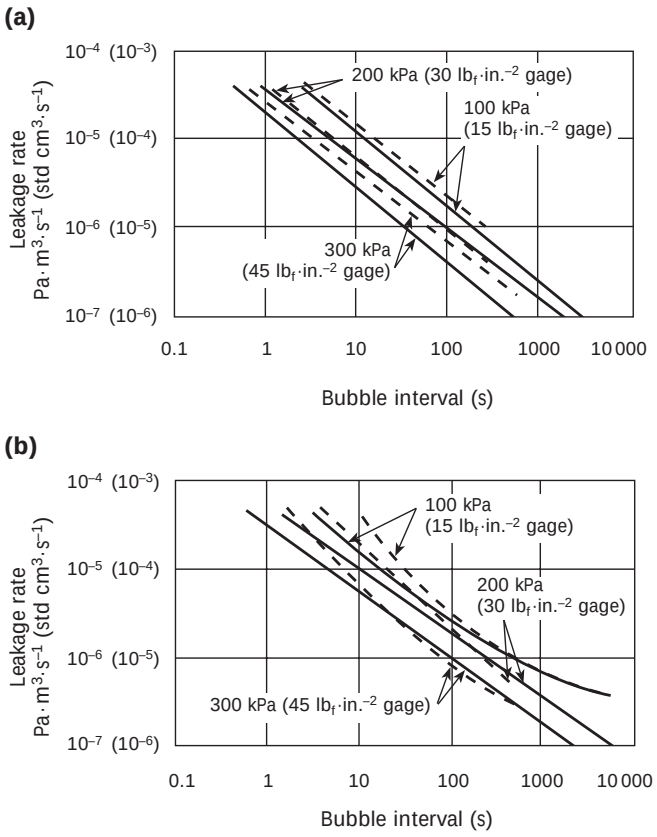


FIGURE 13. Effect of pressure and gas on detectability at 25 °C (77 °F): (a) using mineral oil number 1; (b) using glycol.



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especially when they have low flash points and vapors collect over exposed immersion baths. Safer tracer gases and immersion bath liquids should be used whenever possible.

Sealing and Pressurization of Test Components for Bubble Testing

Before application of internal pressure and immersion of test object surfaces in bubble test liquids, test object surfaces should be cleaned of oil, grease, scale, weld slags and other foreign materials. Cleaning solvents should be those specified for the particular materials and assemblies in applicable test procedures, process specifications or procurement specifications. As applicable, plugs, covers, sealing wax, cement or other suitable material may be used as sealants to exclude inspection fluids from entering test components. Sealing materials must be completely removed on the completion of the test. They must not be injurious to the test parts or assemblies or to the purpose of the equipment.

When large components are to be pressurized for leak testing, two indicating calibrated dial pressure gages should be connected to the component. One gage should be readily visible to the operator controlling the pressure. Where required by process specifications or procurement specifications, a calibrated recording type pressure gage should be substituted for one of the dial gages. For back pressurizing leak testing techniques, one gage attached to the pressurizing chamber is satisfactory.

Unless structurally limited or otherwise specified, the minimum pressure differential between the pressure of the gas within the test object and the external pressure at the greatest depth in the immersion liquid should be 100 kPa (15 $lb_f \cdot in.^{-2}$ gage). The test pressure of the gas during immersion testing in water can be calculated by means of the following: required test pressure = 100 + 10*D* kPa (15 + 0.04*D* $lb_f \cdot in.^{-2}$) gage, where *D* is the maximum depth of the test part in immersion liquid, in meter or inch.

The test pressure must not exceed 125 percent of the maximum allowable working pressure at the test temperature for the test vessel, component or assembly unless analysis shows a higher pressure to be nondamaging. Test pressures and test procedures must conform to any other limitations and requirements specified in applicable codes, test specifications or procurement specifications. The test object surface to be inspected must be at least 30 mm below the surface of the immersion test liquid at all points. The test object must be secured, if necessary, against buoyancy (tendency to float) or uncontrolled movements within the immersion liquid during the period of inspection.

Conditions for Visual Inspection of Bubble Indications in Immersion Liquid

The test object surface area of interest during bubble immersion leak testing must generally be nearly parallel to the surface of the inspection liquid. This will allow bubbles formed anywhere on the inspection surface to flow directly to the liquid-air surface without hitting or being obstructed by fixtures or part appurtenances. This may necessitate visually inspecting a portion of the surfaces and then repositioning the test objects for inspection of other previously masked or hidden surfaces.

Lighting in the area to be examined should be no less than 1 klx (100 ftc) in brightness. Illumination should be free from shadows over the surface areas under inspection. A photographic exposure meter or a light meter can be used for checking the light intensity in the immersion inspection area. It must be possible for observers to place their eyes within 0.6 m (2 ft) of the surface to be examined. The observers' angle of viewing should not be less than 30 degrees to the plane of the surface being examined. Mirrors or magnifying glasses may be used to improve visibility of indications. Preferably, the surfaces to be inspected should be in a horizontal position at a depth below the liquid surface adequate to permit easy observation of bubbles. Care must be taken to eliminate any hazards from pressurized gases or from immersion test liquids for those observing bubble leakage indications.

Interpretation of Bubble Indications in Immersion Bubble Testing

Before visual inspection, all gas pockets formed by immersion of the test object or resulting from gases in liquid suspension must be removed from the test object surfaces. This may be done by any feasible technique of removing the adhering gas such as wiping, brushing, scraping or rolling the test surface. To prevent formation of gas bubbles on the surface of the test part caused by gases in suspension in the immersion solution, the

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temperature of the test part must be within 6 ˚C (10 ˚F) of the temperature of the test liquid into which the part is submerged.

Excessive vacuum above the immersion liquid, when using the vacuum differential technique, may cause the test liquid to boil. When this occurs, the pressure on the liquid in the vacuum box or chamber should be increased until the boiling evolution of bubble has ceased. Contamination of test object surfaces can also lead to evolution of gas bubbles when adhering gas is released. The leak testing operator should be fully aware of the possibilities for each of the preceding effects to interfere with the operator's ease of detecting bubbles from leaks. The operator must take measures to ensure that these and other types of nonsignificant bubble emissions are eliminated during leak testing observations.

Evaluation of Immersion Bubble Test Results

Leakage is usually cause for rejection of the test part in the immersion bubble testing technique except when leakage is specifically permitted by the test specification. When one or more bubbles originate from a single point and are observed to grow or release from that point, the indication shall be interpreted as leakage. The point of origin of the bubble indications is interpreted as the location of the leak (exit point of leakage). Leakage is the cause for rejection of the test part in most industrial leak testing specifications.

When a leak is repairable in accordance with the applicable engineering drawing, process specification or procurement specification, the component under test may be repaired. After repair, the component should be reinspected in accordance with the original immersion bubble test specification. Any test object that shows no evidence of leakage (no bubble formation or emission can be seen) is typically evaluated as being an acceptable part or surface area.

Handling and Disposition of Test Objects after Bubble Testing

After bubble testing by the immersion technique, any fluid or gas that is known to be detrimental to the test object should be thoroughly removed. Before removing parts from the bubble test area, all acceptable parts must be separated from the rejected parts. Each part must be identified in such a manner that the acceptable and rejectable parts cannot be inadvertently mixed or interchanged. When the test object is of such mass or size that the leak test is performed at the site of the part of structure and localized leaks are detected and are to be repaired, the locations of these leaks must be identified in such a manner that the identification cannot inadvertently be removed. A leak location inspection record is recommended.

Production Line Immersion Bubble Test Facilities and Operation

The liquid immersion technique of bubble testing can be applied to a continuous manufacturing process. For example, as the component parts move along the production line toward final assembly, they can be pressurized with dry air and then immersed in clean hot water baths. The water should be treated with a suitable water softener to reduce surface tension and promote bubble growth, thus increasing the sensitivity of the test. Operators in front of large glass windows can detect leaks under optimum viewing conditions. (In many cases, illumination and design of these bubble test facilities can be similar to large, well designed aquarium displays in museums or zoos.) When leaks are detected, the components can be tagged as they leave the immersion inspection baths. The anomalous units are returned for repairs and recycling through the dip tank used for bubble testing. One of the outstanding limitations of the production line conveyorized immersion bubble tests is caused by the amount of entrained air carried into the drip tank on the surface of the test objects.

Time Exposure Photography of Bubble Stream in Immersion Bubble Tests

For photographic recording of bubble streams during immersion leak testing, the test part is pressurized with nitrogen or helium gas and submerged in a liquid environment such as water or liquid refrigerant. A time exposure photograph is then taken of the suspected area of leakage. The time exposure photograph shows the gas bubbles produced as solid stream with an abrupt termination and makes pinpointing the leakage sites much easier. Typically, physical features on the part surface will serve as indexing references to facilitate location of the

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leaks once they have been observed in the photograph. This frequently results in reducing the area that must be visually searched with a magnifier to less than a few square millimeters.

Automated Laser Beam System for Detection of Bubble Leakage Indications of Cartridges

Frankford Arsenal (United States Army) has reported a laser beam photooptical system for detection of leakage bubble streams escaping from military cartridges. This automated cartridge waterproof tester automatically receives each cartridge, tests it for compliance with the waterproof specification and segregates the accept/reject items. The principal component of the system is the vacuum test chamber that contains the immersion test liquid (water at reduced pressure) and houses the electrooptical leak sensors. A vacuum pump accumulator subsystem with variable pressure controls supplies the test chamber with the specified vacuum. The drive motor, laser, vacuum pump and solenoid valves are electrically operated. The test chamber is fitted with automatic feed, process and extraction mechanisms that move the cartridge during the test cycle. The input device and door mechanism are pneumatically operated. A single gear motor is used to drive the mechanism internal to the test chamber and to provide timing functions for the entire sequence.

The laser optical bubble detection subsystem consists of a 3 mW optical laser, an optical beam splitter and a prism. Laser beams scan two collecting funnels for air bubbles escaping from either the bullet or the primer ends of the cartridge.

The leak detection signal readout system consists of two phototransistor detectors, two photoamplifiers, logic control and power supply. This leak detection system provides a 5 V output signal to indicate an acceptable cartridge for the test in progress.

Control of Vacuum and Depth of Immersion Fluid

Testing of cartridges is conducted under the prescribed reduced pressure supplied by a vacuum pump and accumulator connected in series with a parallel arrangement of two solenoid valves, one large capacity valve and one metering valve. These solenoid valves are controlled by two separate pressure switches. The large capacity valve is shut off at a vacuum pressure of 54 kPa (16 in. Hg) and the metering valve is shut off at 50 kPa (15 in. Hg) pressure. The final pressure is held within ±1.5 kPa (±0.25 $lb_f \cdot in.^{-2}$). Test time is cam controlled within 1 s of that specified by the applicable cartridge specification. The test sequence is typically conducted at 100 kPa (30 in. Hg gage) pressure for a period of 30 s.

Water level in the vacuum chamber is maintained at a level 50 to 70 mm (2.0 to 2.7 in.) above the cartridge under test. The water level is maintained manually by adding water through the exit port. A drain is supplied at the bottom of the vacuum chamber.

Laser Optical Subsystem for Automated Bubble Testing of Cartridges

The laser optical subsystem uses a 3 mW laser light source. The laser beam is split into two beams by a beam splitter prism. The two beams are then projected through an optical quality glass window into the test chamber over the front and rear collection funnels. The beam paths are adjustable by loosening the screws of either the beam splitter or prism, depending on which beam needs correction.

The timing controls operate by timing cams using microswitches. The electronic package consists of circuitry necessary to drive an output signal from phototransistor leak sensors and to adjust the output sensor signal to trigger a gate when a preset light level is attained. Circuitry is also provided for an empty feed chute indicator and activates stop system modes when a cartridge is lost or jammed.

Bubble Meter to Measure Gas Leakage Rates in Immersion Bubble Tests

A conceivable technique for quantitative measurements of gas leakage flow through leaks observed with immersion bubble tests would involve collection of rising bubbles into an inverted funnel placed above the points of leakage. If this gas were then conducted (as by glass or flexible tubing) to a suitable flow meter, precise flow rates might be measured.

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PART 3. Bubble Testing by Liquid Film Application Technique

Technique of Liquid Film Application (Solution Film) Bubble Testing for Leaks

The liquid film application technique of bubble testing can be used for any test specimen on which a pressure differential can be created across the (wall) area to be tested. An example of this technique is the application of leak test solutions to pressurized pipe line joints. This test, also known as a solution film test, is most useful on piping systems, pressure vessels, tanks, spheres, compressors, pumps or other large apparati with which the immersion techniques are impractical. The test liquid is applied to the low pressure side of the test object area to be examined so that joints are completely covered with the film of bubble forming liquid. The surface area is then examined for bubbles in the solution film.

Unless otherwise specified, the test object must be pressurized to at least 100 kPa (15 $lb_f \cdot in.^{-2}$ gage) with test (tracer) gas. In no case should the test pressure exceed the specified maximum allowable working pressure for which the test object has been designed unless analysis demonstrates that higher pressures are not damaging.

The area to be inspected should be positioned to allow, if possible, the test liquid to lie on the surface without dripping off. Where necessary, it is allowable to position the test surface so that the inspection liquid flows off the test area, provided that a continuous film remains over the test area. All-position testing may be performed on large pressure vessels, weldments, tanks, spheres, compressors, pumps and other large apparati.

When one or more bubbles originate, grow or release from a single point on the test object surface, this bubble formation should be interpreted as leakage. The point at which bubbles form should be interpreted as the origin of leakage (the exit point of a physical leak). Usually, any component that does not show evidence of leakage is evaluated as acceptable. Leakage is cause for rejection of the test part except as specifically permitted by the test specifications. Where the leak is repairable in accordance with specifications, the component may be repaired and reinspected in accordance with the original accepted leak testing procedures. After testing, any liquid or gas detrimental to the test object should be thoroughly removed.

Selection and Application of Bubble Forming Solution Films

The bubble forming solution used with the liquid application technique of bubble testing should produce a film that does not break away from the area to be tested. The solution film should produce bubbles that do not break rapidly due to air drying or low surface tension. Ordinary unmodified household soap or detergents should not be used as substitutes for specified bubble testing solutions for critical applications. The number of bubbles contained in the solution during application should be minimized to reduce the problem of discriminating between leakage bubbles and bubbles caused by the solution. In principle, a bubble will form only where there is leakage. No liquid should be used that is detrimental to the component being tested or other components in a system.

Example of Soap Solution for Bubble Testing for Noncritical Applications

An industrial fabricator using bubble liquid film application leak testing extensively on large structures has described a special modified soap film solution, used when specifications, standards or codes allow its use. It consists of a household dishwashing liquid detergent or liquid soap mixed with glycerine and water in the following proportions:

1 part of liquid detergent or soap,
1 part of glycerine, and
4.5 parts of water.

A typical small batch might be prepared where each of the above parts is 1 L or where each is 1 pint, for example. The solution is prepared in advance to allow the bubbles and foam to disperse before it is used for bubble testing.

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Modified Soap Solution for Bubble Testing in Freezing Weather

To prevent the soap film solution from icing in the applicator container in cold weather when the temperature is below freezing, alcohol or ethylene glycol (antifreeze) can be added to the soap film solution described above, in the proportion of 1 part of antifreeze to 10 parts of the modified soap solution.

Alternate Modified Soap Solution for Foam Detection of Large Leaks

The following leak detector mixture is used for detecting large leaks when pressure testing or vacuum box testing. This mixture is prepared by combining one part of liquid soap or detergent with one to two parts of water. To prevent the mixture from icing in the container, if the temperature is below freezing, add alcohol or ethylene glycol (antifreeze) in a proportion of about one part of antifreeze to ten parts of mixture. Just before using, the mixture is agitated until a thick foam or suds is formed. This foam solution is used to detect large leaks.

Limitations of Common Soap Solutions for Bubble Emission Tests

The most common bubble test liquid used in nontechnical applications is a simple soap solution such as a diluted dishwashing liquid or liquefied soap flakes. The main advantage of using soapy water for the bubble testing liquid in solution film tests is its low cost. Soap solutions are much less expensive than commercial leak testing liquids described later in this chapter. However, common soap solutions typically have the following disadvantages when used as leak testing liquids.

1. Soaps ordinarily form sticky, gummy curds with the minerals in hard water. The bath tub ring, a common indication of this property of soap solutions, shows how tenaciously soap curd deposits stick to any surface. In fact, the soap curds may plug small leaks, at least temporarily. Soaps that do not form curds with hard water contain additives or complexing agents for mineral salts that may introduce unknown contamination and other complications in bubble testing.
2. Most soap solutions are alkaline, with pH values of 10.5 to 11.5. This alkalinity of soap solutions is acceptable for use in leak testing of noncritical iron or low carbon steel equipment. However, such alkalinity could cause corrosion on aluminum alloys if allowed to remain in contact with the metal for some time. Neutral soaps generally contain additives that reduce the foaming ability and foam stability of the solution film unless still other counter additives are used.
3. Soaps are salts that conduct electricity and they often contain salt impurities or salt additives. This may be important in leak testing of electrical and electronic equipment because any residue might result in electrical leakage paths.
4. Soaps may contain chlorides as impurities and some soaps contain borax as an additive (because of its cleaning power). Chlorides and borates are undesirable when testing stainless steels or titanium because they promote stress corrosion cracking. Many commercial surfactants contain chlorides. For example, the cationic types of surfactants are generally chloride salts. Corroded welds might result from similar conditions and would be prohibited in nuclear generating and other critical equipment, for example.
5. Many soaps contain chemically unsaturated compounds which, under certain conditions, are dangerous when in contact with concentrated oxygen. For example, if soap residue is left on pipe threads and the pipe connection is tightened or loosened while in contact with oxygen, an explosion could result. Soaps should not be used in leak testing of oxygen systems unless they have been tested chemically and found to be free of unsaturated compounds. For use where residue may come into contact with liquid oxygen, only surfactants that meet the United States Army Ballistic Missile Agency's oxygen impact test, ABMA-PD-M-44 (July 1958), should be used.[3]

Despite the disadvantages listed above, soap and water solutions are very commonly used for bubble testing on noncritical items.

Advantages of Commercial Chemical Bubble Testing Solutions

Technical applications and specifications for bubble testing in industry typically indicate that a solution of commercial

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leak testing liquids shall be used. Soap suds or household detergents and water are not considered to be satisfactory leak test liquids for critical bubble leaks. The test liquid should be capable of being applied free from bubbles so that bubbles appear only at leaks. The liquid selected should not bubble except in response to leakage. Typical properties of commercial leak testing liquids (in contrast with the properties of soap solutions listed earlier) are the following.

1. Suitable leak test liquids meet specifications calling for a neutral range of pH between 6 and 8. If a higher pH is required for a particular purpose, it usually can be supplied.
2. Suitable leak test liquids do not form deposits even when mixed with hard water. This avoids the possibility of accidental plugging of leaks by formation of curds or other deposits on surfaces.
3. Suitable leak test liquids are typically formulated with viscosity such that a small amount spreads over the test area and stays in place for an extended period of time.
4. Commercial leak test liquids are often available in convenient containers such as small plastic squeeze bottles, bottles with daubers, brushtop bottles, spray bottles and the like. Bulk containers of leak test fluid are also typically available for large scale usage.
5. Suitable leak test liquids are stabilized and immune to bacterial action and maintain desired properties over long storage periods before use.
6. Suitable leak test liquids typically are designed to allow test surfaces to dry to a clean state, so that cleaning after bubble testing is usually not necessary. This requirement is not always met because most liquids using softeners leave the softener as a residue.

In addition, many commercial leak test liquids are designed for use under special conditions, such as high or low temperatures, on reactive metals or plastics, on liquid or gaseous oxygen systems or on electronic components. Such liquids offer specific advantages and may prevent hazards or damage to test materials that might result from soap solutions or improperly selected test liquids.

Characteristics of Commercial Bubble Testing Solutions

The great variety of commercially available liquid film solutions for bubble testing permits selection of products optimized for specific applications and problems. Typical factors to be considered in selecting the detection fluid for specific applications include the following.

1. What gas is it desired to detect? Specific test liquids are available for detection of substances that include compressed air, oxygen, hydrogen, flammable gases, refrigerant gases, carbon dioxide, ammonia and many toxic gases. Nonorganic test liquids, free from oils, fats, ammonia or other materials that would be inflammable in contact with pure oxygen, are available and are mandatory for use in hazardous cases.
2. What materials are involved in the test objects to be inspected for leaks? Stainless steels, titanium alloys and even polyethylene pipes and valves are either subject to stress corrosion or are easily stress cracked in the presence of certain chemicals. The leak test liquid for use on sensitive materials must be selected to avoid possibilities of damage or deterioration. Also important is the problem of cleanup of test surfaces after leak testing. Bubble test liquids that evaporate to leave clean test object surfaces are desirable when feasible.
3. How small a leak is it desired to detect? Leak testing liquids for bubble tests are available in a wide range of sensitivities. Some are extremely sensitive, whereas other products are made for the detection of large leaks. Consultation with manufacturers of proprietary leak detection liquids may be desirable in critical applications.
4. How large is the area to be tested and where is it located? Various types of applicators are available with which to apply the leak test liquid to out-of-the-way spots, in bubble film application testing. Other products have superior stability characteristics that enable them to provide stable films that stay in place over extended time periods on large areas while inspection is carried out.
5. In what temperature range will the leak testing be done? Manufacturers of commercial leak testing liquids can provide solution film products for use at temperatures varying from –55 to 210 ˚C (–67 to 410 ˚F). Specific bubble test liquids are formulated for specific temperature ranges and environments.

No single leak test liquid can be considered to be adequate or desirable for all inspection conditions encountered in solution film bubble testing. Thus, a selection is desirable for each class of tracer gas (or gas within a pressurized system) and for various types of test object materials and operating conditions.

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Leak Testing Fluid Concentrates for Dilution in Water

Commercial low cost leak tests liquids can be prepared from chemical concentrates that are mixed in various proportions with water to provide solutions for use in air from low to high temperatures. The foaming concentrate is usually mixed as 1 part of concentrate in 160 parts of water for regular leak testing in air above freezing temperatures. The mixed solution is applied to the test surface with brush or swab. Large leaks instantly form large bubble clusters. Very small leaks form clusters of white foam that build up for many minutes to aid in pinpointing leak locations. The liquid contains no grease, oil or soap, and there is no need to remove it before painting.

A low temperature version of the leak test concentrate is designed for leak testing below freezing temperatures. The recommended dilution in water varies with the operating temperature of the detection liquid. For temperatures in the range of 0 to –10 ˚C (32 to 14 ˚F), one part of concentrate is mixed with four parts of water. However, below –10 ˚C (14 ˚F), one part of concentrate is mixed with two parts of water. Application is by brush or swab, with the brush kept sopping wet. It is not proper to work up a lather in this inspection liquid.

Leak Testing Liquid for Aerospace Oxygen Systems

Leak testing liquids made to meet United States Air Force Specification MIL-L-25567D(1)[4] are precision formulated neutral compounds for leak testing of oxygen and air lines, cylinders, tanks, fuel lines, pneumatic controls and sealed components on aircraft or missiles. Type I fluid is designed for use at temperatures from 2 to 70 ˚C (35 to 160 ˚F). Type II fluid is intended for use at temperatures from –54 to +2 ˚C (–65 to +35 ˚F). These leak test liquids permit safe use on oxygen, nitrogen, helium, air and other gases. They contain no oils, fats, ammonia or other materials that could be flammable in contact with pure oxygen. The solution pH is 6.0 to 7.5 at 21 ˚C (70 ˚F). Residual solids are rated as no more than 0.40 percent. There is no need to remove the film solution after application. The solution is reported to be noncorrosive, nontoxic, noninflammable and noninjurious to skin, eyes, plastics, rubber and finishes.

Bubble Test Liquid Concentrate for Use with Pure Oxygen and Compressed Gases

A leak test solution concentrate is designed for commercial and industrial use for leak testing of lines, cylinders and tanks of pure oxygen and compressed gases. This solution concentrate contains no oil, grease or any other ingredient that could combine with pure oxygen to form either a flammable or explosive mixture. This solution is safe with either high or low pressure oxygen and all other compressed gases. The solution is applied by dauber, paint brush or squeeze bottle. In solution film bubble tests, large leaks form white foam that builds up for half an hour or more, aiding detection. This solution does not have to be removed after testing either for appearance or for painting of test surfaces. This material has been tested and approved by both governmental and commercial laboratories, including the high pressure bomb test with pure oxygen.

Solution Film Leak Testing Fluid for Spray Application to Large Areas

A leak test fluid for spray application by large tank sprayers or hand sprayers spread rapidly over and around test surfaces and can be applied to parts or systems that can be leak tested under air or (natural) gas pressure. Tube and pipe connections need be sprayed only from one side because the liquid wraps around and wets the opposite side. Spray application is much faster than application with a brush or dauber. Applications include leak testing of pneumatic lines, controls, panel boards, hydrogen cooled generators, refrigeration and air conditioning systems, gas lines, tanks and cylinders.

Solution Film Leak Testing Liquid for Tests of Chlorine Systems

Leak testing of systems pressurized with chlorine gas is feasible with a specially formulated test solution that traps the escaping chlorine gas in clusters of bubbles. When the bubbles break, they sometimes emit highly visible puffs of smoke that help to locate the points of leakage. This particular formulation is used in sewage treatments plants, water purification systems and chemical synthesis operations. Because chlorine vapor is heavier than air, it tends to fall away from the leak sources. This may give the impression that the leak is in an adjacent area below the actual leak. Because of the chlorine health hazards, the maximum allowable concentration for

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an 8 h exposure is only 1 $\mu L \cdot L^{-1}$. The longer the gas continues to escape before the leak is located, the greater the danger. Bubble testing permits relatively rapid leak location, as compared with sniffer tests or other leak testing techniques and so minimizes the danger of personnel exposure over long periods.

Leak Testing Liquid for Use on Refrigeration and Air Conditioning Units

A commercial leak testing liquid developed specifically for detecting leaks in refrigeration and air conditioning equipment and systems works on the bubble emission principle but is not a soap solution. This chemical solution contains no oil or grease and dries clean. It is nontoxic and nonflammable. The liquid is applied to the outside of the connection or surface to be tested by brush, swab, squirt bottle or spray. Large leaks show up immediately as clusters of large bubbles. Small leaks cause a buildup of white foam that becomes clearly visible in 10 s to a minute or more, depending on the rate of leakage. This ball of white foam remains clearly visible for as long as 30 min. Thus, this technique can be used for testing large numbers of connections or components. Even if the leak itself is out of sight, the cluster of foam is usually visible. Tests on controlled leaks that lose refrigerant gas at the mass rate of 0.5 kg (1 lb) in 100 yr are clearly detected with this liquid in less than 60 s.

Film Solution for Leak Tests of Vacuum Systems and Electronic Components

Film solution leak testing liquids have been developed that are suitable for leak testing of transparent vacuum system components, for leak testing of sealed electronic components and for detection of large leaks into opaque parts of vacuum systems. Certain types of leak detection fluids have been developed that are completely nonionic and do not conduct electricity. Others are engineered to be compatible with most types of vacuum systems. On evaporation, their residue content is very small and does not develop toxic, corrosive or flammable conditions. Leaks in small sealed electronic components (even electrolytic capacitors that contain some gas) can be found by immersing them in a solution film leak testing solution and then removing them and placing them under a glass bell jar or transparent enclosure that is evacuated. The thin film of test solution that remains on the outer surfaces of the component shows bubbles or foam at points of leakage, before the absolute pressure is reduced sufficiently to cause the film solution to boil.

For leak testing of vacuum systems whose interior is visible (as through glass ports or within glass tubing) the test solution is applied to areas suspected of possible leakage on the outside surface of the system. A test solution with very good wetting properties penetrates small holes or porous welds and foam becomes evident at points of in-leakage. If the interior of the vacuum system is not visible, it is possible to shake the container of test solution and apply foam to any suspected leak areas. The test solution provides a strong, long lasting foam that persists when indications form. This use does not apply to high vacuum systems whose leaks are very small. However, the disappearance of foam (on the outside surface as it is sucked into the system) indicates leaks of medium size.

Another form of film solution leak testing fluid contains fluorescent dye tracer. This solution is used for tests on vacuum systems that can be disassembled for examination. The fluorescent solution is applied to the outside surfaces of the evacuated system, which later (after allowing time for penetration through leaks) is taken apart and examined under near-ultraviolet radiation. Penetration at any point is then indicated by flowing fluorescent indications (similar to those of fluorescent liquid penetrants). Improper positioning of O-rings, dysfunctional port or door seals and imperfect gaskets, for example, are easily detected by this technique.

General Technique for Solution Film Bubble Tests

A solution film test is performed with a differential pressure applied across the pressure boundary under test. A film of leak testing solution free of bubbles is applied to all suspect areas and areas requiring test on the lower pressure side of the test boundary. The operator then observes the film of test solution for bubbles indicating small leaks. The solution film test is particularly appropriate for detecting small leaks when pressure testing (Fig. 14) or testing with a vacuum box at a moderate vacuum level (above 50 kPa or 8 $lb_f \cdot in.^{-2}$ absolute). Figure 15 shows typical designs of vacuum boxes used in leak testing. Vacuum boxes are designed to withstand external atmospheric pressure (100 kPa or 15 $lb_f \cdot in.^{-2}$ absolute) and are shaped to fit the contour of the test boundary being bubble tested (see Fig. 16).

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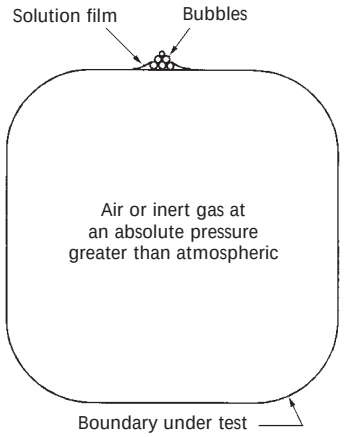
FIGURE 14. Pressure technique of film application bubble testing



Alternative Technique Using Thick Layer of Suds or Foam

Alternatively, a mixture of thick foam or suds is applied to all suspect areas and areas requiring test on the lower pressure side of the test boundary. The operator then observes the surface of the foam or suds for blowouts indicating large leaks. This technique is used for detecting very large leaks when testing with a vacuum box at low vacuum levels (15 to 30 kPa absolute or 2 to 4 $lb_f \cdot in.^{-2}$ absolute). Blowout leaks will clean the original foam or suds off the leak very quickly. No subsequent indication of bubbles occurs. Test operators must be aware of this condition and observe the foam blanket as it is applied.

FIGURE 15. Examples of vacuum boxes used for bubble emission tests on large structures: (a) standard aluminum vacuum box for bubble leak testing of straight weld seams; (b) cross sectional view; (c) inside corner weld seam vacuum box.

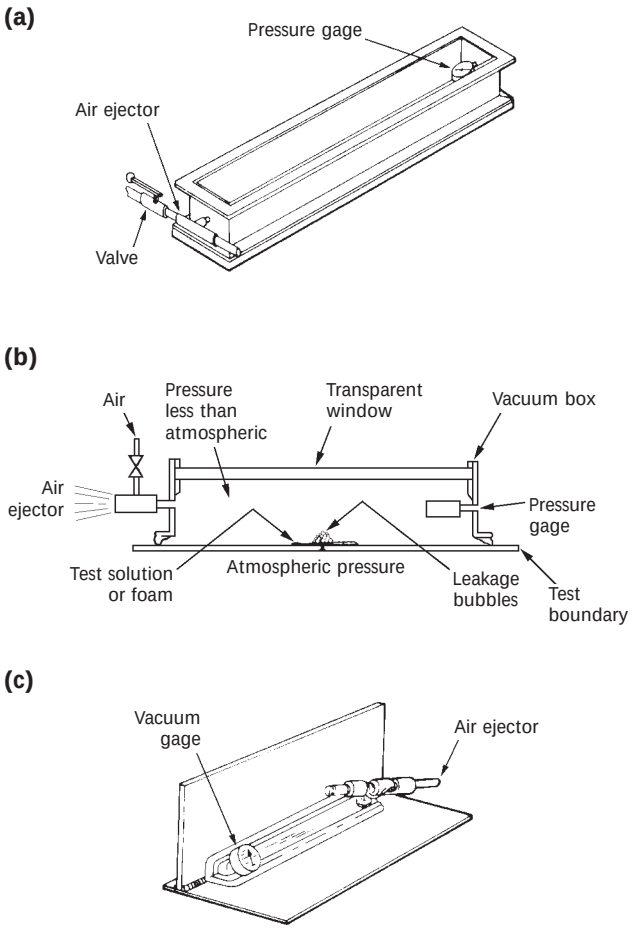
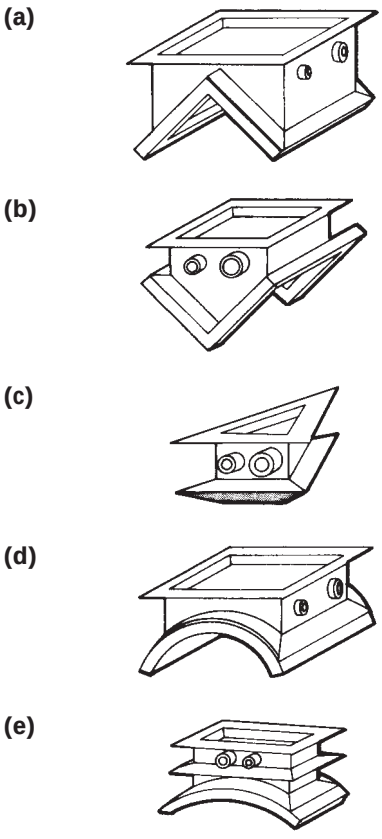


FIGURE 16. Vacuum boxes to fit special structural shapes: (a) for outside straight seams; (b) for inside corner intersections; (c) for inside straight seams; (d) for circumferential pipe seams; (e) for circumferential tank seams.



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Sensitivity of Solution Film Bubble Tests

The sensitivity of the solution film bubble type of leak test in a shop or field environment will enable detection of leakage of 10^{-3} to 10^{-4} Pa·m^3·s^{-1} (10^{-2} to 10^{-3} std cm^3·s^{-1}) when the differential pressure across the leak is 100 kPa (1 atm). When the factors affecting leak test sensitivity are rigidly controlled, as in a laboratory or research investigation, it is possible to detect leakage in the range of 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}) with a pressure differential of 100 kPa (1 atm), except in broad areas of very fine weld porosity. Factors affecting the sensitivity of leak testing by the solution film bubble technique include the following:

1. differential pressure across the test boundary (for tests near pressure of 100 kPa or 1 atm, the sensitivity will vary approximately with the difference in the squares of the end pressures);
2. viscosity of the pressurizing gas (the sensitivity of the bubble test will vary approximately inversely to the viscosity of the tracer gas);
3. surface tension of the leak solution (surface tension of the bubble forming solution should be lowered to increase the leak test sensitivity);
4. cleanliness of the test object surface area being inspected (to which the solution film is applied) and the cleanliness of the opposite side of the pressure boundary (interior wall of the test vessel);
5. skill and experience of the operator;
6. adequacy of lighting in the area where bubbles must be observed and freedom from glare caused by bright lights in the field of vision or by background illumination with excessive contrast;
7. time required to develop bubble and duration of observation time used to see bubble indications from leaks (the test sensitivity increases with an increase in duration of time of observation); and
8. environmental or weather conditions in the leak testing area.

In reference to weather, it should be noted that when tests are conducted outdoors, factors such as the temperature, wind and precipitation can be detrimental and may require postponement of tests or selection of suitable times and sites for bubble testing. Strong winds tend to disperse the leak test solution and bubbles formed by leakage, thus masking the areas of suspected leaks. Precipitation tends to wash away the testing solution as it is applied or to dilute the solution with water. When it is very hot and the sun is shining brightly, it is difficult to conduct the bubble test due to the fast evaporation of the moisture from the solution film test liquid. To conduct bubble tests at temperatures below freezing, it is absolutely necessary to use a specially prepared leak testing solution designed for low temperatures.

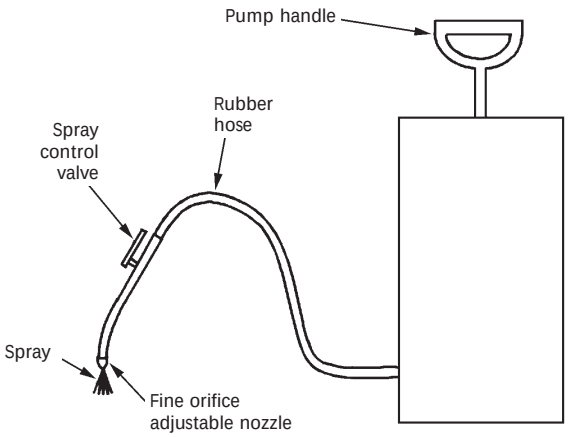
Practical Procedures for Pressure Bubble Testing in the Field

Before leak testing of large steel construction, tanks, pipes, pumps and assemblies, it is essential to remove all slag, mud, dirt, debris and contaminants from the weld seams, plates, pipe joints and other areas to be tested. When inspecting for small leaks, the test solution is applied to the test surface in a continuous film free of bubbles by one of the following techniques listed in order of decreasing preference: (1) spray application using a pump type garden spray can with a fine orifice (Fig. 17); (2) spray application using a plastic squeeze bottle or an oil squirt can; or (3) brush application using a short handle painter's brush, 25 to 75 mm (1 to 3 in.) wide.

When using a brush, do not apply the leak detector solution by stroking movements. Apply the leak detector solution by holding the wetted brush just above the test area and allowing the solution to flow over the test area.

Adequate lighting must be provided around the areas being tested. For best contrast, it is desirable to shine the light beam nearly parallel to the test surface. To provide a further increase in test

FIGURE 17. Hand pump pressure can garden spray unit for film application of bubble leak detection liquids.



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sensitivity, the film solution and bubble indications can be observed with the aid of a pocket magnifying glass of 2× to 3× power.

Air pressurization of simple test objects such as reinforcing pad plates of pressure vessels can be done economically with a hand operated tire or bicycle pump equipped with a shutoff valve and pressure gage. All openings in the test boundary are blanked by covering and sealing them. The test boundary is then pressurized by systems such as a tire pump. Warning: Positively do not use oxygen, acetylene or any flammable fluid or toxic gas as the pressurizing medium.

In the event of inclement weather, such as strong wind or precipitation, the solution film bubble test can be postponed or portable shields can be used over and around the areas being tested.

When leakage is observed, the areas of the leaks are marked and repaired after completion of the leak test. Before repairing any leaks or doing any work that might cause a spark, the vapor space within test enclosures should be tested to make sure that it is free of explosive mixtures.

Estimating Approximate Leakage Rates by Solution Film Tests

The following are examples of techniques that can be used to estimate leakage rates from bubbles formed by solution film bubble tests. At best, test techniques are very crude but could be very valuable in estimating the size of a leak or leaks found with solution film bubble tests of an evacuated cryogenic vessel that had initially failed a pressure rise test. The approximate results obtained could tell the operator whether further testing were necessary or if repair of the leak or leaks would be sufficient to enable the vessel to pass a second pressure rise test.

Appearance of Single Bubble at Leak

Suppose that a leak is indicated by the appearance of a single bubble at the point of leakage in a solution film test where the pressure differential is 100 kPa (1 atm). For ΔP values other than 100 kPa (1 atm), the leakage rate can be determined by using the pressure relationship for viscous flows. To determine the approximate leakage rate, the operator can measure the time that elapses before the bubble reaches a specific size. The estimated leakage rate for the case of a single bubble could be determined by Eq. 5:

$$\textbf{(5)}\quad Q = \frac{\pi d^3}{60\,000\,t}\ \mathrm{Pa \cdot m^3 \cdot s^{-1}} = \left(\frac{\pi d^3}{600\,000\,t}\ \mathrm{std\ cm^3 \cdot s^{-1}}\right)$$

where d is bubble diameter (millimeter) and t is time (second). To illustrate Eq. 5, suppose that it takes 7 s for a single bubble to reach an estimated diameter of 6 mm (0.25 in.). The leakage rate in this case would be equal to $\pi\,(6^3)/(60\,000 \times 7) = 1.6 \times 10^{-3}$ Pa·m^3·s^{-1} (1.6×10^{-2} std cm^3·s^{-1}).

Appearance of Many Tiny Bubbles at Leak

Suppose that a leak is indicated by the appearance of many tiny bubbles in the solution film. Because it is impossible to estimate the volume of the bubbles for a leak of this type, the operator could collect the bubble in an inverted test tube, which was previously calibrated in cubic centimeter. The elapsed time to collect 1 cm^3 (0.06 in.^{3}) is the reciprocal of the leakage rate. The estimated leakage rate could generally be determined by Eq. 6. In the centimeter-gram-second system of units, estimated leakage rate = (volume displaced)/(elapsed time):

$$\textbf{(6)}\quad \mathrm{Q} = \frac{V}{t}$$

In SI units, Pa·m^3·s^{-1} = $V/10t$ where V is in cubic centimeter and t is in second and tests are performed at normal atmospheric pressure of 100 kPa. If it took 85 s to collect 1 cm^3, the leakage rate is 1/850 Pa·m^3·s^{-1} = 1.3×10^{-3} Pa·m^3·s^{-1} (1.3×10^{-2} std cm^3·s^{-1}).

Calibration of Bubble Tests with Reference Standard Leaks

Reference standard physical leaks have been developed to provide known flow rates of various tracer gases or air. These calibrated leaks can be used with various bubble test fluids and pressurized gases to provide approximate calibrations relating bubble size and rates of emission to gas leakage rates. Gas flow meters can also be used to meter gas rates of flow to bubble testing calibration systems. However, it is generally possible to make approximate estimations of leakage rates from known bubble testing procedures, but precise calibration requires more advanced laboratory instrumentation.

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PART 4. Bubble Testing by Vacuum Box Technique

Application of Vacuum Box Bubble Testing

Vacuum box bubble testing provides for the detection of through thickness discontinuities in welds and pressure boundaries of systems containing air at atmospheric pressure. It is used during construction to test pressure boundary welds of incomplete systems that cannot be pressurized. It is also used to test pressure boundary welds that are inaccessible for leak testing when the entire system is pressurized. It may also be used to create a pressure differential for increasing the sensitivity of penetrant leak testing techniques.

Typical discontinuities detectable by this technique are cracks, pores and lack of fusion. A bubble forming solution is applied to the surface to be examined. A vacuum box with a viewing window large enough to view the test area and to allow sufficient light to enter the box for proper examination is placed over the test surface and then evacuated. A calibrated pressure gage is placed in the vacuum box system to verify the required pressure differential under test. The surface area visible through the vacuum box window is then viewed for evidence of through thickness discontinuities by the formation of bubbles on the surface. Through thickness discontinuities are indicated by the formation of a continuous chain of bubbles in the film solution. Through thickness indications are usually considered to be unacceptable and such welds should be repaired and retested. The formation of single small bubbles may or may not be considered relevant, depending on the type of test object and its intended applications.

Design of Vacuum Boxes for Bubble Testing in the Field

Vacuum boxes are available for rounded surfaces, corner seams and vertical seams. Typical designs of vacuum boxes for bubble testing in the field are illustrated in Fig. 15. Figure 15a shows a standard vacuum box with pressure gage in the vacuum enclosure. Figure 15b is a sectional view of that same vacuum box with the pressure gage within its vacuum enclosure. Figure 15c shows an inside corner weld vacuum box design, with a vacuum gage external to the vacuum enclosure. Figure 16 shows typical examples of commercially available vacuum boxes for various structural shape configurations. Each vacuum box has connecting fittings for external devices to pump air out and maintain a vacuum. The box should be able to withstand an external pressure of 100 kPa (1 atm). Flexible gaskets are provided to seal the enclosure to the test surface when pressure is applied to the vacuum box. A flat vacuum box should be of convenient size such as 150 mm (6 in. wide) × 750 mm (30 in.) opposite the open bottom. When a vacuum is developed within the void space of the box, the open bottom end is sealed against the test surface by a suitable gasket at the bottom edge of the box. Suitable connections, valves, lighting and gages should be provided, as described below.

Desirable Features of Vacuum Boxes for Bubble Testing

Vacuum boxes of varying configurations for application to specific shaped weldments can be purchased commercially or custom built (Fig. 16). Desirable features for a vacuum box are as follows.

1. Ability to readily admit natural or artificial light. This is done through the windows of tempered plate glass or of flexible transparent plastic material. Boxes built completely of transparent plastic material admit the most light.
2. Close proximity of viewing window to the surface of the weldment being inspected. This is accomplished by having the box shaped to the configuration of the surface area being tested.
3. Light weight for easy manipulation by one person.
4. Capability for easy initial seating when starting evacuation and good sealing properties to hold the vacuum. The features that have the most effect are the shape of the gasket (Fig. 18)

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and the flexibility of both gasket and vacuum box.

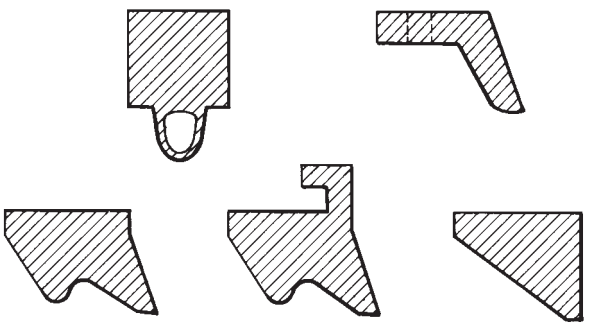
5. Equipped with a highly portable, readily available evacuation device with sufficiently high flow to be able to overcome a certain amount of seal leakage and rapidly evacuate the box to the required pressure differential.
6. Easily read dial gage. This will be a pressure gage if mounted inside the box or a vacuum gage if mounted externally on the box. The internal mounting provides better protection for the gage. However, when the gage is mounted externally, the vacuum box can be made with less depth. This places the viewing window closer to the weldment surface.
7. Quick acting valve for either shutting off the evacuation device or controlling the level of pressure differential.

Finally, a vacuum box for bubble testing should be checked for workability before performing leak testing to determine that the condition of the box components is such that it can attain a higher pressure differential than required for the test. For example, if the required pressure differential is 35 kPa (5 $lb_f \cdot in.^{-2}$), it would be prudent to want the box to be capable of attaining at least 55 kPa (8 $lb_f \cdot in.^{-2}$).

Design and Selection of Gaskets for Vacuum Boxes

The gasket system used with a vacuum box is critical to the ease with which the box can be handled and sealed to the test surface to hold a vacuum. The gasket must be shaped so the initial area of gasket contact with the test surface is small to make seating of the vacuum box easier. After initial seating, either the box or gasket or both should be capable of deflecting as the external pressure on the box increases. Then the contact area between gasket and weldment surface increases to a better seal. Boxes made completely of transparent plastic material are lighter in weight and are more flexible than boxes made partly of metal (usually aluminum). Hard rubber gaskets of 20 to 40 on the durometer scale provide a good flexibility. Some typical gasket cross sections are shown in Fig. 18. Figure 19 demonstrates why more pressure is obtained on a tapered sealing gasket versus a flat gasket with the same force applied to the vacuum box.

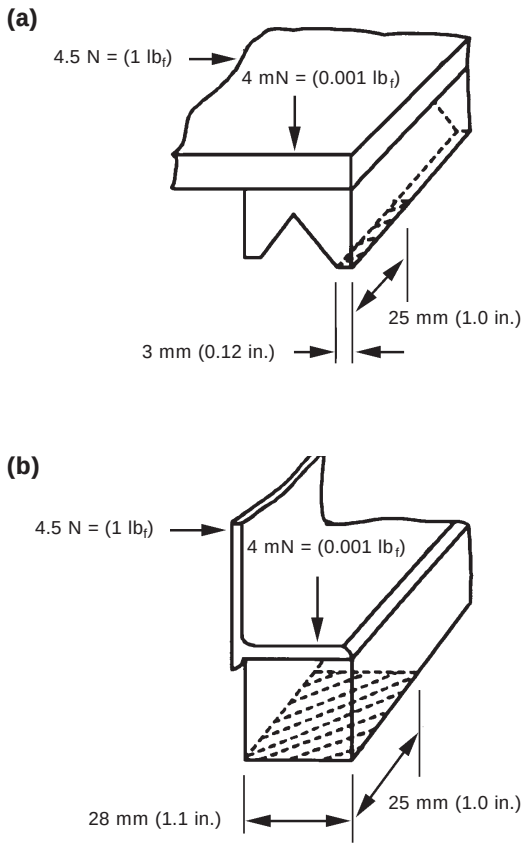
FIGURE 18. Cross sections of typical flexible gaskets used for sealing vacuum boxes to test surfaces for film solution bubble emission leak testing.



Evacuation System for Vacuum Box Leak Testing

In typical vacuum box bubble testing, the interior volume of the test object is open to the atmosphere or is filled with gas or air at 100 kPa (1 atm pressure or 15 $lb_f \cdot in.^{-2}$ absolute). The differential

FIGURE 19. Comparison of seating pressure for different gasket cross sections with the same force acting on the vacuum box: (a) pressure on tapered gasket = 4 mN/0.003 (0.025) = 53.3 kPa (= 7.7 $lb_f \cdot in.^{-2}$); (b) pressure on square gasket = 4 mN/0.028 (0.025) = 5.7 kPa (= 0.83 $lb_f \cdot in.^{-2}$).



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pressure that causes gas flow through leaks is created by the partial evacuation of the vacuum box. The evacuation system for vacuum box testing must be able to offset gasket leakage when initially seating the box. It must also be capable of quickly attaining and holding the desired vacuum for the test. The two most widely used evacuation systems are (1) a small box mounted air ejector connected to a compressed air supply and (2) a small portable vacuum pump.

The vacuum box, when placed over the examination area, should be evacuated to a specified pressure differential with respect to atmospheric pressure. The pressure differential can be verified by the dial gage. This vacuum should be maintained for a minimum specified time after the vacuum has been obtained. An overlap of at least 50 mm (2 in.) should be used for each subsequent area of examination along the seam.

Dial Gages for Vacuum Box Leak Testing

A pressure or vacuum dial gage must be readily visible to the operator controlling the pressure within the vacuum box during leak testing. Indicating pressure gages used in testing should preferably have dial graduations covering a range of 0 to 100 kPa (0 to 15 $lb_f \cdot in.^{-2}$ or 0 to 30 in. Hg). All gages used shall be calibrated against a standard dead weight tester, a calibrated master gage or a mercury column and recalibrated at intervals as required by the application test specification, standard or code.

Temperature of Test Surface during Vacuum Box Leak Testing

As a standard technique, the temperature of the surface of the part to be examined should not be below 4 ˚C (40 ˚F) nor above 52 ˚C (125 ˚F) throughout the examination. Local heating or cooling is permitted provided temperatures remain in the range of 4 to 40 ˚C (40 to 105 ˚F) during testing. When it is impractical to comply with these limitations, other temperatures may be used if the procedure is qualified in accordance with applicable specifications. In freezing weather, a nonfreezing film solution must be used for bubble testing.

The solution application time is critical, particularly if the surface is warm. At temperatures between 4 and 40 ˚C (40 and 105 ˚F), the solution should not be applied more than 1 min before examination for bubble emissions. Higher temperatures may be used, provided they do not exceed the maximum temperature compatible with the leak testing solution used.

Preparation of Test Surface for Vacuum Box Leak Tests

Before starting vacuum box leak testing, the surface areas to be tested should be free of oil, grease, paint and other contaminants that might mask a leak. If liquids are used to clean the component it should be thoroughly dried before testing. In general, satisfactory results may be obtained on welded structures or components when the surface is in the as-welded condition. However, before the vacuum box examination, the surface to be examined should be cleaned of all slag, scale, grease, paint and other material that would otherwise interfere with the test procedure or interpretation of results. Typical cleaning agents that may be used are wire brushes, detergents, organic solvents, descaling solutions and paint removers.

After wet cleaning, surfaces to be examined may be dried by normal evaporation or with forced hot air. A minimum period of time should be established and included in the written procedure to ensure the cleaning solvents have evaporated before the application of the bubble solution.

Pressure Test Objects during Vacuum Box Leak Testing

In many cases, vacuum box bubble testing is selected because the test object cannot be sealed off to be pressurized. In these cases and even with closed systems that are not pressurized, the pressure differential across the leak is the difference between the internal pressure of the test object (atmospheric if vented) and the external partial vacuum in the vacuum box.

However, in many cases, pressurizing the internal volume of the test object can increase the pressure difference and the rate of leakage through existing leaks, even during vacuum box leak testing. Before pressurizing the test component, all openings should be sealed using plugs, covers, sealing wax, cement or other suitable material that can be readily and completely removed after completion of the test. The pressure before examination should be held for some specified minimum soak time. Unless otherwise specified, the test gas will normally be air; however, other gases such as nitrogen or helium may be used.

Before using a very sensitive leak testing technique, it may be expedient to perform a preliminary test to find gross leaks. This may be done in any manner

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that will not seal or mask leaks during the specified test.

Selection of Bubble Test Liquid for Vacuum Box Tests

The leak test liquid used for bubble testing with vacuum boxes is typically a commercially available test fluid with the following characteristics.

1. It wets welded seams quickly and thoroughly when brushed or sprayed on because of a contained surfactant compound.
2. It bubbles vigorously at leaks and gives a copious stable foam.
3. It contains negligible halogen and sulfur.
4. It suitable for use on vertical welds.
5. It formulated to resist drying.
6. It will not boil easily when a vacuum is applied over it.
7. It will not freeze because of special formulation. It is optionally formulated not to freeze in freezing weather.

The leak test liquid is brushed or sprayed on a section of welded seam longer than the box. The box is immediately placed over this section of seam and suction is applied. Leaks in the weld will quickly be shown by bubbles and foam. In case of doubt, the box is removed, the film solution is applied again and the test is repeated.

Verification of Bubble Forming Ability of Leak Detector Solutions with Artificial Leaks

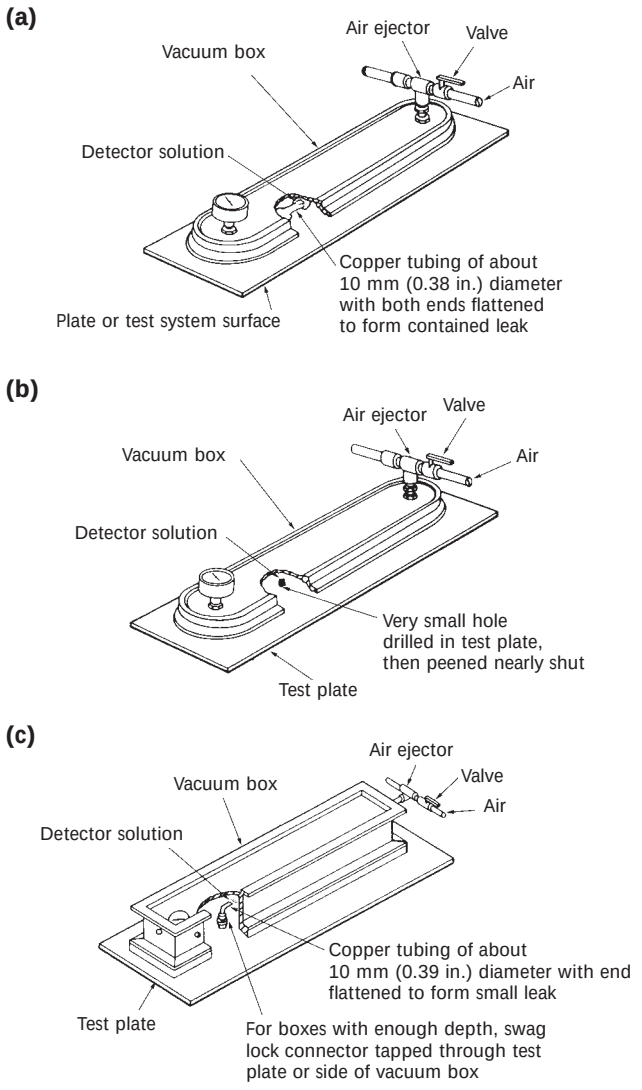
Some leak testing specifications require that the bubble forming capability of a bubble test leak detector solution be verified against a known path leak before and periodically during a test. Of the numerous ways this can be accomplished, the following are several suggested techniques for using a vacuum box for checking the bubble forming capability of a solution.

The first arrangement, shown in Fig. 20a, uses an artificial leak containing its own air supply. It is simply a piece of copper tubing pinched flat and bent slightly on each end. When it is laid on a plate, detector solution can be pooled at each end over the slit and a pressure differential can be created with a vacuum box seated over the tubing. The pinched tube will continue to emit bubbles until the pressure inside the tubing equalizes with the lower pressure in the box. By the time the artificial leak is needed again, there has been enough time for it to refill with air.

The second suggested arrangement is shown in Fig. 20b. The artificial leak is formed by drilling and peening nearly shut a very small hole in a plate slightly larger than an available vacuum box. Applying the leak detector solution over the peened hole and using a vacuum box to create a pressure differential will reveal the bubble forming capabilities of the solution.

A third (similar) arrangement is shown in Fig. 20c. Its artificial leak consists of a

FIGURE 20. Alternative methods for using artificial leaks in vacuum boxes to verify bubble forming capabilities of bubble leak detection solutions: (a) copper tubing leak for use with deep or shallow box; (b) drilled and peened hole leak, for use with deep or shallow box; (c) flattened copper tubing leak, for use with deep box.



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piece of tubing flattened on one end and inserted into a compression type fitting threaded either into the side of a metal vacuum box or into a plate slightly larger than a deeper vacuum box. Again, the solution is applied to the flattened end of the tubing and the capabilities of the solution are revealed when the pressure differential is created by evacuating the box.

Other techniques that might be used are direct pressurization of a piece of tubing flattened on one end or direct pressurization to force air or gas through a micrometer needle valve.

When difficulty is encountered, it must be remembered that leaks can close up. If a solution does not bubble, it is advisable to check to be sure that the leak is still open before drawing a final conclusion concerning the performance of the bubble testing solution.

Visual Examination and Interpretation of Bubble Indications

When performing the visual examination during vacuum box bubble testing, access to the area to be viewed should permit placing the eye within 0.60 m (24 in.) of the surface to be examined, at an angle of no less than 30 degrees with the surface to be examined. Natural or artificial lighting may be used to illuminate the area to be examined. The minimum intensity of lighting in the area to be examined should be 0.50 to 1.10 klx (50 to 110 ftc).

All indications of bubbles should be evaluated in terms of the applicable acceptance standards. If no bubble or foam indication of leakage is observed, the component is considered acceptable without further bubble testing. In most cases, the area under test is acceptable when no continuous bubbling is observed. As bubbles are observed, the position of bubble formation should be marked on the surface of the test object or on applicable drawings, to permit precise location of leaks to be repaired. The component can then be depressurized, if necessary, and the leak repaired as required. After repairs have been made the repaired area or areas should be retested in accordance with the same leak testing procedures.

Personnel performing leak tests should be qualified to levels of competence comparable to those outlined in ASNT *Recommended Practice No. SNT-TC-1A*, in *ANSI/ASNT CP189: Standard for Qualification and Certification of Nondestructive Testing Personnel* or in other applicable guides.

Practical Procedures for Vacuum Box Bubble Testing in the Field

The following additional practices are recommended for field applications of vacuum box solution film bubble tests for leaks.

1. The vacuum box valve and gage should be checked for workability before starting the solution film bubble test. The sealing gasket and transparent window should be checked for deterioration and cracks. The vacuum box should be tested in advance to ensure that it can seal and maintain a differential pressure of some value greater than the differential pressure required or specified for the test in question.
2. The transparent window on the vacuum box should be clean at all times to ensure good visibility of bubble indications by the operator performing the test. A bucket of clean water and clean dry wiping cloths should be kept available for this purpose.
3. If freezing weather exists at the time and location of a test, the weld joints should be heated carefully until the metal is slightly warm to the touch before applying the bubble test fluids and the vacuum box. Such heating will help evaporate any moisture and thaw any ice that could possibly be plugging leaks.
4. As soon as the vacuum box gasket is seated, the valve is opened to the air ejector that draws air out of the vacuum box. To obtain a firm seal at the gasket, hand pressure is applied to the end edges of the vacuum box and finger pressure is applied to the gasket at the welds. (If the box does not have a tight seal or is not firmly seated, air will be drawn into the box and may blow solution film onto the underside of the transparent window. When this happens, time will be lost in cleaning the transparent window or a leak indication may be overlooked by the inspector.)
5. When the vacuum box becomes effective, the operator should observe the test solution film or foam during evacuation. This can help to prevent overlooking indications of large leaks that tend to blow holes through the solution film or foam instead of forming visible bubbles.
6. When performing two-phase vacuum box leak of the same area, the first test should be made at a low differential pressure of, say, 15 to 30 kPa (2 to 4 $lb_f \cdot in.^{-2}$ differential). The minimum

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time for observing the test solution film for bubble indications of leakage is 5 s. For the second test, the vacuum in the box must reach some differential pressure of, say, at least 55 kPa (8 $lb_f \cdot in.^{-2}$ gage) or more with a minimum observation time of about 10 s.

7. Successive positions of the vacuum box (as along a weld seam) are overlapped by at least 50 mm (2.0 in.). This ensures that the areas under the gaskets of the vacuum box in one position are leak tested when the box is moved to an adjacent position.

Typical Requirements for Bubble Test Reports and Test Records

In most cases, copies of test procedures and of test personnel qualifications and examination results are maintained in permanent files by the responsible contractors, constructors or testing organization.

Each vacuum box bubble testing report should contain the following information as a minimum: (1) test date, (2) operator name, (3) test equipment description, (4) test pressure, (5) test results and (6) sketch showing leak locations.

A copy of the qualified procedure should be readily available to nondestructive testing personnel performing leak testing. The test report should be maintained in accordance with requirements of applicable codes and procedure specifications.

Example of Procedure Specification Requirements for Vacuum Box Leak Testing

The American Society of Mechanical Engineers' *Boiler and Pressure Vessel Code* is typical in requiring that the vacuum box leak testing procedure be documented. Each fabricator or constructor must certify that the required written examination procedure is in accordance with applicable specification requirements.

The required written procedure should record, in detail, at least the following leak testing information: (1) size of vacuum box; (2) type of gasket material; (3) maximum length of weld examined in each test; (4) brand name and specific type (number or letter designation, if available) of bubble solution; (5) details of the technique of preexamination cleaning and of drying; (6) details of the technique of bubble solution application, including the length of time that the solution remains on the surface before examination, plus the temperature of the surface during the examination if not within the 4 to 40 ˚C (40 to 105 ˚F) range; and (7) technique of postexamination cleaning, if performed.

Requalification of the leak testing procedure is required in the following circumstances: (1) when any prior processing that may affect the bubble solution examination is changed, including processes that may close any discontinuities or leave interfering deposits; (2) when a change or substitution is made in the type of precleaning material or techniques; and (3) when a change or substitution is made in the type of bubble solution material.

Record copies of procedure and personnel qualifications and examination results should be maintained in accordance with the requirements of applicable codes, specifications or manufacturing and regulatory organizations.

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PART 5. Procedures and Applications of Bubble Testing in Industry

Range of Applications of Bubble Testing

Bubble testing for leak location is probably one of the most widely used nondestructive tests because its simplicity permits its use by workers with minimal training (as in soap bubble testing for leaks in gas lines in the home or tests of inflated tires or inner tubes in the automobile service station). Because of its low cost and quick results, the bubble test is also widely used on consumer products where other tests are not feasible because of their equipment cost or the need for interpretation of test signals whose source and significance are not immediately obvious.

Even for highly trained nondestructive testing personnel whose experience has not included leak testing, the extent of bubble testing in industry and in heavy construction may come as a surprise. The examples of applications and of their procedures suggest the diversity of bubble testing.

Applications of Bubble Testing in Fabrication of Structural Components

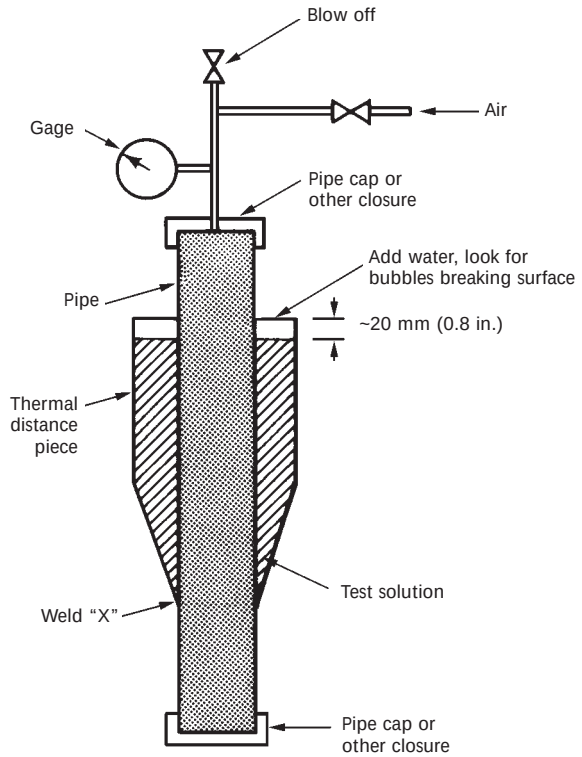
The bubble test may be used to test vessels of any size or configuration that can withstand internal pressure and to which access is possible. It is used to test nonevacuated cryogenic storage vessels that normally have allowable leakage rates that do not economically warrant a more sensitive test. If it can detect the minimum allowable total leakage rate, the bubble test may be used as a final test. Alternatively, the bubble test may be used as a preliminary test before performing a more sensitive leak test, such as a helium mass spectrometer leak test. In this case, the bubble test is used to find and eliminate detectable leakage that (if not corrected) could hinder or slow down the more sensitive type of leak test. For example, the bubble test is generally used as a preliminary test on the inner vessel of double walled evacuated cryogenic vessels, by techniques described next.

Arrangements for Pressure Technique Solution Film Leak Testing

Arrangements for sealing, pressurization and application of films of bubble testing liquid are sketched in Figs. 21 to 26. Figure 21 shows an arrangement for bubble testing of thermal distance pieces for double wall, low temperature and nonevacuated cryogenic vessels. Setups for solution film bubble tests of welded joints in reinforcing pad plates are shown in Figs. 22 to 24. Connections for leak testing of sumps for flat bottom vessels are shown in Fig. 25.

The solution film bubble tests are conducted on the thermal distance piece before it is installed in a vessel. The bubble test of reinforcing pad plates can be made at any time after the nozzle is welded in place but before the hydrostatic

FIGURE 21. Arrangement for bubble testing of thermal distance piece for double wall vessel.



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pneumatic tests or hydrostatic tests. Tests on a sump are made before the bottom is laid, regardless of any previous test in the shop.

Figure 26 shows the test arrangement for bubble testing of an annulus pipeline for nonevacuated double wall vessels. The leak test is made after the pipeline is welded in place but before the hydrostatic or hydropneumatic test. Pressure technique bubble tests are also made on vessels that will withstand internal pressure in accordance with applicable specifications or contracts.

Solution Film Bubble Testing of Entire Pressure Vessels

During erection and before performing a solution film bubble test of an entire vessel, it is desirable to conduct preliminary bubble tests for various types of vessel fittings. Leaks in fittings and reinforcing plates might possibly be detected when the entire vessel is under test. However, by eliminating these leaks beforehand, it is more likely that a test of the entire vessel will be successful the first time it is made.

The procedure for leak testing an entire vessel by the solution film bubble technique typically includes the following steps.

1. Before bubble testing, clean all vessel areas to be tested and make them free of weld slag and other contaminants.
2. Pressurize the vessel in accordance with test specifications and procedures (usually to design pressure).
3. Apply the test solution to the vessel areas designated in the test instructions, in a thin, continuous, bubblefree film.
4. In event of indicated leakage, mark the area or areas of the leak or leaks and repair them after the bubble test of the entire vessel.

The film should be observed as applied, for large leaks will tend to blow the

FIGURE 22. Arrangement for bubble testing of reinforcing plate for thermal distance piece of double wall cryogenic vessel.

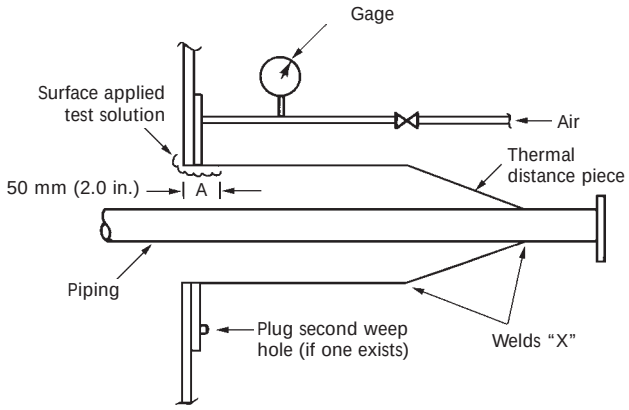


FIGURE 23. Arrangement for bubble testing of welded reinforcement plate for thin wall vessel fittings.

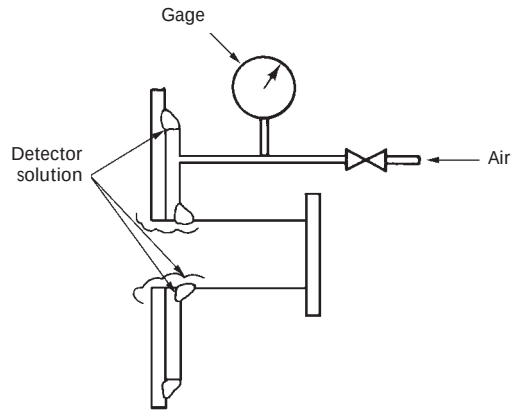


FIGURE 24. Location of inspection areas of reinforcement plate for thin wall vessel fittings.

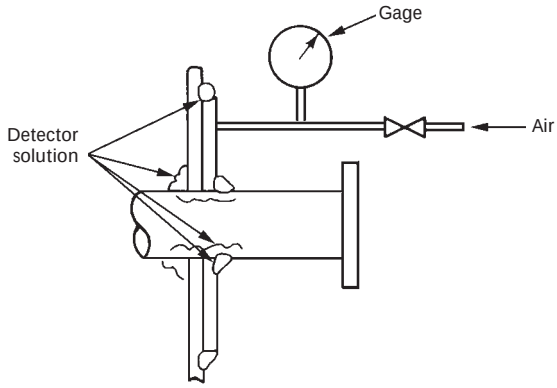
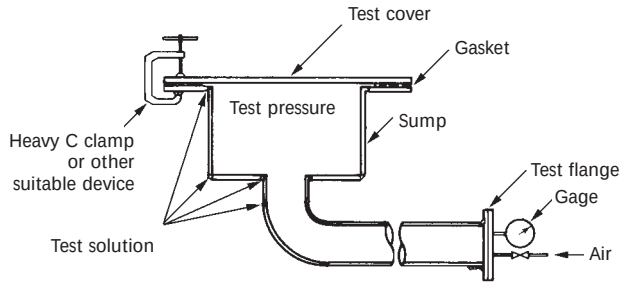


FIGURE 25. Arrangement for closing open ends, pressurizing and application points for bubble test fluid or welded tank sump assembly.



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solution film free rather than forming easily discernible bubbles. The solution film should be checked visually for bubbles for at least 15 s after completion of the application of the solution. It is essential to cover the weep holes of the reinforcing plates with a film of test solution. In the case of two weep holes in a single reinforcing plate, be certain to cover both weep holes simultaneously with the test fluid. This should be done even if the reinforcing plates were tested beforehand.

In the event of inclement weather, at the discretion of the test conductor, the test may have to be postponed or portable shields may be used over and around the areas being tested.

Arrangements for Vacuum Box Technique Solution Film Leak Testing

Arrangements for solution film bubble testing by the vacuum box technique are shown for various test configurations in Figs. 27 through 30.

Figure 27 shows two vacuum box location setups for tests of annulus piping for nonevacuated double wall vessels. The bubble tests are made on an annulus pipeline after it is welded in place but before the hydrostatic or hydropneumatic test. Figure 28 shows the arrangement of vacuum box and air ejector for tests on

FIGURE 26. Arrangement for testing bottom structures of double wall vessel structure.

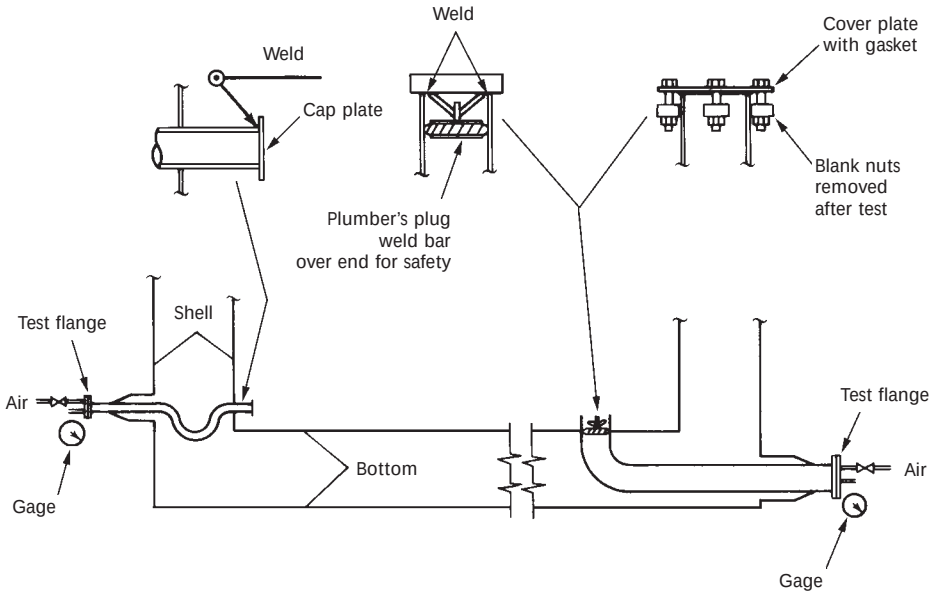

FIGURE 27. Arrangement for vacuum box bubble testing of annulus piping of double wall, flat bottom vessel.

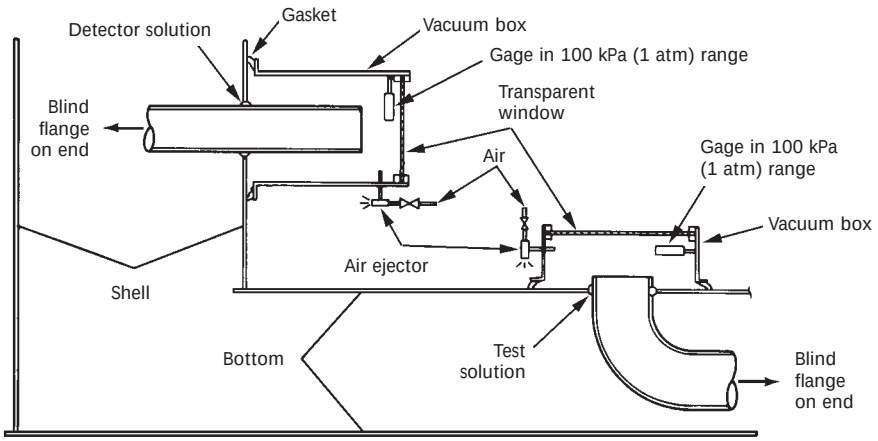

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FIGURE 28. Arrangement for vacuum box bubble testing of sump in flat bottom vessel.

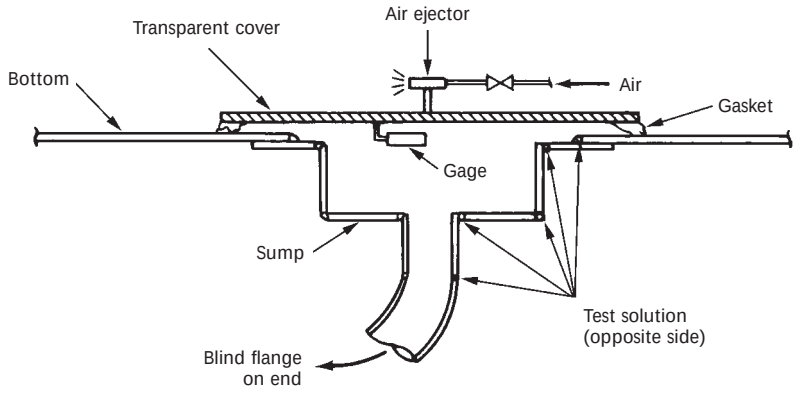


FIGURE 29. Arrangement for vacuum box bubble testing of bottoms, corner welds and anchor straps of flat bottom vessels.

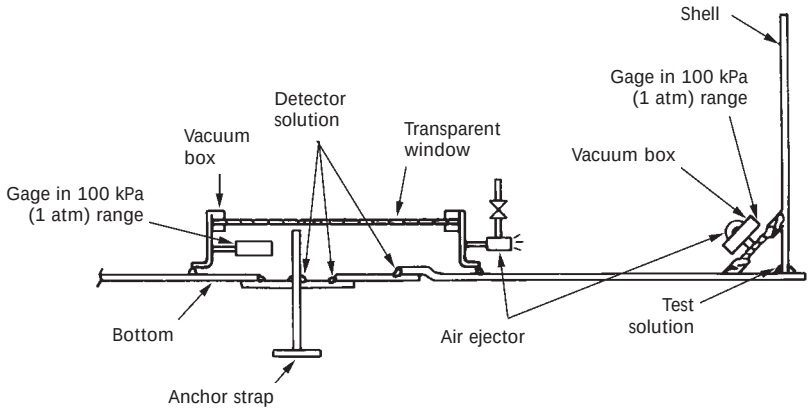
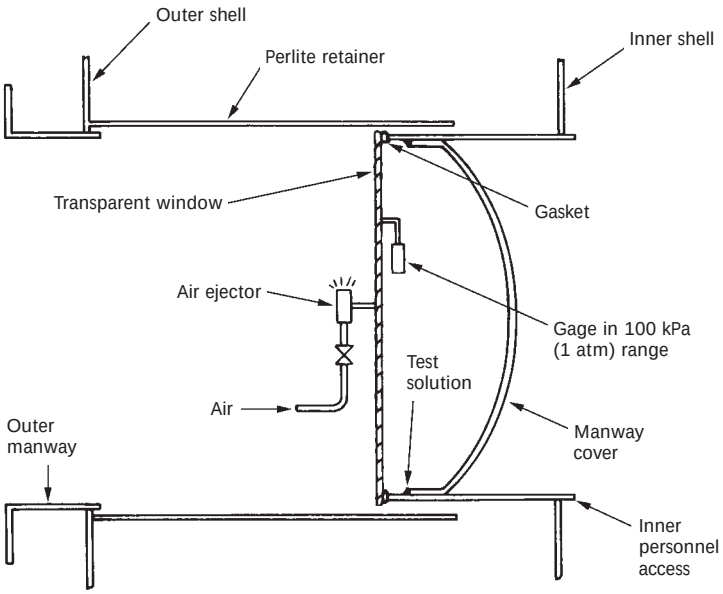


FIGURE 30. Arrangement for vacuum box bubble testing of welds in personnel access areas of double wall vessel.



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sumps for flat bottom vessels. Here, tests are made on the sump after it is welded to the vessel bottom but before the hydrostatic or hydropneumatic pressure tests.

Figure 29 shows arrangements for vacuum box bubble testing of bottoms, corner welds and anchor straps of flat bottom vessels. Tests are made on all anchor strap stubs before butt welding the anchor strap and installing insulation. Vacuum box bubble tests are made on all flat bottom seams welds, damaged areas and corner welds both before and after the hydrostatic or hydropneumatic tests. Similar tests are made on welded inner vessel personnel access passageways for nonevacuated double wall vessels, after they are welded in place and following the hydrostatic or hydropneumatic tests.

Application of Bubble Tests to Nitrogen Pressurized Telephone Cable

Telephone utility companies have reported solution film bubble tests to inspect for damage in telephone cables. For this test, nitrogen gas is injected within the cable sheath under suitable pressure. The bubble testing solution is applied to the exterior surface of the cable. Holes in the sheath of the telephone cable are detected when the gas leaks out and forms bubble indications.

Because the cables are typically carried overhead on poles, access is limited. Cable maintenance personnel can climb the poles and make an inspection for as far as they can reach from the pole. To do this, they carry the test liquid in a bucket and apply it with a special brush to spread the solution over the cable's exterior surface. However, such an inspection is time consuming and difficult. Often, very small gas leaks in the telephone cable sheath are difficult to locate because a certain amount of bubble formation would appear on the cable with each stroke of the brush.

A testing solution is available that forms bubbles large enough to be seen from the ground and that does not give false alarms by bubbling up where no leaks exist. To apply the liquid along the cable span between poles, a roller type wheel trolley system carries a tank of detection liquid and two sprays that apply the liquid to the cable sheath. The cable test unit is pulled along by a man on the ground, by use of sections of the pole from a tree trimmer. This system has greatly decreased the time required for cable inspection for leaks.

The spray technique is intended to be used to locate the leaks at approximate locations indicated by pressure gradients. It provides the advantage that rough gradients, instead of fine gradients, can be used to indicate the general location of the anomaly. When the general location is established, the trouble is found by spraying one or more spans of cable in which the leak is indicated. The entire length of cable that the pressure gradients indicate required spraying should be covered to avoid the possibility of leaving relatively large, pressure lowering leaks.

Precise Locating of Leaks in Areas of High Cable Pressure Gradients

The purpose of the spray technique, as used on cables maintained under continuous feed pressure, is primarily to find the leaks indicated by gradients. Because these cables are continuously under pressure, no supplementary cylinders of pressurized gas are generally required. Cable pressure ranging from 7 to 35 kPa (1 to 5 $lb_f \cdot in.^{-2}$) are generally suitable for leak location work by the spray technique. At pressures in excess of 35 kPa (5 $lb_f \cdot in.^{-2}$), the jet action of the gas vented through the hole in the sheath is generally too rapid for the formation of visible bubbles. However, higher pressures are recommended during hot weather, when compressive stresses develop in the sheath and tend to close the cracks and retard the escape of gas.

Preparation of Test Solutions for Cable Leak Tests

Preparations for spraying telephone cables should include an adequate supply of clean water free of excessive amounts of sulfur or calcium. The water and the leak test concentrate are thoroughly mixed in the spray tank in the recommended proportions. Then the top is placed on the tank and locked securely. The tank is charged with compressed air at 125 to 170 kPa (18 to 25 $lb_f \cdot in.^{-2}$ gage) pressure for warm weather solution, either by means of the hand pump or with a nitrogen cylinder. The cold weather solution is sprayed at pressures of 200 to 240 kPa (30 to 35 $lb_f \cdot in.^{-2}$).

Spraying Leak Testing Solution onto Telephone Cables

After selecting the proper spray tips, the nozzles are positioned so that their sprays cross slightly and completely envelop the top 180 degrees of the cable sheath. The lower half of the cable will be wet by the solution flowing down both sides of the cable to the drop off point at the bottom. It is characteristic of this solution to cling to the sheath in this manner. This

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technique should not be used where the test solution will drip on polyethylene sheath cables.

When using the hand sprayer, the tool is held in a position so the fan shaped spray adequately covers the surface being inspected. The hand sprayer is intended for spraying vertical runs of cable and horizontal runs from a closeup position, as from a ladder platform truck. It may also be used in manholes when considerable spraying is required. A flash leak tester and a squeeze bottle are intended for use in manholes and other restricted areas where a small amount of spray is adequate.

Observing Bubbles Formed While Scanning Overhead Cables

When fluid starts spraying from the nozzles, the sprayer is propelled along the strand. The forward speed is governed by the rate at which full wetting of the cable sheath is secured, as observed from the ground. Leaks will be indicated by clusters of bubbles. Observe for large leaks continuously as the sprayer moves forward. Large leaks must be spotted as the spray hits them, because there is a tendency for clusters of bubbles to blow away if the hole is large or if the internal pressure is high. After spraying about 7 m (20 ft) of cable, observe that portion for bubbles caused by medium and small leaks. When a telephone pole is reached, spray as close to it as possible, then vertically raise roll sprayer from strand or cable and transfer it around the pole. Spray cable as the transfer is made or, where this is not feasible, use the hand held sprayers.

During windy weather, it is important to observe continuously for evidence of breaks in the sheath, as the wind tends to blow the bubbles away as soon as they are formed. Wind also tends to form bubbles not associated with sheath breaks, particularly on lashed cable. This pattern is soon recognized.

If a leak is indicated at or near a branch cable, inspect at least one span of the branch cable. If the leak indicated by the initial gradient is not found and there is reason to believe that pressures in the general area are too low, refer to the rough gradient section for reestablishing leak location. If the foregoing operations do not reveal the leak and the gradient indicates a precise location, it should be inspected at close range. The hole in the sheath may be large enough to prevent its location from the ground by the spray technique.

Precautions in Bubble Testing of Overhead Telephone Cables

During cable tests, it is important to observe all general safety precautions applicable to overhead lines. The spray technique using some cable leak test concentrates should not be used on polyethylene sheath cables, as it is damaging to the sheath. If the concentrate solution accidentally contacts the polyethylene sheath, it should be washed off with clear water. In addition, the precautions to be observed while using the hand and roll sprayers are as follows.

1. Before proceeding along the cable, check to see that all components are securely assembled and that the sprayer is firmly seated on the stand.
2. Keep the roll sprayer at a safe distance from power wires at all times.
3. Obtain assistance when inspecting cable at street and railroad crossings.
4. Never use tank pressure in excess of that specified by the manufacturer.
5. Exercise care to avoid getting any spray on the public, even though it is not injurious to the skin or fabrics of any type.
6. If large drops of solution fall on automobile surfaces, flush them off with water, as its detergent action will give the false impression of color fading, particularly on dusty surfaces.
7. Avoid using water having a high calcium and/or sulfur content and never one with a silt content.
8. Always use a clean container for carrying or obtaining water.
9. Results will be below average on cables adjacent to railroads on which diesel engines are used, as the greasy residue of oil combustion tends to prevent adequate adhesion of the solution to the cable.

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8

CHAPTER

Techniques and Applications of Helium Mass Spectrometry

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PART 1. Principles of Mass Spectrometer Leak Testing with Helium Tracer Gas

Basic Techniques for Leak Detection with Helium Tracer Gas

All techniques of leak detection using a mass spectrometer leak detector involve the passage of a tracer gas through a presumed leak from one side to the other side of a pressure boundary and subsequent detection of the tracer gas on this lower pressure side. Figures 1 to 5 show some typical basic setups for leak testing with helium tracer gases. For each practical application, there is usually one helium leak testing technique that gives optimum results.

Factors to be considered when selecting helium leak test techniques include the following: (1) size, shape and location of equipment to be tested, (2) choice between pressure or vacuum or both for testing, (3) maximum leakage rate specified or that can be tolerated, (4) degree of automatic leak testing operation required, (5) number of parts or complexity of the system to be tested and (6) choice of conventional or counterflow leak detector.

Basic techniques for helium leak testing include the following.

1. In the helium tracer probe technique (Fig. 1), the mass spectrometer leak detector is connected to the internal volume of an evacuated test object (such as a vessel or piping system) while a helium spray tracer probe is moved over the external surface to detect the specific locations of leaks.
2. In the helium detector probe technique (Fig. 2), the test object or system is pressurized internally with helium or a gas mixture containing helium. The mass spectrometer leak detector is connected to the hose of a scanning probe that collects samples of gas leaking from the external surface into the surrounding atmosphere. To verify probe response before scanning the test object, the probe should be moved past the orifice of a known helium source at the same speed and distance as will be used for the test object. The detector probe technique can be used to determine leak locations but is inadequate for leakage measurement or for finding leaks smaller than 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}).
3. When vacuum leak testing by the hood technique (Fig. 3), the mass spectrometer leak detector is connected to the evacuated interior of the system under test. The test object or system is then placed under a hood or within a chamber containing helium gas or an air helium mixture usually at atmospheric pressure. This technique can be used to quantify the total leakage rate of the system. However, it cannot be used to determine the specific locations of leaks.
4. In the bell jar test technique (Fig. 4), sealed components filled with helium or a gas mixture containing helium are placed in an evacuated testing

FIGURE 1. Helium leak testing of evacuated vessel or system with tracer probe.

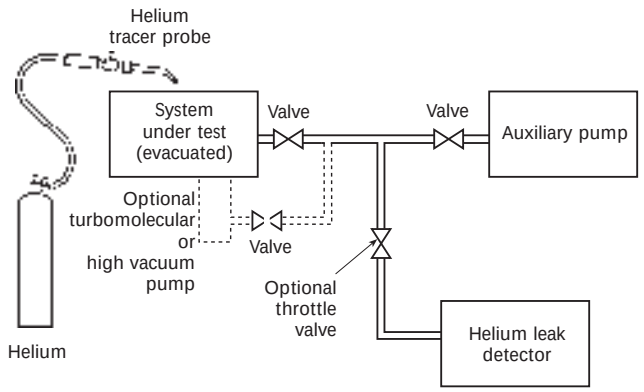
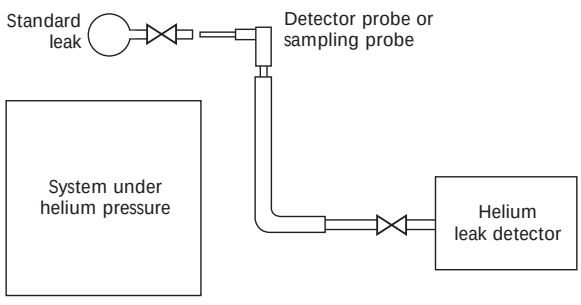


FIGURE 2. Helium leak testing of pressurized vessel or system with detector probe.



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chamber. The mass spectrometer connected to this vacuum chamber detects helium leaking from any part of the surfaces of the sealed test objects in the vacuum chamber. This test does not permit location of leaks on the test object surfaces.

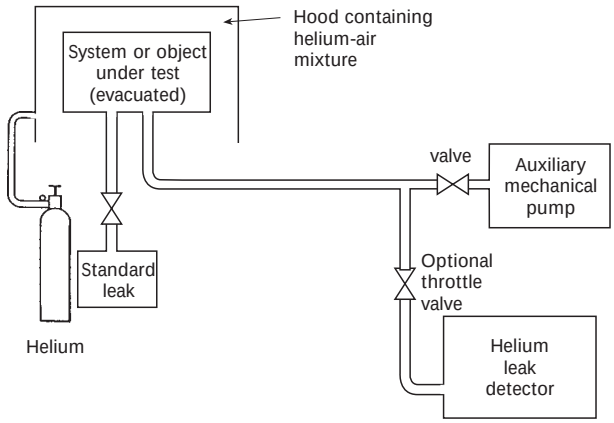
5. In leak testing large evacuated systems, the accumulation technique is used to increase sensitivity beyond that which can be obtained by dynamic testing. This technique is also used on production line testing of evacuated components passing through a helium hood for a period of time before sampling by a helium mass spectrometer leak detector. In the accumulation technique of leak testing for pressurized objects (Fig. 5), leaking helium tracer gas is allowed to collect for a period of time before being sampled by the leak detector. This technique, also used in $\mu L \cdot L^{-1}$ testing, can be adapted to several different leak testing situations, as described elsewhere. The accumulation technique does not usually permit leak location.

Vacuum System Limitations of Helium Leak Detectors

The vacuum system of the mass spectrometer helium leak detector usually consists of mechanical roughing pump, mechanical backing pump or forepump, oil vapor diffusion pump or turbomolecular pump, cryogenic pumping surface (cold trap, for conventional leak detectors) and associated valves and gages.

The effect of this associated high vacuum system on overall performance of the helium leak detector can never be overlooked. It directly influences such leak testing parameters as (1) spurious background helium signal; (2) minimum detectable leakage rate; (3) response time; (4) throughput, which determines the leak detector's ability to test large or gassy pieces or to *back* another vacuum system's diffusion pump; and (5) downtime due to mass spectrometer contamination or filament burnout.

FIGURE 3. Hood technique of leak testing of evacuated components inserted into hood or envelope containing a helium atmosphere.



Causes of Spurious Background Signals in Helium Leak Testing

In helium leak testing, spurious background signals may arise from sources

FIGURE 4. Leak testing of sealed components internally pressurized with helium tracer gas and enclosed in a bell jar.

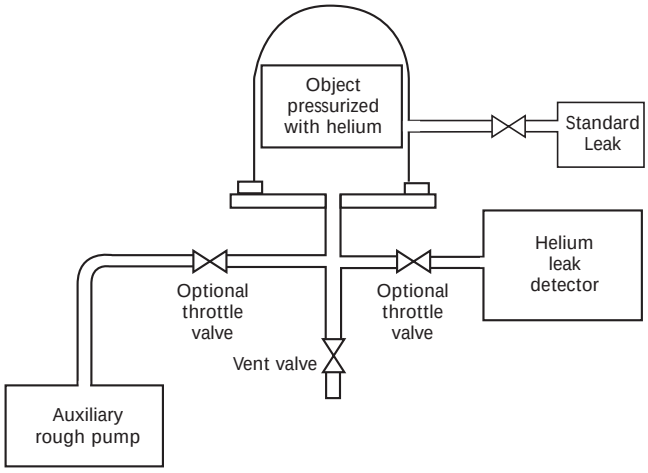
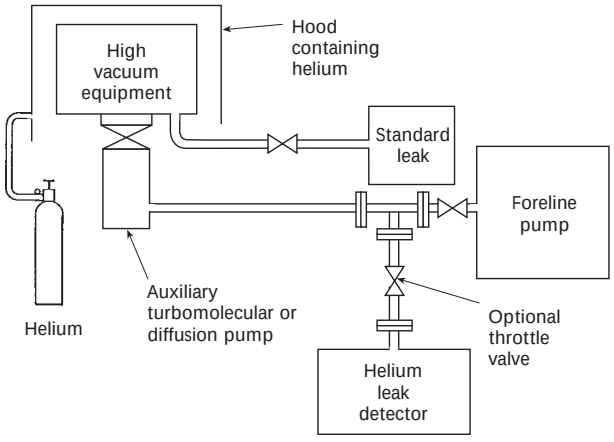


FIGURE 5. Proper connection of helium mass spectrometer between high vacuum pump and foreline pump for leak testing of diffusion equipment and large vacuum systems at pressures below the 10 mPa (0.1 mtorr) optimum operating pressure of helium mass spectrometer.



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such as (1) helium contamination of the atmosphere surrounding the test object; (2) ion scattering due to gas pressure too high in the mass spectrometer tube; (3) hydrogen and hydrocarbon contamination of the mass spectrometer tube; and (4) elastomeric gaskets, greases, rubber hose, painted surfaces and castings, which, when exposed to high concentrations of helium, tend to soak up helium and later become sources of helium outgassing.

These sources of background tend to reduce the ability of the helium leak detector instrument to find very small real leaks.

Effects of Atmospheric Helium Leakage into Mass Spectrometer System

Too high a pressure in the mass spectrometer due to an atmospheric leak can give rise to a helium background signal. Atmospheric air contains about 1 part helium in 200 000 parts of air. The deflection on the leakage rate meter due to atmospheric helium may be 10 to 100 times larger than the minimum detectable helium leakage signal. This is one of the basic limitations of the helium detector probe technique.

If the leak test uses argon tracer gas, the situation is even more serious because the normal argon concentration in air is about one percent.

Gas Handling Capacity of Mass Spectrometer Vacuum System

The throughput of a vacuum system is a measure of the mass flow of gas being handled. This is given by the product of the total pressure and the effective volumetric pumping speed (at that pressure). Therefore, instrument throughput is increased by operating with a high pressure in the leak detector sensing element and with a high pump speed. However, the maximum sensing element pressure permitted because of mass spectrometer limitations is usually 25 to 40 mPa (0.2 to 0.3 mtorr). This limits the throughput capability of a leak detector to about 6×10^{-4} Pa·m^3·s^{-1} (6×10^{-3} std cm^3·s^{-1}). If the leak detector diffusion pump is throttled as in accumulation testing, the gas handling capacity will decrease. The quantity of gas that must be pumped per unit time to maintain a desired vacuum system pressure is known as the gas load. If the gas load of the item under test is larger than the throughput of the leak detector, the instrument must be throttled and/or some of the gas load must be bypassed to a auxiliary pump system (see Fig. 3). This results in a loss of leak testing sensitivity because some of the tracer gas is also bypassed. This is frequently necessary when using a conventional (noncounterflow) leak detector.

Measuring Flow Rate of Helium with Leak Detector

The effective pumping speed of the diffusion pump of the conventional leak detector can be held constant by not adjusting the gas flow path (by not adjusting any valves in the system). The pumping speed of the diffusion pump is the volume flow rate. The output signal indicates the partial pressure of helium in the sensing element. The product of the two equals the *helium mass flow rate* or throughput:

$$Q_{\mathrm{He}} = P_{\mathrm{He}} S \quad (1)$$

where Q_{HE} is helium flow rate (Pa·m^3·s^{-1}); P_{HE} is partial pressure of helium (pascal); and S is pumping speed of helium (m^3·s^{-1}). For quantitative measurements, the instrument can be calibrated by admitting a known rate of helium flow into the instrument. It is not necessary to know the pumping speed, but it must be held constant during calibration and test.

Mass Spectrometer Detection of Helium, Neon and Argon Tracer Gases

Occasions have arisen where it is necessary to use a tracer gas other than helium to locate leaks. Because the helium leak detector is a mass spectrometer, it is possible to construct leak detectors for other tracer gases. Argon and neon, for example, are being used as tracer gases with modified leak detectors. These leak detectors can detect helium, neon and argon by merely turning a switch to select the tracer gas that is to be detected. The presence of other gases, even the other tracer gases, will normally have no effect on the sensitivity or detection of the specific tracer gas the instrument is tuned to detect. (For clarity, this discussion treats only tests that use helium as the tracer gas.)

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Protective Devices Used with Mass Spectrometer Leak Detector

In conventional helium leak detectors, a liquid nitrogen cold trap is used to trap out condensables such as water vapor entering the system. Because diffusion and mechanical pumps remove condensables quite slowly, a cold trap is necessary for rapid cleanup of the leak detector in applications where the rapidity of the test is important. The cold trap is also a protective device for the leak detector. Trapping of condensable vapors keeps them from contaminating the vacuum system and sensing element. It also freezes or traps out oil vapor that backstreams from the diffusion pumps or from surging, which occurs whenever a pressure burst is admitted to the leak detector. A second cold trap externally mounted on the inlet of the leak detector can serve as an additional protective device, especially when large, dirty systems are being vacuum tested.

Another protective aid used with mass spectrometer leak detectors is the automatic protection valve, which separates the test object from the leak detector. This valve remains open as long as the pressure in the leak detector is at a safe, low level. If for some reason the test object admits too high a pressure to the leak detector, this valve will close automatically, protecting the leak detector from high, sudden and long pressure rises that often result in filament failure.

Converting Helium Leakage to Air Leakage Rate

Based on kinetic theory, when converting helium leakage rates to rates of leakage for other gases, it would be useful to know the type of leak that exists. Theoretically, helium flow through small leaks, in the range of 1×10^{-7} Pa·m^3·s^{-1} (1×10^{-6} std cm^3·s^{-1}) or less, is 2.73 times the air leakage rate. However, leaks large enough to be governed by viscosity will permit air flow as much as 1.4 times greater than the flow of helium.

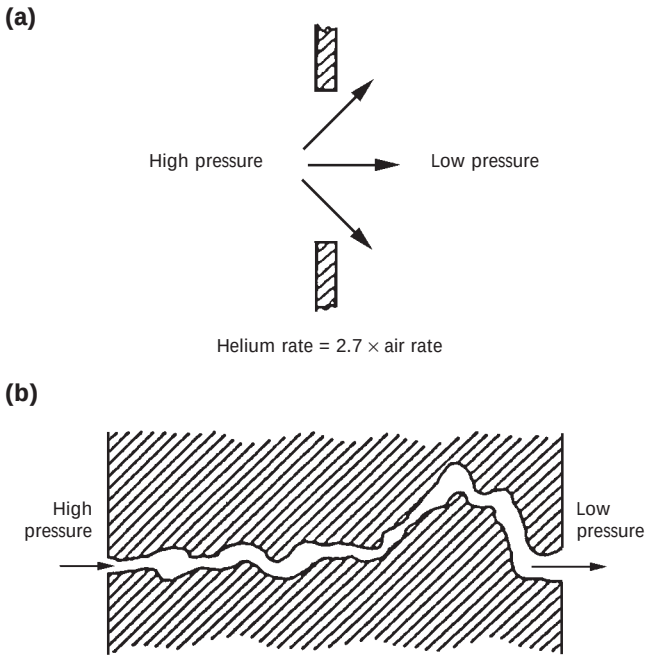
The kinetic theory of gases predicts that the flow of one gas relative to another through an ideal leak under molecular flow conditions (roughly below one millionth of atmospheric pressure) will be inversely proportional to the square root of the average molecular weight. Because the molecular weight of air is about 29 and helium is 4, it calculates that helium will flow 2.7 times as fast as air through this leak (see Fig. 6a). The implication is that the minimum detectable leak in terms of air is 1/2.7 of that for helium.

Actually, the *ideal* leak referred to in kinetic theory is a circular opening in a wall, whose diameter is at least 10 times the wall thickness. In the real world most leaks are tortuous, sometimes multiple paths much longer than the cross section; more like irregular wormholes (see Fig. 6b). Air leakage rates can vary from many times smaller up to almost equal to the helium rates. One can say only that a leak rate measured in helium is generally conservative.

Partial Pressure Measurement Factors Used with the Helium Leak Detector

The mass spectrometer leak detector measures the partial pressure of a tracer gas, usually helium. The composition of dry air at sea level is given in the chapter on tracer gases. The normal percentage of helium in the atmosphere is about 5 μL·L^{-1}. Because the average total pressure is 101.325 kPa (760 torr), the partial pressure of helium in the atmosphere is about $(5 \times 10^{-6}) \times 100 =$ 0.5 Pa or about 0.004 torr. In the average mass spectrometer leak detector, the total

FIGURE 6. Theoretical versus observed differences between flow rates of helium and air: (a) kinetic theory of molecular flow through hole whose diameter ≥ 10× length; (b) tortuous path whose length is greater than cross section, as in most leaks. Helium rate may be equal to air rate for large leak or many times larger for small leak.



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pressure in the helium sensor is reduced with vacuum pumps to less than 0.01 $\mu L \cdot L^{-1}$ of atmospheric pressure. This reduces the partial pressure in the helium sensor to $1 \times 10^{-8} \times 0.5$ Pa = 5×10^{-9} Pa or about 4×10^{-11} torr. Extremely small changes in helium partial pressures can be detected.

Calibration of the Helium Leak Detector

A standard calibrated helium leak with a reservoir of 100 percent pure helium at slightly higher than atmospheric pressure is generally used to calibrate the helium leak detector. The leaking membrane is a silica quartz bulb or other permeable membrane (such as heat resistant glass) with diffusion leakage. Calibrated helium leaks are obtainable in range of 3×10^{-7} to 3×10^{-11} Pa·m^3·s^{-1} (3×10^{-6} to 3×10^{-10} std cm^3·s^{-1} of helium). The calibration temperature is labeled on each standard leak and a temperature correction factor is also given. By using comparison tests with calibrated leaks of known leakage rates, it is possible to determine unknown leakage rates of test objects. In estimating leakage rates from comparison tests, consideration must be given to any factors that deviate from the standard leak, such as gas flows, pressure differentials and mixed test gases.

If calibration at leakage rates greater than 3×10^{-7} Pa·m^3·s^{-1} (3×10^{-6} std cm^3·s^{-1}) is required, a fluorocarbon resin or capillary leak with a reservoir of helium is available.

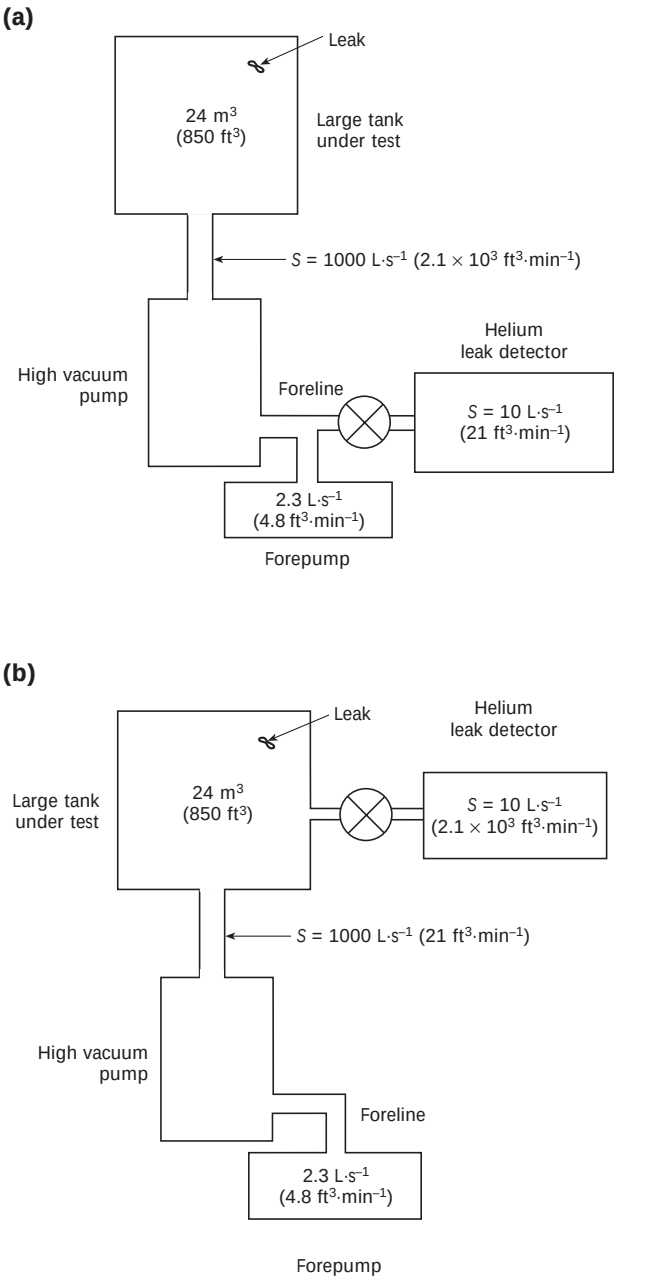
Pumping Arrangements for Leak Tests of Sizable Objects

Often, it is necessary to perform vacuum leak tests of sizable objects such as fuel storage tanks. Two arrangements for testing sizable tanks are shown in Fig. 7. Figure 7a shows the correct connection for vacuum testing for most applications involving leak testing of systems of sizable volumes. In this case, the leak detector is connected into the foreline of the auxiliary diffusion pump. This ensures an adequate flow of sample gas to the leak detector. Under the following test conditions, for example, the response time would be very close to 24 s.

1. The test object volume is 24 m^3 (850 ft^3).
2. The pumping speed of the leak detector is 0.01 m^3·s^{-1} (21 ft^3·min^{-1}).
3. The pumping speed of the system diffusion pump is 1 m^3·s^{-1} (2.1×10^3 ft^3·min^{-1}).
4. The pumping speed of the system mechanical pump is 2.3×10^{-3} m^3·s^{-1} (5 ft^3·min^{-1}).

The leak detector would receive 81 percent of the helium because it shares with the forepump rather than with the diffusion pump. These conditions permit good performance in helium leak detection.

FIGURE 7. Pumping arrangements for vacuum leak testing of large volumes: S = 1000 L·s^{-1} (16 000 gal·min^{-1}): (a) correct connection; (b) incorrect connection.



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Effect of Incorrect Arrangement for Vacuum Leak Testing

Figure 7b shows an incorrect arrangement for vacuum testing. With this arrangement, the response time is based solely on the leak detector diffusion pump speed.

1. With the system diffusion pump valved out, the leak detector will pump 100 percent of the helium. System response will be 24 m^3 at 0.01 $m^3 \cdot s^{-1}$, or 2400 s (40 min).
2. With the system diffusion pump valved in, the leak detector will pump only 1 percent of the helium. Therefore, the mass spectrometer signal will be 100 times smaller.

Because of this incorrect arrangement, either of these conditions would result in (1) excessive time for obtaining leak indications or (2) reduced leak sensitivity for the helium leak detector.

Precautions in Making Vacuum Connections to Test Objects

Poorly designed test connections can be a major source of difficulty in leak testing evacuated test objects and systems. Common sources of trouble are leakage in connections and excessive helium contamination. To avoid these difficulties, the following warnings should be observed:

1. Excessive amounts of plastic and rubbery materials (elastomers), especially rubber tubing, should be avoided, because these materials can absorb helium. Thus, when a large leak is encountered, the material will absorb appreciable quantities of helium that are difficult to remove by pumping. The contaminated material will then give false indications on succeeding tests. Rubber tubing is particularly bad because not only is it subject to helium contamination, but it eventually becomes contaminated with other materials that prevent the attainment of the vacuum required for good leak detection. Some rubber may be necessary to make vacuum connections but its use should be kept to an absolute minimum. However, alternative flexible compounds or metal bellows tubes are available that have less helium absorption. These should be considered where they are feasible.
2. Apply lubricants to gaskets in moderation. A good low vapor pressure vacuum grease will be of considerable aid in making vacuum tight joints. Like rubber, however, excessive vacuum grease causes helium contamination. Also, large quantities of grease will act as a dirt catcher and the system will soon be so dirty that a good vacuum will be unattainable. Only a light film of grease should be applied to the gaskets used and the excess should be wiped off.
3. Commercially available O-rings (molded flexible gaskets having a circular cross section) make very reliable and convenient vacuum seals. About three quarters of the O-ring thickness is recessed in a circular groove. When joints are made, the O-rings are compressed by a fourth of their diameter. Engineering data sheets available from O-ring manufacturers list permeation rates and make recommendations as to the design of the grooves; if these recommendations are followed, good vacuum joints result. As mentioned above, when lubricating O-rings, only a thin lubricant film should be applied.
4. Flat rubber or synthetic gaskets should be avoided whenever it is possible to use O-rings. However, when it is necessary to use flat gaskets, their thickness should be held to a minimum so that the vacuum system is exposed to the smallest possible amount of rubber. Silicone rubber and fluorocarbon resin should be avoided because of their high permeation rates and helium retention.

Operator Precautions in Vacuum Testing

Observing the following precautions will help operators to establish successful helium leak test procedures on vacuum systems:

1. The interior of objects to be leak tested should be as clean as possible. In particular, they should be free of water and greases. These materials evaporate in large volume at reduced pressures. This burdens the pumps at the same time that it dilutes the helium that may enter through a leak.
2. If the system uses a direct flow mass spectrometer leak detector and includes a protection throttle valve or system isolation valve, the system should first be closed whenever any change or adjustment is to be made to the test system. This will prevent accidental admission of air to the leak detector. The throttle valve should never be opened unless the auxiliary pump valve is first opened.

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Variables Influencing Sensitivity of Helium Leak Testing of Evacuated Objects by Tracer Probe, Hood and Accumulation Techniques

In addition to the direct effect of helium mass spectrometer sensitivity, the following factors affect the helium leakage rate test: (1) time duration of the test; (2) volume of the vacuum system; (3) percentage of helium tracer gas constantly surrounding the test boundary; (4) pressure in the vacuum system; (5) pressure in the sensing element of the helium mass spectrometer leak detector; (6) location, length and size of connection between the helium mass spectrometer and the vacuum system; (7) helium background within the vacuum system; (8) cleanliness, surface area, surface finish and material composition of the test boundary exposed to the vacuum system; (9) stability of electrical power supply to the helium mass spectrometer leak detector; (10) temperature of test object; (11) effective pump speed of the vacuum pump system at the test boundary; (12) ratio of the total gas flow (throughput) of the vacuum pump system to the total gas flow (throughput) of the helium mass spectrometer; and (13) percentage of helium tracer gas blanketing the evacuated test boundary.

The relationship of some of these variables is expressed by Eq. 2 for static tests:

$$\textbf{(2)} \quad Q = \frac{xKVP}{tHP_t}$$

where Q = total leakage rate ($Pa \cdot m^3 \cdot s^{-1}$) x = helium leak indicator signal, in scale division; t = elapsed time (second); H = helium concentration surrounding test (mole fraction); P_t = absolute pressure in leak detector sensing element (pascal); K = system calibration factor (pascal per division); P = absolute pressure in evacuated space (pascal); and V = volume of evacuated space (cubic meter).

Test Sequence for Helium Leakage Rate Test

A vacuum system to be leak tested should be cleaned to remove all loose dirt and rust, debris and hydrocarbons such as oil or grease. Direct loss of system sensitivity occurs through loss of mass spectrometer sensitivity caused by organic material contamination. The vacuum system should first be adequately cleaned and then inspected with ultraviolet radiation for fluorescent indications of oil or grease.

Inadequate cleaning causes excessive outgassing load. This results in increases in pumpdown time and, in turn, longer time to achieve required system sensitivity. Pumpdown time, in addition to response time and system sensitivity, is a major factor controlling the selection of an auxiliary vacuum pump system for leak testing by this technique. Pumpdown time is usually the major factor that controls the selection of a permanent vacuum pump system.

The following steps must be observed in the helium leakage rate test.

1. Remove all weld slag, dirt, moisture, rust and hydrocarbons from all areas of the test boundary to be evacuated for the test. This includes removal of liquid penetrant residues and paints such as red lead and zinc chromate.
2. Block all openings in the pressure boundary to be evacuated for the test.
3. Arrange the helium mass spectrometer so that the space on the evacuated side of the boundary being tested can be periodically sampled. To maintain the desired mass spectrometer sensing element pressure, make the mass spectrometer connection as short and as large in diameter as possible. Use a vacuum valve in the connection at the boundary of the evacuated space.
4. Tune the helium mass spectrometer to ensure that instrument sensitivity is at its optimum or peak. Some newer mass spectrometer leak detectors can be adjusted so that the leakage rate display is adjusted to the temperature corrected standard leakage value.
5. Connect a standard leak to the system as far from the mass spectrometer connection as practical. The standard helium leak should have a leakage rate equal to or less than the total allowable leakage rate for the test boundary.
6. Connect the test component and evacuate the test boundary to the pressure specified. The connecting hose has a vacuum conductance C, where $C \propto d^3 \cdot l^{-1}$ where d is hose inside diameter and l is hose length. The smallest partial pressure of helium in the annular space is inversely proportional to C. It is desirable to minimize the influence of the helium background (partial pressure) in the annular space by making C large. The connecting hose should be as short as possible and no narrower than 13 mm (0.5 in.) inside diameter.
7. Open the vacuum valve on older, conventional flow mass spectrometers. (Automatic counterflow or conventional mass spectrometer leak

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detectors will cycle normally into test mode.) Adjust the instrument sensing element pressure to some specific level (usually 10 mPa or 0.1 mtorr) by using either the instrument throttle or accumulator valve. The system pressure and size and length of the helium mass spectrometer connection will determine whether a throttle or accumulation technique is used. The same valve settings must be used when the system is calibrated.

8. To determine system sensitivity, calibrate the test systems with the standard helium leak as follows. Place a balloon inflated with 100 percent helium over the inlet to the capillary tube standard leak, or use a helium permeation standard leak. With the mass spectrometer sampling the evacuated test system, open the vacuum valve to the standard leak. Figure 8 shows an example graph of x/t obtained with a calibrated helium leak with a leak rate of 5.6×10^{-5} Pa·m^3·s^{-1} (5.6×10^{-4} std cm^3·s^{-1}). With this 100 percent helium leak open to the test system and the helium mass spectrometer, the measured slope for this leak indicated an x/t value of 120 scale divisions per hour.
9. Calculate and plot on graph paper the allowable leakage line of slope x/t for the test system. Use the static leakage test equation derived from Eq. 2 in scale division per hour:

$$\frac{x}{t} = \frac{96.6\,QHP_t}{KPV}$$

The number 96.6 is the combined conversion factor for the mixed system of units.

10. If the system calibration has caused excessive helium background in the evacuated system, partially vent the system and to reduce the original pressure to dilute the background to an acceptable level.
11. If the evacuated test boundary is single wall construction, shroud all

FIGURE 8. Example of actual static leak rate test, showing system calibration with system standard leak. The instrument was a direct flow helium mass spectrometer with cold trap. System standard leak rate = 5.6×10^{-5} Pa·m^{3}s^{-1} (5.6×10^{-4} std cm^3·s^{-1}). During system calibration P_t = 30 µtorr.

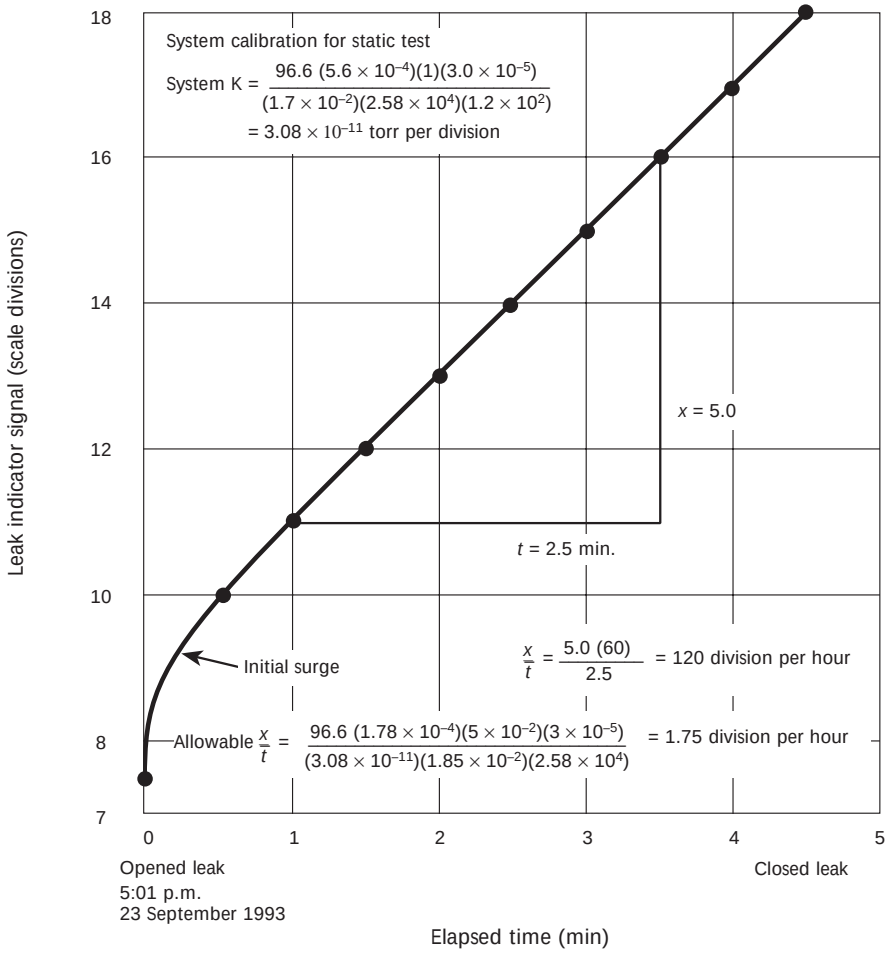

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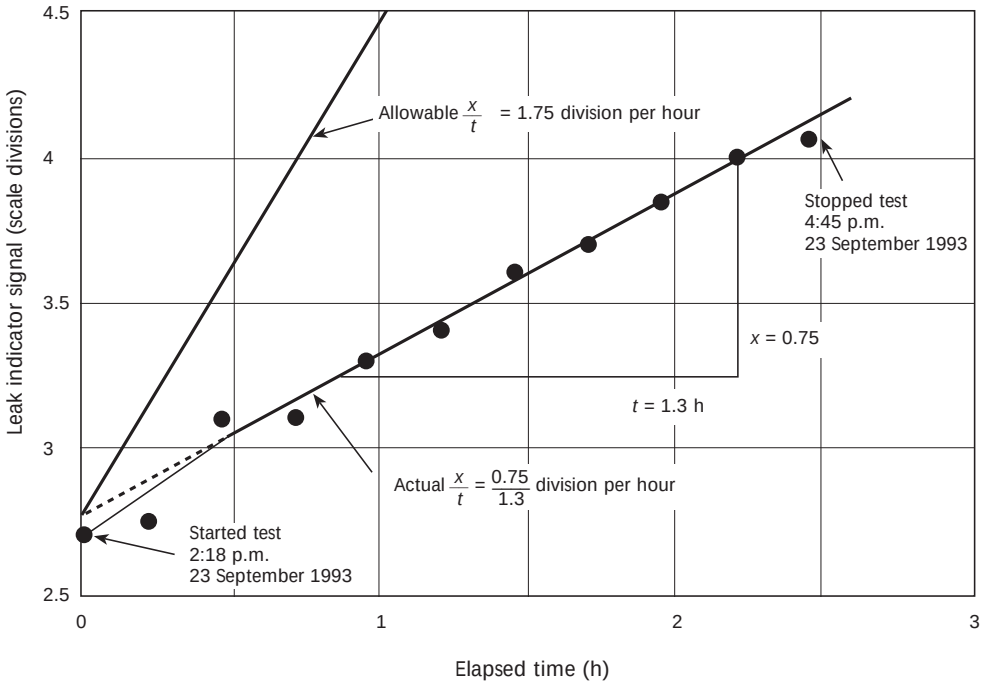
FIGURE 9. Graph of actual static leak rate test. During system calibration, the vacuum system pressure remained constant at 2.27 Pa (17 mtorr). During the test, the vacuum system average pressure was 2.47 Pa (18.5 mtorr).



designated test areas or the entire surface of the test boundary in polyethylene sheeting. However, if the evacuated test boundary is the inner vessel of a double wall vacuum cryogenic vessel to be used for liquefied natural gas, liquid oxygen or liquid nitrogen, the outer vessel will act as a shroud.

12. Inflate the polyethylene bags with helium or pressurize the interstitial space of a double wall cryogenic vessel with the required or specified concentration of helium in air or inert gas such as nitrogen. Note: it is recommended that only a small quality of helium be applied initially with the system being sampled. If there is no noticeable increases in signal within a short time, then continue injecting the rest of the helium. This approach can prevent wasting large quantities of costly helium in the event that large excessive leakage was overlooked in earlier stages of testing.
13. Periodically, sample the evacuated system at regular intervals with the helium leak detector. Record and plot leakage signal magnitude x as a function of testing time t on graph paper at these intervals until the slope of this line is established (see Figs. 8 and 9). If the pressure in the evacuated system increases during the test due to a temperature rise, use an average value of P. If the slope of the plotted line of test data is less than the allowable leakage rate x/t determined in Step 10, the leakage rate of the test boundary is less than the allowable and the vessel is satisfactory. If the slope of this leak testing line exceeds the allowable leakage rate x/t determined in Step 9, the total leakage rate of the test boundary is in excess of the allowable. Then, the leak or leaks that exist must be detected and repaired and the system retested. The actual total leakage rate Q is determined by solving the leakage rate test equation (Eq. 2) for Q by using the leak testing system scale calibration factor K determined in Step 8 and the actual x/t slope value determined in step 13 during the system test. These values are shown in Figs. 8 and 9.

Time Constants for Response and Cleanup of Helium in Large Vacuum Test System

The time for leak detector response to helium and the cleanup time of helium are characteristics of the test system as a whole and do not depend on the leak detector alone. Factors affecting leak testing response times are the geometry of

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the vessel under test, connecting lines, auxiliary pumps, the leak detector and the amount of helium introduced into the leak. It is obvious that a test vessel having a large volume or many small constrictions will cause long delays. A high speed pump may help reduce the delay. Mathematically, the response and cleanup time constants can be calculated:

$$T_c = \frac{V}{S} \quad (3)$$

where V is volume of system; S is pumping speed effective at the connection to the vessel for helium; and T_c is response time at which leak signal is equal to 63 percent of the maximum possible leak signal or, for cleanup, the time to decay to 37 percent of maximum possible indication.

The response time constant is defined as the time for a leak detection system to yield a signal output equal to 63 percent of the maximum signal attained when the tracer gas is applied indefinitely to the system under test. The cleanup time constant is the time required after the helium is removed for the helium indication to be reduced to 37 percent of its maximum value. If one uses a measurement time delay of 2.3 T_c, then response will be 90 percent. If a delay of 5 T_c is used, then response will be essentially the maximum attainable leak signal magnitude.

Effect of Conductance between Leak Detector and Test Vessel

Generally, the factor controlling response and cleanup time in a test system is the conductance of the tubing between the leak detector and test vessel. If the instrument is connected to the vessel by a 1.5 m × 13 mm inside diameter (5 ft × 0.5 in.) hose, the conductance limiting pumping speed S will be about 0.5 $L \cdot s^{-1}$ (1 $ft^3 \cdot min^{-1}$). If the vessel has a volume of 2 m^3 (70 ft^3), then the time constant T_c = 4000 s or about 1.1 h. Experience shows the importance of using hose lines of short length and large diameter (especially the latter). When pumping the test vessel directly with the leak detector, the response and cleanup time will be determined by the effective pumping speed of the leak detector instrument itself.

The leak detector may be directly connected to the vessel or into the foreline of the auxiliary pump system. Helium is applied to the outside of the vessel by capturing it between the vessel and plastic sheeting. In the case of the leak tests of a double walled dewar vessel, the helium tracer gas may be placed either in the inner tank or in the annular space only if inner vessel design enables evacuation of the inner vessel. On very large dewars or cryogenic vessels, this is not usually the case.

The response time formula of Eq. 3 is then used to determine the amount of time it will take for a leak to indicate 63 percent of its total leakage. For example, if a leak detector with a pumping speed of 14 $L \cdot s^{-1}$ (30 $ft^3 \cdot min^{-1}$) were connected directly to a 200 L (7 ft^3) vessel, the response time constant would be 14.35 s.

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PART 2. Tracer Probe Technique for Leak Testing of Evacuated Objects[1,2]

Technique for Locating Leaks in Evacuated Equipment with Helium Leak Detector

Test objects and systems that can be evacuated can be tested for leaks most conveniently by scanning the external surfaces that are open to the atmosphere, with a manually held helium spray probe as sketched in Fig. 1. The mass spectrometer helium leak detector is connected directly to the interior volume of the system under test at a point between the test system and an auxiliary vacuum pump (if the test object size calls for an auxiliary pump). After the object or system under test has been evacuated, the exterior surfaces or suspected areas of the test object are sprayed with a fine jet of helium from a helium probe.

The probe is supplied by a hose connected to a pressure regulator and a tank of compressed helium gas. A portion of any helium tracer gas entering the vacuum system through a leak is drawn into the mass spectrometer leak detector. The increase of helium entering the mass spectrometer tube may be indicated both audibly and visually by alarms. The concentration (partial pressure) of the helium in the spectrometer tube is indicated by the leak rate display. Careful scanning with the helium probe permits positive location of leaks.

Tracer Probe Technique for Helium Leak Testing of Evacuated Test Objects

The tracer probe is used to spray helium on the object to be tested (see Fig. 1). A large helium flow may be used to check the entire surface of a test object. A small helium jet can be used to locate leaks precisely within areas subject to leakage. The only leaks that will be detected, of course, will be those that have been subjected to the helium spray and permit helium tracer gas to enter the evacuated interior volume.

Where the vacuum pumps of the leak detector can maintain adequately low pressure within the system under test, the leak detector can be connected directly to the system with the auxiliary pumps valved out. For larger systems with their own permanent high vacuum and mechanical vacuum pumps, the leak detector can be connected to the foreline between high vacuum and mechanical pumps (Fig. 5). The system mechanical pumps are then throttled as much as possible without allowing an increase in system pressure.

Procedures for Helium Spray Techniques for Vacuum Testing

The following detailed procedures will be very useful in locating the position of a leak.

1. Tracer probing for a leak in an object under vacuum should proceed from the upper side of the test object to the lower side. Then the escaping helium, which rises in air, will flow back only over areas already tested.
2. When initially testing individual joints, time is saved by using a generous flow of helium continuously (e.g., from flexible small diameter tubing). When a leak is indicated, its exact location can be determined by means of a finer probing. By using a fine probe, the operator can limit narrowly the area covered by helium. The leak detector signal will be at a maximum when the probe is directly over the leak.
3. A very large leak will give an indication even when the probe is at some distance away. To prevent this time delaying occurrence, the leak should be located, possibly by less sensitive techniques, and then either repaired or temporarily sealed. Vacuum putty or plastic may be used for temporary seals if care is exercised to remove all the putty before repairing the leak.
4. When an area appears to contain a leak but does not produce a consistent and repeatable leak detector response, a large leak in some other location is to be suspected. The varying helium leak indication may be due to erratic puffs of helium being blown to the large leak.

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5. When two possible points of leakage are close to one another, it is sometimes difficult to determine which of them is responsible for a leakage indication. It is then necessary to mask one leak (say with a plastic bag) so as to exclude its possible influence. A fine probe and a minimum flow of helium will also help to discriminate between two adjacent points of leakage.
6. Numerous different types of leaks can give the same typical leakage indication. The indication is delayed with a slow buildup of the leak signal and then a very slow cleanup. The signal may even stay constant for some time. Such indications are usually due to porosity, flanges with flat gaskets and rubber tubing joints. This peculiar behavior is due to the great length of the leakage path plus the trapping of helium (at atmospheric pressure) in crevices in leaking joints. Similar effects are produced by leaks in volumes that are behind constrictions or that are otherwise being pumped slowly.
7. Testing of subunits before they are incorporated into an assembly or system simplifies testing of the system. Then only connections or joints between units require examination.

Response and Cleanup in Vacuum Testing with Helium Tracer Gas

Two requirements for fast, accurate tracer probe leak testing are of utmost importance.

1. The evacuated system should react as rapidly as possible when a leak is probed. That is, it should have a short response time.
2. When the tracer gas is removed from the leak, the leakage indication should fall to zero in the shortest possible time. That is, the leak detector should have a short cleanup time.

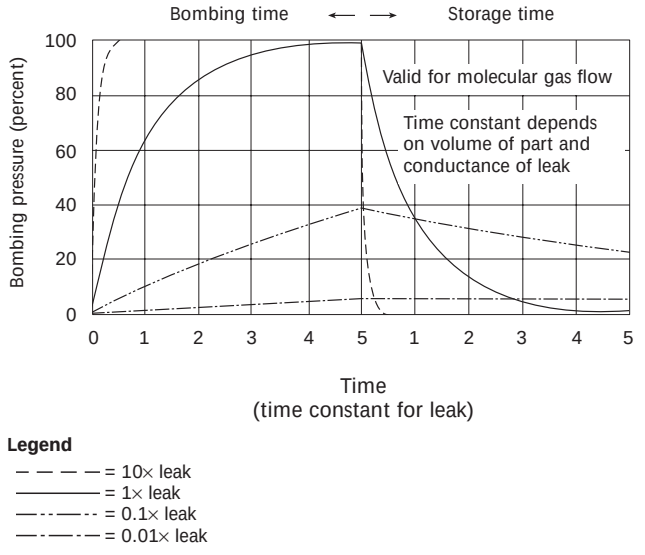
If these requirements are not met, the leak testing process is delayed to a great and sometimes intolerable extent. To illustrate this, picture a section of weld being probed at a constant rate. If the response time is long, the leakage indication will appear some time after the probe has been moved well beyond the leak. The probe will then have to be backtracked slowly until a second signal is obtained. This second signal cannot be observed distinctly until the first signal has been removed or cleaned up. Therefore, the cleanup time is as important as the response time. In fact, if a large leak is encountered it is sometimes necessary to wait much longer than the system response time before the helium level is low enough to permit leak testing to proceed.

In vacuum testing objects or vessels that have internal volume of several liters or more, the response and cleanup times of the helium signal are characteristics of the test system as a whole and are not dependent on the leak detector alone. Factors affecting the leak signal response and cleanup times are (1) geometry and volume of the vessel under test, (2) pressure within the evacuated system, (3) conductance of the connecting lines, (4) auxiliary pumps if used, (5) leak detector type and (6) amount of helium introduced into the system by the leak.

Effect of Conductance and Pumping Speed on Response Time

When a vessel is tested without auxiliary pumps, the factor controlling response and cleanup time is the conductance of the tubing between the leak detector and test vessel. If a leak detector having a pumping speed at the flange of 20 $L \cdot s^{-1}$ (42 $ft^3 \cdot min^{-1}$) for helium, is connected to the vessel by a 12 mm (0.5 in.) inside diameter, 1.5 m (5 ft) long hose, the limiting pump speed will be about 1.4 $L \cdot s^{-1}$ (3 $ft^3 \cdot min^{-1}$) for helium. If the vessel has a volume of 2000 L, the response time will be 2000/1.4 = 1430 s or about 25 min. This would mean that helium would have to be over the leak for 25 min for a 63 percent maximum

FIGURE 10. Internal pressure as a function of helium bombing duration and storage time after bombing, assuming molecular gas flow of test objects.



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reading of total leakage, or 2.1 h for a 99 percent reading of total leakage.

When pumping the same test vessel directly into the leak detector, the response and cleanup times will be determined by the pumping speed of the leak detector only. If the leak detector has a pumping speed at the flange of 20 $L \cdot s^{-1}$ (42 $ft^3 \cdot min^{-1}$) for helium, the time constant will be 100 s or about 1.7 min. This discussion shows the importance of using connecting lines of short length and large diameter between the leak detector and the system under test (see Figs. 3 to 5).

FIGURE 11. Arrangement for sampling probe (sniffer probe) detection of out-leakage from helium pressurized test object to air at atmospheric pressure.

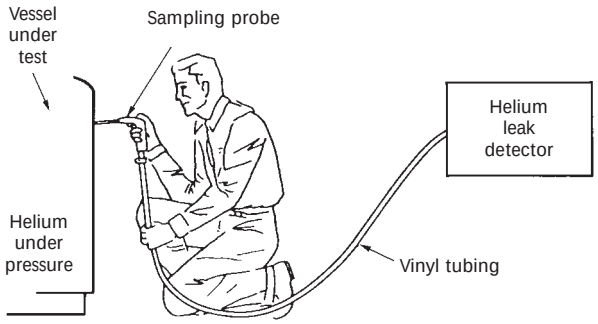
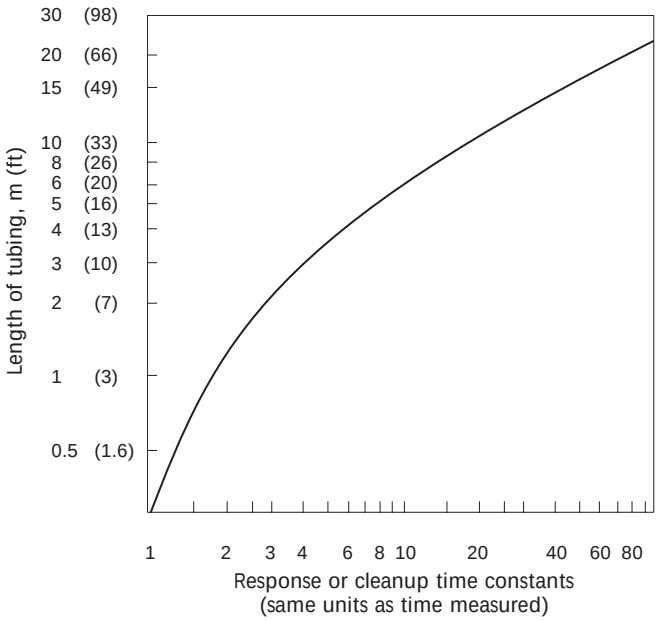


FIGURE 12. Response and cleanup time constants when using a helium sampling probe with 13 mm (0.5 in.) inside diameter tubing when used with a conventional leak. Response time constant = time to 63 percent of maximum leakage rate; cleanup time constant = time to 37 percent of maximum leakage rate; full response = 5 time constants.

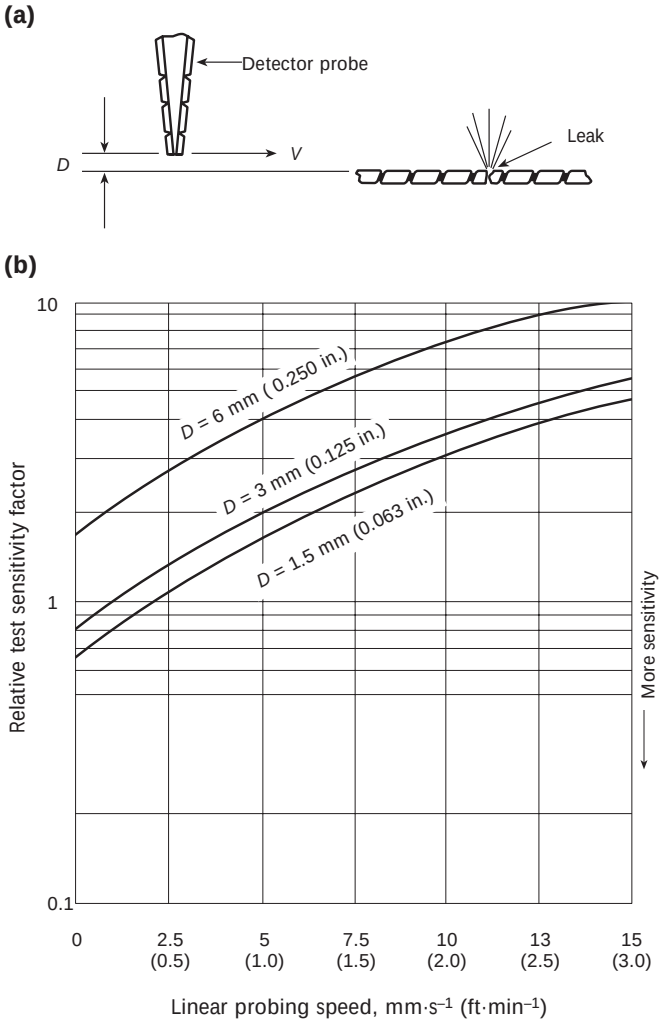


Capabilities of Tracer Probe Leak Detection with Helium Mass Spectrometer

The ASTM techniques of tracer probe helium leak detection[1] are for testing and locating the sources of gas leaking at the rate of 1×10^{-10} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-9} std $cm^3 \cdot s^{-1}$) or greater. The test may be conducted on any object to be tested that can be evacuated and to the other side of which helium or other tracer gas may be applied. These tracer probe helium leak testing techniques are Method A, Method B and Method C.

1. Method A is used for objects that can be evacuated but have no integral pumping capability.
2. Method B is used for test objects with integral pumping capability.

FIGURE 13. Relative helium leak testing sensitivity as a function of liftoff *D* and of speed *V* of scanning with detector probe: (a) schematic; (b) sensitivity curves.



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3. Method C is used for test objects as in Method B, in which the vacuum pumps replace those normally used in the leak detector.

These techniques require a helium leak detector that can detect a leak of 1×10^{-11} Pa·m^3·s^{-1} (1×10^{-10} std cm^3·s^{-1}).

Summary of Tracer Probe Methods A, B and C, Recommended by ASTM

Method A of the American Society for Testing and Materials (ASTM) is used to helium leak test objects that can be evacuated to a reasonable test pressure by the leak detector pumps in an acceptable length of time. This requires that the object be clean and dry and usually no larger than 100 L (0.1 m^3 or 4 ft^3) in volume. Also, to cope with a larger volumes or relatively dirty devices, auxiliary vacuum pumps having greater capacity than those in the mass spectrometer leak detector may be used in conjunction with the mass spectrometer leak detector. The leak test sensitivity will be reduced under these conditions.

ASTM Method B is used to leak test equipment that can provide its own vacuum (that is, equipment that has a built-in pumping system) at least to a level of 0.5 kPa (a few torr) or lower.

ASTM Method C is used when a vacuum system is capable of producing internal pressures of less than 30 mPa (0.3 mtorr) in the presence of leaks. These leaks may be located and evaluated with either a residual gas analyzer or by using the spectrometer tube and controls from a conventional mass spectrometer leak detector — if leakage is within the sensitivity range of the residual gas analyzer or mass spectrometer leak detector under the conditions existing in the vacuum system.

Test Conditions That Interfere with Tracer Probe Helium Leak Testing

Series leaks with an unpumped volume between them (Fig. 14b) are difficult, if not impossible, problems in helium leak testing. The tracer gas enters the first leak readily enough because the pressure difference of helium across the first leak is near full atmospheric pressure. However, it may take many hours to build up the partial pressure of helium in the volume between the two leaks so that enough helium enters the vacuum system to be detected by the mass spectrometer leak detector.

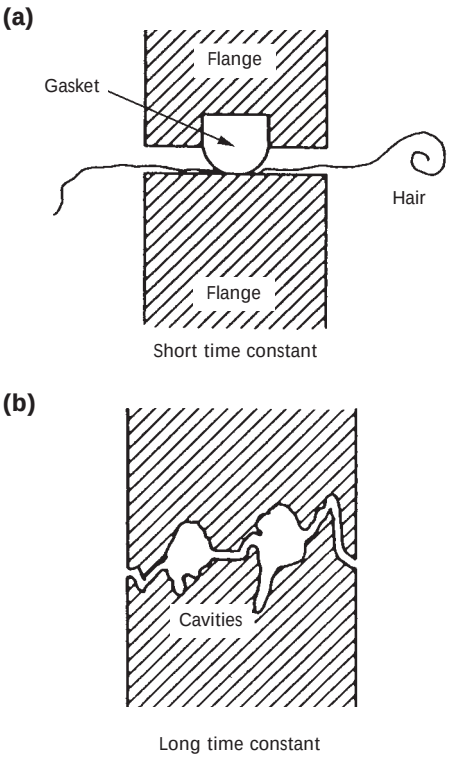
These are also called *virtual leaks*. This type of leak occurs frequently under the following conditions: (1) double welded joints and lap welds, (2) double O-rings, (3) threaded joints, (4) ferrule and flange tubing fittings, (5) castings with internal voids, (6) flat polymer gaskets and (7) unvented O-ring grooves.

In general, the solution is in proper design to eliminate these conditions. However, when double seals must be used, an access port between them should be provided for attachment to the mass spectrometer leak detector. Leaks may then be located from each side of the seal. After repair, the access port can be sealed or pumped continuously by a holding pump on large vacuum systems.

Temporarily plugged leaks often occur because of poor manufacturing techniques. Water, cleaning solvent, plating, flux, grease, paint etc. are common problems. To a large extent, these problems can be eliminated by proper preparation of the parts before leak testing. Proper degreasing, vacuum baking and testing before plating or painting are desirable.

In a device being tested, capillary tubing located between the leak and the leak detector can make leak testing extremely difficult. Test sensitivity is drastically reduced and response time increased. If there is a volume at each end of the capillary, each such volume should

FIGURE 14. Effect of nature of leak on time constant of leakage measurements: (a) direct leak; (b) series or compound leak.



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be attached to the leak detector during testing. If this is impossible, the device should be surrounded with a helium atmosphere while attached to the leak detector for a long time to ensure leak tightness. When unusually long pumping times are necessary, the connections to the leak detector (and all other auxiliary connections) that are exposed to the helium should be double sealed. The space between the seals should be evacuated constantly by a small auxiliary roughing pump. This prevents helium from entering the system through seals that are not a part of the device to be tested.

Equipment for Helium Leak Testing of Small Devices by Using the Mass Spectrometer Leak Detector

Leak testing of small devices requires a helium mass spectrometer leak detector having a minimum detectable leakage rate as required by the test sensitivity. Use can be made of auxiliary pumps capable of evacuating the object to be tested to a pressure low enough that the mass spectrometer leak detector may be connected. (If the object under test is small and clean and the mass spectrometer leak detector has an integral roughing pump, the auxiliary pumps are not required.) Suitable connectors and valves are used to connect to the mass spectrometer leak detector test port. Compression fittings and metal tubing should be used in preference to vacuum hose. A vacuum gage is used to read the pressure within the test object before the mass spectrometer leak detector is connected. A helium tank and regulator with attached helium probe hose and jet are used for the tracer probe.

Standard Leaks Used in Tracer Probe Helium Leak Testing

Standard leaks of the capsule type contain an internal helium supply. The quartz or permeation type leak should have a leakage rate volume approximately 10^2 to 10^3 times greater than the minimum leakage detectable by the leak detector. During calibration of the mass spectrometer leak detector, the standard leak is attached to the mass spectrometer leak detector. The mass spectrometer leak detector is tuned to achieve maximum sensitivity in accordance with the manufacturer's instruction. Standard leaks are used to simulate the reaction of the test system to helium spray. The capillary type leak should have a leakage rate about the same or slightly smaller than the test requirement.

Slow Leak Response Effects of Welds and Joints in Large Vessels

A number of different types of seals may give a delayed and slow buildup of the leak signal followed by a very slow cleanup time. Such indications are usually due to porosity, flanges with flat gaskets, tubing connections and tortuous paths in welds or soldered joints. This peculiar behavior is due to the great length of the leakage path plus the store of helium in the crevices or joints. This situation emphasizes the fact that helium must remain over suspected leakage areas for a sufficient period of time to detect a leak of this type.

Metallic Enclosures for High Sensitivity Helium Tracer Probe Leak Testing

Boxes, housing, enclosures or hoods for the vacuum technique of producing a local area of pressure differential for high sensitivity tracer probe leak detection or leakage measurement tests should be: (1) as lightweight as possible; (2) designed to withstand external pressure of 100 kPa (1 atm) while the interior volume is at extreme vacuum levels; (3) constructed of metal for production leak testing; (4) free of rough interior surfaces for thorough cleaning; (5) shaped to closely fit the contour of the surface to which the box, housing, enclosure or hood is to be temporarily sealed; (6) capable of being sealed to the items(s) being leak tested with a leaktight seal (here and in the following discussion, *leaktight* means that there is no leakage detectable within the sensitivity of the leak testing system); and (7) equipped with valved connections for a system standard leak, gaging sensor (optional), vent (optional) and a hose or tubing connection to the evacuation pump system and high sensitivity tracer gas leak detector.

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Materials and Construction of Metallic Enclosure for High Sensitivity Vacuum Leak Tests

The welded boxes, housings, enclosures or hoods used for high sensitivity vacuum leak testing are usually constructed of aluminum or stainless steel. These materials are more readily cleaned and have a lower outgassing rate than carbon steel. If they are sufficiently large, they may be designed with stiffeners to reduce their weight. These enclosure assemblies should be welded with full penetration welds or with continuous internal fillet welds and intermittent external fillet welds. This prevents formation of trapped spaces between continuous double fillet welds that form potential areas of virtual leakage.

Sealing Enclosure for High Sensitivity Vacuum Leak Tests

If the area to be leak tested has a machined sealing surface, the box, housing, enclosure or hood should be equipped with a flange for effecting a leaktight gasket seal. If there is no machined surface against which to form a leaktight seal with a gasket, then the box, housing, enclosure or hood should not be equipped with a flange. Instead, while the vacuum pump system is operating, a leaktight seal should be effected with a pliable sealer such as putty or the housing seal as shown in Fig. 15. The valved connections on the assembly for either required or optional items should be of high vacuum quality. Before using a box, housing, enclosure or hood for production work, it should be leak tested to determine that the complete assembly is sufficiently leaktight for the production leak testing to be performed.

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PART 3. Hood Technique for Leak Testing of Evacuated Objects[2,3]

Technique for Leakage Rate Testing of Evacuated Equipment under Helium Filled Hood

Often, a test for total leakage rate is required or desired. The best way to conduct this type of test is to enclose all or part of the evacuated system under test with an atmosphere containing helium, as sketched in Fig. 3. The helium will enter the system under test if any leaks are present. The internal volume of the test object is connected directly to a vacuum pump. The leak detector in turn is connected to the vacuum pump system. Helium leaking from the hood into the test object is detected by the leak detector.

This test permits the determination of a total leakage rate for the entire system if the auxiliary mechanical pump inlet valve is closed. Testing can be done without throttling (and reduced sensitivity) if a counterflow leak detector is used. Various materials such as rubber sheets, plastic bags and metal hoods can be used for envelopes. The hood or envelope technique not only combines certainty of detection with the highest sensitivity but also is suitable for leak testing equipment moving on assembly lines.

For testing larger, high vacuum equipment at pressure below 25 mPa (0.2 mtorr), the helium leak detector is connected to the equipment at a point between the auxiliary diffusion pump and the mechanical foreline pump, as in Fig. 5. The pressure at this location is still low enough to permit any mass spectrometer leak detector to operate at its maximum sensitivity. The diffusion pump compresses the gas into the foreline between the diffusion pump and the mechanical pump, so that its pressure is higher than that in the evacuated system under test. This increases the partial pressure of helium entering the sensor of the mass spectrometer leak detector. Thus, the instrument detects in-leakage to the evacuated system under test with greater sensitivity than if it were connected directly to the system. This technique is also suited for testing large vacuum vessels.

Detecting Porosity by Helium Permeation with the Hood Technique

The porosity of almost any material, including metals and ceramics, can be measured by evacuating a vessel made of the material, immersing it for an extended period in an atmosphere of known helium concentration and then measuring the helium concentration in the interior of the vessel with the leak detector. The soak time depends on vessel wall thickness and the temperature. Helium permeates some polymers and glasses (even in the absence of porosity), as in some types of helium standard leaks. (Caution is appropriate when leak testing glass envelope electron tubes to avoid replacing their vacuum with helium.)

Hood Technique for Applying Helium to Evacuated Test Objects

A convenient and rapid in-leakage test can be performed on a test object with a hood as shown in Fig. 3. By using a hood, a helium atmosphere can be maintained around the test object and a measurement of total in-leakage can be made that could constitute a go/no-go test. The actual location of the leaks cannot be found by this hood technique, however, and vacuum testing with a small helium spray can be used for actual leak location. Both the hood and tracer probe techniques may be used in sequence for production leak testing. The test objects can be tested in the helium hood for go/no-go leakage. The test objects can also be scanned with the helium tracer probe to actually locate the leaks so that repair can be made. The hood may consist of a plastic bag filled with helium. On large complex systems being vacuum tested, small areas may be individually bagged with helium, thus saving the time and expense of bagging an entire system.

Vacuum Testing of Large Vessels in Helium Filled Enclosures

Many large vessels are constructed where in-leakage must be determined. this is accomplished by evacuating the vessel and applying helium to the outside of the

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tank. Helium may be applied to the entire tank at one time by capturing it between plastic sheeting and the vessel for determining total in-leakage of helium. Alternatively, helium may be sprayed on suspected leakage areas to determine the point of leakage.

If the vessel to be evacuated is large, it is usually desirable to use a high speed auxiliary turbomolecular pump or a diffusion pump in conjunction with a mechanical pump (see Fig. 5). In these cases, the leak detector is connected into the foreline of the auxiliary pump. This way an adequate flow of sample gas to the leak detector will be ensured because the pressure in the foreline is higher than that in the leak detector.

Connected directly to the vessel, the detector would be robbed of helium by the auxiliary pump if it is allowed to pump on the vessel. If the leak detector pumps can maintain a low pressure in the vessel under test (less than required by the leak detector when the auxiliary pumps are valved off), the mass spectrometer leak detector instrument may be directly connected to the vessel. In either case, once the vessel has been evacuated to a low pressure by the auxiliary pumps, the leak detector may be valved into the system.

Response Time Technique for Vacuum Testing of Large Vessels

One time saving technique that may be used in vacuum testing large vessels is the response time technique. In this technique, the leak detector may be connected directly to the vessel or into the foreline of the auxiliary diffusion pump. Helium is applied to the outside of the vessel with a hood or plastic bag. In the case of a dewar type vessel, the helium may be placed in the inner tank or in the annular space. The response time for the leak detector to indicate 63 percent of the total leakage is determined again by the ratio of the vessel volume to the pumping speed for helium at the leak detector's connection to the vessel.

For example, if a leak detector with a pumping speed of 10 $L \cdot s^{-1}$ for helium were connected directly to a 2.4 m^3 (85 ft^3) vessel, the response time to reach 63 percent of the total leakage indication would be 240 s or 4 min. The time to reach 99 percent of the total leakage would be about 1100 s or 18.5 min. Thus, the total leakage rate could be approximated by multiplying the leakage rate indicated after only one response time (equivalent to the system time constant) by the factor 1/0.632 = 1.58.

Measuring Helium Leakage Rates during Vacuum Testing

The sensitivity for leak testing is a function not only of the leak detector sensitivity but of the nature of the test object and the pumping equipment. Consequently, it is desirable to be able to measure the sensitivity of the test system as a whole. This is required by at least two standards.[2,3] For such a test, a calibrated leak is installed in the test system at a point such that it will be subjected to the same pumping conditions as the test object. Then, the size of an unknown leak can easily be calculated by comparing its output meter reading on the leak detector with that caused by the calibrated leak. For example, an unknown leak that causes a deflection three times that caused by the calibrated leak has a leakage rate three times that of the calibrated leak, provided that the helium tracer gas concentration is the same for both the calibrated and the unknown leaks. When making these measurements, the operator should leave unchanged the setting of the valves that affect the pumping speed of the detector.

An example illustrating the above conditions would be approached in the following manner. The response time or time constant for the system would be established for helium. The sensitivity of the leak detector, if not already known, would then be calculated:

$$\textbf{(4)} \quad \text{sensitivity} = \frac{\text{leakage rate}}{\text{output}} \times K_T$$

where sensitivity is helium sensitivity of the leak detector in $Pa \cdot m^3 \cdot s^{-1}$ per scale division. Output is in net meter scale divisions, as shown on the output meter of the instrument for a standard capillary or permeation leak multiplied by range switch setting. K_T is temperature correction, shown on the standard leak, usually 3 percent per degree celsius (1.5 percent per degree fahrenheit). Division is smallest leakage rate meter scale graduation.

Test specifications often require that the minimum detectable leakage rate be known. This will define the size of what is the smallest leak that can be reliably detected. It is calculated by the following:

$$\textbf{(5)} \quad \text{MDL} = \text{sensitivity} \times \text{noise}$$

where MDL is minimum detectable leakage rate in $Pa \cdot m^3 \cdot s^{-1}$ (std $cm^3 \cdot s^{-1}$); sensitivity is sensitivity of leak detector, $Pa \cdot m^3 \cdot s^{-1}$ (std $cm^3 \cdot s^{-1}$) per division; noise

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is divisions indicated on output meter in the form of spurious outputs (noise, drift and helium background variations), multiplied by a factor of 2 for ideal conditions. For practical field test conditions, the multiplying factor must be increased considerably.

Semiautomatic Test Port for Rapid Vacuum Leak Testing

A rapid vacuum leak test of production parts such as bellows, connectors and glass-to-metal seals can be made with an automatic test port as sketched in Fig. 16. In this setup, the test object is attached to the test port by means of a quick disconnect fitting. The part is automatically roughed out through the auxiliary pump valve to a preset pressure and opened to the leak detector through the leak detector test valve. Helium is then applied to the exterior of the unit under test either by a hood or by helium spray. After the unit has been tested, the leak detector protection valve is closed and the vent valve opened. The part can then be removed and another part attached for test. The total test time can be as low as 6 s per unit. The test port also protects the leak detector from high pressure rises as the leak detector test valve will close if the pressure at the source rises above 25 mPa (0.2 mtorr).

FIGURE 15. Arrangement used for high sensitivity pressure/vacuum helium leak testing with evacuated metallic enclosures.

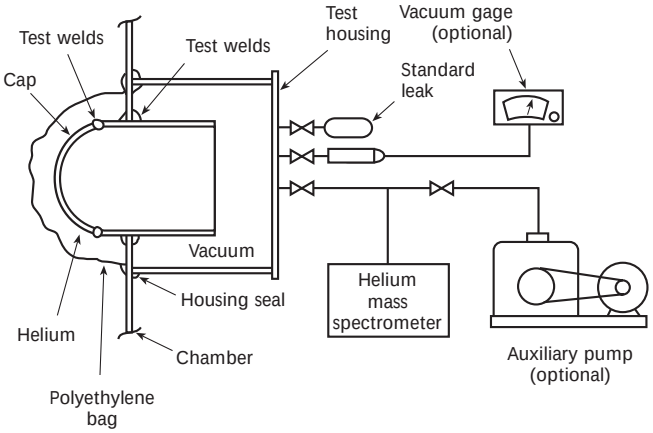
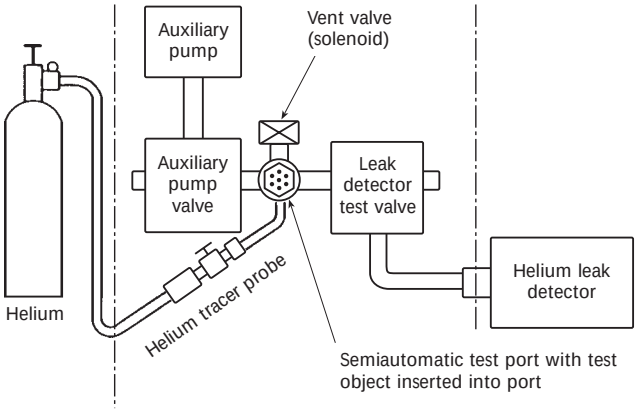


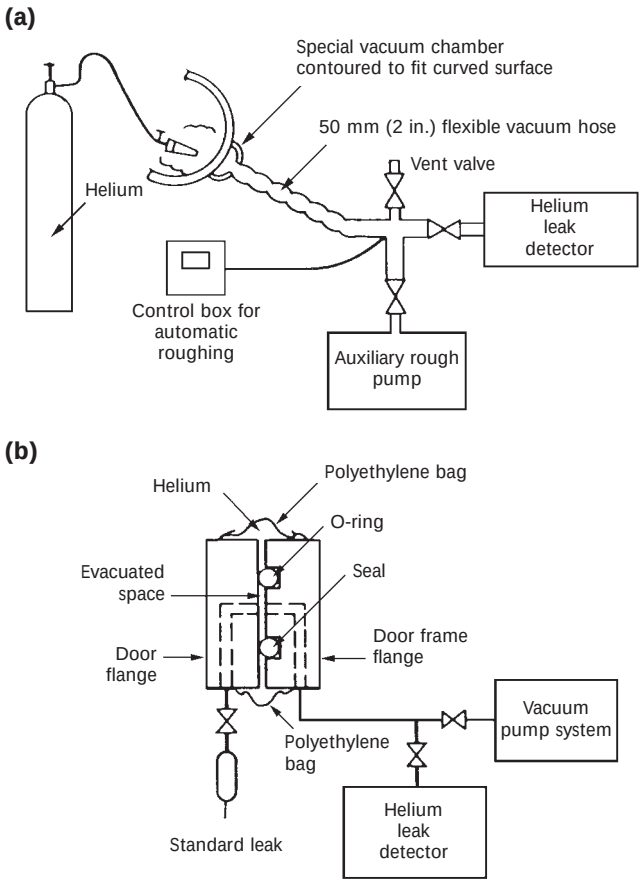
FIGURE 16. Typical setup for rapid leak testing, with semiautomatic test port.



Equipment Arrangements for Rapid Helium Leak Testing of Welds in Structures

Leak tests of welds in large vessels or sections of large structures can be made rapidly with special test arrangements illustrated in Figs. 15, 17 and 18. In each arrangement, a small welded section is sealed by a closely fitting vacuum chamber volume connected to a vacuum pump and to the leak detector. The

FIGURE 17. Special equipment for leak testing of welds: (a) leak testing of welds in contoured areas of large vessels; (b) leak testing of double O-ring seals on chamber doors.



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valving used is that of the automatic test port. Helium is sprayed or bagged on the other side of the weld and any leakage in that area is read by the leak detector. This application offers a means of locating leaks in welds in various sections of a tank before final assembly. The speed of testing can be high when the weld surface is smooth and the small vacuum chamber fits its closely. With rough weld surfaces, leakage at the vacuum seal with the surface can lower both test sensitivity and speed. Figure 17b is the cross section of a large vessel door flange, showing how to evacuate the volume between two concentric O-rings.

Procedures for Hood Technique Helium Leakage Tests of Large Vacuum Systems

A hood technique leakage test is performed (1) by evacuating the boundary under test, (2) by blanketing all or part of the test boundary with helium and (3) by detecting leaks or measuring total leakage through the test boundary using a helium mass spectrometer with the arrangements shown in Fig. 18. This test is more commonly done as a helium spray probe leak test (Fig. 18a) when used for preliminary leak detection and location. The test is performed by the helium bag or hood test method when total leakage is to be measured (Figs. 18b and 18c).

Estimating Leak Test Response Time and Cleanup Time for Hood Tests

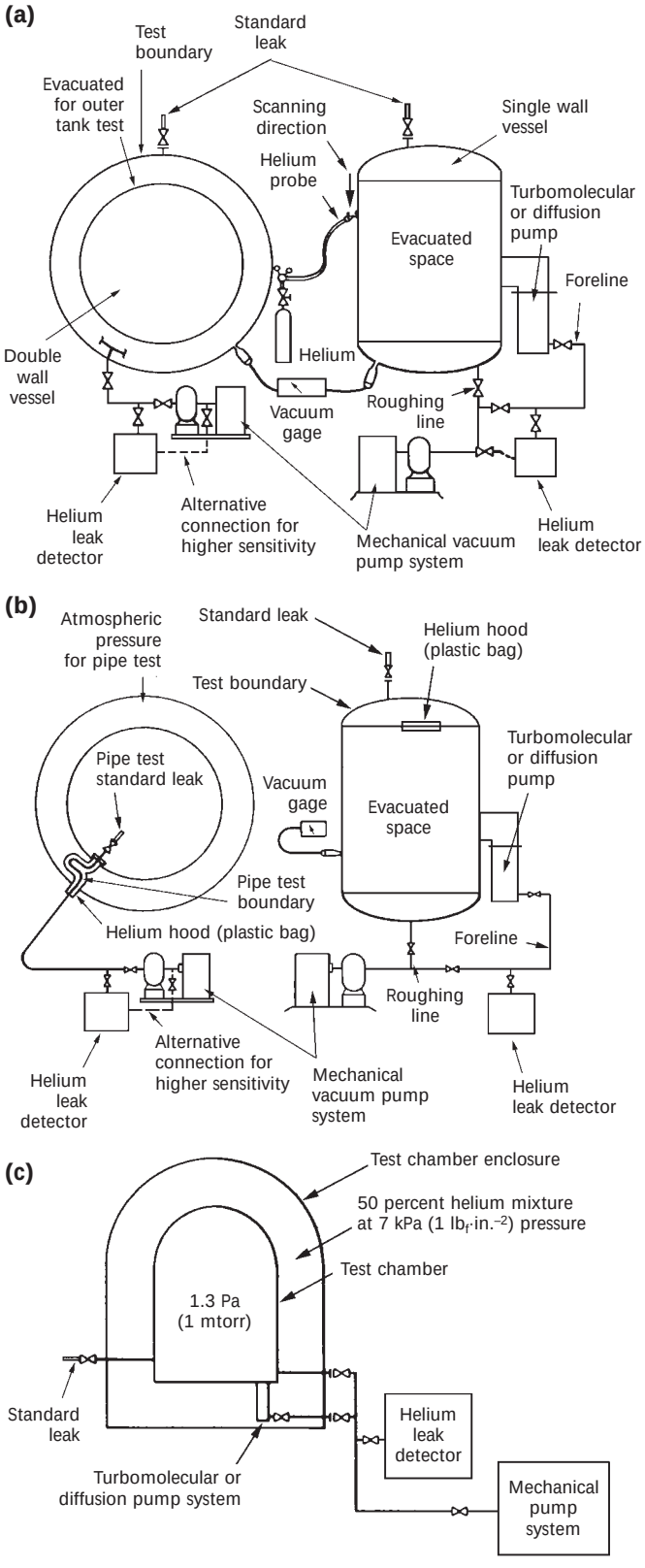
To determine if the test method is practical for testing a particular vacuum system, Eq. 6 can be used (1) to determine the appropriate leak detector response and cleanup time when effective pump speed is known or (2) to determine the approximate effective pump speed required for a given response and cleanup time when a temporary vacuum pump system must be installed for this test.

$$S = \frac{V}{T} \quad \textbf{(6)}$$

where T is response or cleanup time (second); V is volume of evacuated test boundary (cubic meter or cubic foot); S is effective pump speed at the test boundary during test (cubic meter per second or cubic foot per second).

Response time is not to be confused with waiting time. On large evacuated

FIGURE 18. Leak testing of large vessels for leaks into evacuated space: (a) helium probing of large single wall or double wall vacuum systems; (b) helium hood leak test of pipe in large double wall vacuum system and a large single wall vacuum system; (c) helium leak testing for leaks into internal volume from annular space between double walls.



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systems, a waiting time of a few seconds or more can exist between the time the tracer gas is applied to the test boundary and the time it reaches the helium mass spectrometer leak detector (see Fig. 19).

Practical limits of response and cleanup time for large systems should not exceed about 5 min. Longer times make testing extremely time consuming. As can be seen from Eq. 6 for a given system volume, the larger the effective pump speed the shorter the response time. For this reason, this test is most commonly used on vacuum chambers having high speed diffusion pumps in their system. On these systems, the helium leakage test is used for both preliminary testing and measurement of total leakage rates.

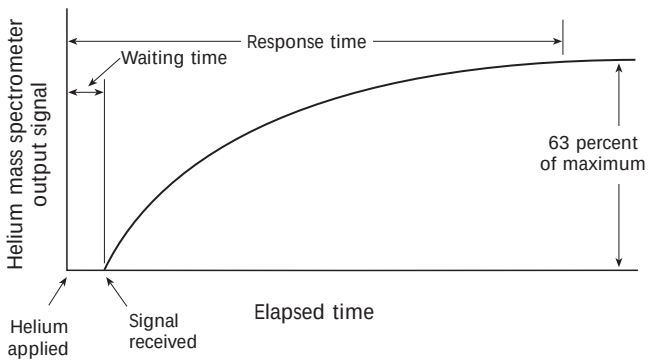
On double wall evacuated cryogenic vessels that usually have only mechanical pump systems with low pump speeds in the 0.1 to 1 Pa (1 to 10 mtorr) pressure range, this test cannot be used to determine total leakage rate because of excessive response time. However, the short waiting time permits this test for preliminary testing of cryogenic vessels. In this case, response time is less important because the purpose is to pinpoint leaks, not measure total leakage rate. When pinpointing leaks, the tracer gas need only be applied with a probe to the test boundary for a very short period for the leak indicator to show a detectable signal.

For example, a 10 000 m^3 (350 000 ft^3) environmental chamber has an effective pump speed during testing of 54 $m^3 \cdot s^{-1}$. Equation 6 helps determine what approximate response and cleanup time can be expected:

$$T = \frac{V}{S} = \frac{10 \times 10^3}{5.4 \times 10^1}$$

$$= 1.85 \times 10^2 \text{ s}$$

FIGURE 19. Graph showing waiting time and response time (time constant) for dynamic leak testing with helium mass spectrometer.



To determine the effective pump speed of a vacuum system, use the pump speed curve for the vacuum pump or pumps in question (see Fig. 20). From this curve, obtain the rated speed for the pump for the known or specified test pressure. Normally, rated pump speed would be corrected using conductance of the system components to obtain effective pump speed. However, for the purpose of testing estimation, determine effective pump speed:

$$S = \frac{S_r}{\text{SF}} \quad (7)$$

where S_r is rated pump speed from curve (cubic meter per second or cubic foot per minute) and SF is service factor.

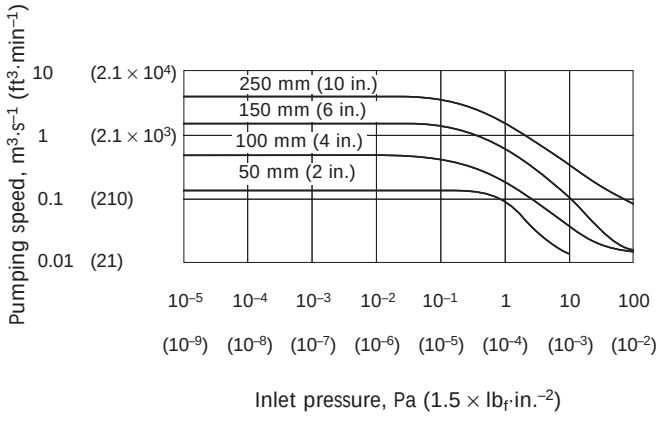
Use SF = 4 for a diffusion pump with a cold baffle and SF = 2 for an unbaffled diffusion pump. These service factors are based on the assumption that the chamber opening for the diffusion pump is as large in diameter as the throat of the pump.

For example, a 250 mm (10 in.) diffusion pump is available for leak testing. Minimum backstreaming of oil is required, so a cold baffle must be installed with the diffusion pump. What will be the approximate effective pump speed? From Fig. 20 and Eq. 7, pump speed curve $S_r = 4.2$ $m^3 \cdot s^{-1}$ (in the pressure range of 0.01 to 10 mPa). If SF = 4, then $S = (4.2/4) = 1.05$ $m^3 \cdot s^{-1}$.

Determining Throughput to Leak Detector

The sensitivity of a vacuum system is a direct function of the ratio of the mass flow of gas (throughput) being pumped from the vacuum system to the mass flow of gas (throughput) in the leak detector. Because throughput = pressure × effective

FIGURE 20. Typical curves relating vacuum pump speed to operating pressure at inlet to diffusion pumps.



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pump speed, this relationship is shown by Eq. 8:

$$(8) \quad Q_s = Q_m \frac{Q}{Q_1} = Q_m \frac{PS}{P_1 S_1}$$

where Q_s is system sensitivity (pascal cubic meter per second); Q_m is leak detector sensitivity (pascal cubic meter per second); Q is system throughput (cubic meter per second); Q_1 is leak detector throughput (cubic meter per second); P is system pressure (pascal); S is system effective pump speed (cubic meter per second); P_1 is leak detector sensing element pressure (pascal); S_1 is leak detector effective pump speed (cubic meter per second).

Determination of Sensitivity of Helium Leakage Rate Tests

Equation 8 shows that as system pressure decreases, the mass flow decreases to the leak detector. By throttling the system mechanical pumps, a higher and higher portion of the system diffusion pump throughput may be backed by the helium mass spectrometer. Increasing the volume of flow to the instrument by throttling of the vacuum system mechanical pumps will result in a direct increase in instrument sensing element pressure. As decreasing system throughput approaches the throughput capability of the leak detector, the ratio of the two throughputs, as shown in Eq. 8, decreases. If the mechanical pumps can be completely throttled from the system. The system throughput becomes the instrument throughput. Then the throughput ratio in Eq. 8 is equal to a value of one and the system sensitivity approaches helium mass spectrometer leak detector sensitivity.

The minimum leakage (system sensitivity) that will produce a detectable output signal on the helium mass spectrometer leak indicator and response time estimated by using Eq. 6 will establish the feasibility of the test.

For example, a 700 m^3 (2.5×10^4 ft^3) spherical vacuum chamber will have an effective pump speed of 20 $m^3 \cdot s^{-1}$ (4.24×10^4 $ft^3 \cdot min^{-1}$) during test and must be evacuated to 0.1 mPa (1 µtorr). Assuming an instrument sensing element pressure of about 10 mPa(0.1 mtorr) and an estimated effective pump speed of 10^{-2} $m^3 \cdot s^{-1}$ (10 $L \cdot s^{-1}$ or 21 $ft^3 \cdot min^{-1}$), what will be the theoretical sensitivity for this system based on a 100 percent helium mixture? Is the vacuum pump system adequate for a reasonable response time? System throughput $Q = (20)(10^{-4}) = 2 \times 10^{-3}$ $Pa \cdot m^3 \cdot s^{-1}$ (2×10^{-2} std $cm^3 \cdot s^{-1}$). Leak detector throughput $Q_1 = (10^{-2})(10^{-2}) = 1 \times 10^{-4}$ $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-3} std $cm^3 \cdot s^{-1}$). Estimated throughput ratio using Eq. 8 is:

$$\frac{Q}{Q_1} = \frac{2 \times 10^{-3}}{1 \times 10^{-4}} = 20$$

Assume minimum detectable helium mass spectrometer signal to be one scale unit. If system sensitivity $Q_s = 4 \times 10^{-10}$ $Pa \cdot m^3 \cdot s^{-1}$ (4×10^{-9} std $cm^3 \cdot s^{-1}$), based on 100 percent helium, response time T follows:

$$T = \frac{V}{S} = \frac{700}{20} = 35 \text{ s}$$

When effective pump speed and system sensitivity are known or specified and the system pressure required to attain the specified system sensitivity must be estimated, determine throughput ratio. Then use Eq. 8 to estimate the system pressure required.

For example, a 300 m^3 (10 500 ft^3) vacuum vessel has an allowable total leakage of 1×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-6} std $cm^3 \cdot s^{-1}$). A 250 mm (10 in.) unbaffled diffusion pump with an effective pump speed of 2 $m^3 \cdot s^{-1}$ (4000 $ft^3 \cdot min^{-1}$) is available for leak testing. What approximate theoretical pressure must be attained to achieve required system sensitivity using 100 percent helium? Is this diffusion pump adequate from the standpoint of response time? Assume minimum detectable helium mass spectrometer signal to be one scale unit. Throughput ratio $Q/Q_1 = 200$.

Assume instrument sensing element pressure $P_1 = 10$ mPa and estimated effective pump speed $S = 0.01$ $m^3 \cdot s^{-1}$ (40 $ft^3 \cdot min^{-1}$). Using Eq. 8, required system pressure is derived as follows:

$$P = \frac{Q}{Q_1} \frac{P_1 S_1}{S} = 200 \frac{10^{-2} \times 10^{-2}}{2} = 100 \times 10^{-4} \text{Pa} = 10 \text{ mPa}$$

From Eq. 6, response time $T = V/S = 300/2 = 150$ s (adequate).

The helium mass spectrometer leak detector should be connected to the foreline of the diffusion pump (see Fig. 3). Here, the higher pressure results in a helium partial pressure above the minimum detectable for the instrument (see Fig. 21). The approximate

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relationship among the economical helium conductance C, instrument connection length l and diameter d has been established as $C = d^3/l$. Therefore, to prevent the instrument connection from being a major factor affecting system sensitivity, the helium mass spectrometer connection to the pump foreline should be as short as possible and no smaller than 13 mm (0.5 in.) inside diameter.

The more helium background in a vacuum system, the less the system sensitivity. This is due to the masking of leakage output signals that are smaller than the output signal from the background. System sensitivity is directly proportional to the measured helium tracer gas concentration during leak testing, as shown by Eq. 9 for test sensitivity:

$$\textbf{(9)} \quad Q_s = Q_c \times \frac{100\%}{\text{Test \% helium}}$$

where Q_s is test sensitivity, Q_c is calibration sensitivity and percentage is test percentage of helium. For example, the sensitivity of a system during calibration with 100 percent helium is 1×10^{-7} Pa·m^3·s^{-1} (1×10^{-6} std cm^3·s^{-1}). During test, the helium concentration is lowered to 25 percent. What is the system sensitivity (minimum detectable leakage) during leak testing?

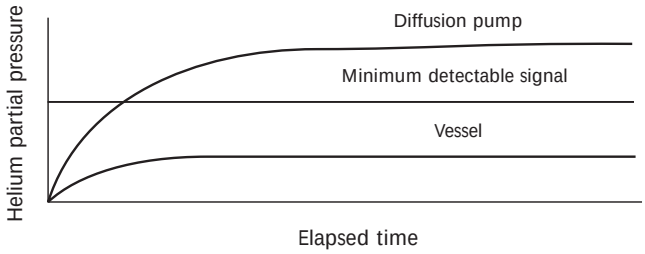
$$Q_s = \left(1 \times 10^{-7}\right)\frac{100}{25}$$
$$= 4 \times 10^{-7}\ \text{Pa}\cdot\text{m}^3\cdot\text{s}^{-1}$$
$$\left(= 4 \times 10^{-6}\ \text{std}\cdot\text{cm}^3\cdot\text{s}^{-1}\right)$$

Refrigeration System Leakage Measurement with Helium Filled Enclosures

The basic problem in performing an outside-in leak test of long tube (such as a refrigeration coil) is the long response time for leaks far from the evaluation point. This problem can be solved by measuring the viscous flow of air through the tube during the test. This flow at nearly sonic speed will entrain any in-leakage and sweep it to the leak detector.

Careful control of the flow is required. The control for this purpose also serves as a pressure dropping device with the result that pressure inside the coil system remains at a differential of about 100 kPa (1 atm) below the outside of the unit. By hooding the unit with helium, any leaks that are present will allow helium to enter the unit and then be rapidly carried by the moving air stream towards the vacuum pump. The leak detector is attached near to and in parallel with the vacuum pump. A proportionate sample of the air/helium mixture is drawn into the leak detector. A fixed throttling restrictor incorporated with the automatic test valve on the leak detector provides fixed flow splitting.

FIGURE 21. Effect of placing mass spectrometer helium leak detector in foreline of diffusion pump (upper curve) where the higher pressure results in a helium partial pressure above the minimum detectable for the instrument.



This leak testing technique is semiautomatic. The operator is required to attach a carrier gas/restrictor line to one end of the refrigeration system, the leak detector to the other end, both by quick connectors, and then to initiate test by activating a function switch. The no-go point is precalculated and set by the threshold control on the audio alarm to automatically indicate the reject point. A gross leak condition is automatically signaled by audio alarm.

Because the helium filled hood test technique incorporates dynamic flow, the problems associated with helium accumulation are nonexistent. Determining the test system sensitivity is relatively easy because a reference leak can be attached to the manifold line to permit direct leak rate calibration. Production leak testing rates by this technique are governed by the basic configuration of the unit under test as with other techniques. A complete test cycle for a typical household type refrigeration coil unit with minimum restrictions is about 15 to 20 s. This includes connection, carrier flow equilibrium, soak, leak indication and disconnect time.

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PART 4. Accumulation Technique for Leak Testing of Evacuated Objects

Helium Accumulation Leak Testing of Large Double Wall Tanks and Vessels

Helium accumulation leak testing of double wall tanks and vessels is performed after the test boundary is evacuated (see Fig. 22) and the vacuum pump system has been isolated. The first step involves blanketing all of the test boundary with helium. Then, the total leakage through the test boundary is measured with a helium mass spectrometer, as shown in Fig. 22. The determination of total leakage rate is accomplished when helium passes through openings in the test boundary and accumulates in the evacuated annular space connected to the helium mass spectrometer. This leak testing technique is most commonly used to determine the total leakage rate of inner vessels for vacuum insulated liquid oxygen, liquid hydrogen and liquid nitrogen cryogenic storage tanks that are designed with no capability for either a permanent or temporary high speed diffusion or turbomolecular vacuum pump system.

Sensitivity Capabilities of Helium Accumulation of Leakage Rate Test

The maximum sensitivity of leakage rate tests for large vacuum systems with little or no effective pump speed at test pressure, such as shown in Fig. 22, is limited mainly by the economics of time, helium costs and system volume. For a system volume of 300 m^3 (10 000 ft^3), the maximum system sensitivity or total leakage rate that might be economically measured is in the range of 10^{-5} to 10^{-7} Pa·m^3·s^{-1} (10^{-4} to 10^{-6} std cm^3·s^{-1}).

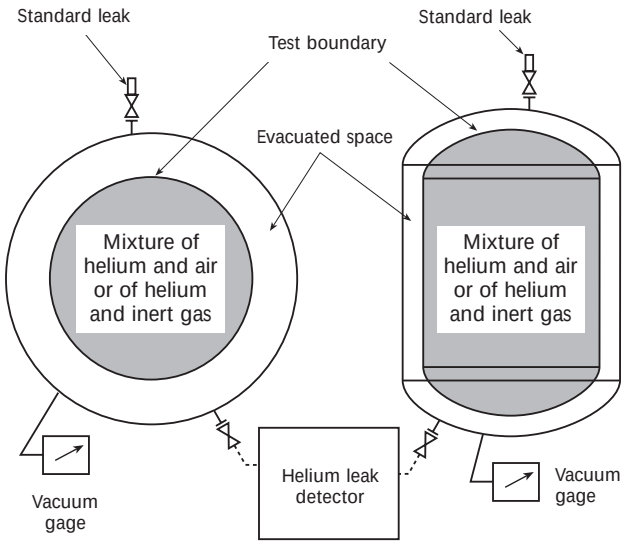
Refrigerant System Leakage Measurement by Helium Hood Accumulation

The procedure by this technique is to evacuate the component being tested to approximately 260 mPa (2 mtorr) by a mechanical vacuum pump. The pump is then removed and the component is placed under a helium filled hood for 5 to 10 min. During this period, if leaks are present, helium will be drawn into the internal volume of the component. After soaking under a helium hood, the component is attached to the leak detector by a quick connector.

The leak detector, incorporating a semiautomatic test port station, activates an audio alarm if the pressure in the unit under test indicates a gross leak. If the component passes the gross leak test, the operator switches the instrument to fine leak test with audio and visual no-go indication. The fine leak test allows acceptance or rejection of the component based on measurement of the accumulated partial pressure of helium inside.

To select a soak time sufficient to ensure a practical helium level for detection purposes, calculate the rate of helium partial pressure that will build up inside the component being tested at the leakage rate specified for rejection. From the partial pressure determination, a no-go point can be calculated based on a leak detector sensitivity in µmol·mol^{-1}. A typical detection level per division for helium is 0.1 µL·L^{-1}. With a manifold pressure of 25 mPa (0.2 mtorr), the partial

FIGURE 22. Helium accumulation leak testing of large double wall tanks and vessels.



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pressure detection limit for helium is 2.5 nPa (20 ptorr).

It is essential to ensure that equilibrium of helium has occurred inside coils, volume etc. of the unit before test or to correct for lack of equilibrium. An indication that equilibrium has not occurred exists when the calculated data do not correlate with operational failures. Slow diffusion of gas inside the unit may be caused by mechanical restrictions inside the unit such as capillaries, filters or long lengths of coils. Production test time for a single unit can be on the order of 15 s. Operator decision is eliminated because test limits are preestablished. The operator is required only to connect the leak detector to the unit being tested and initiate the test cycle switch. Alarm logic is energized automatically whenever the reject level is detected.

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PART 5. Detector Probe Technique for Leak Testing of Pressurized Objects[3,4]

Detector Probe Technique of Leak Testing with Mass Spectrometer

Figure 2 illustrates the technique of pressure testing with a helium detector probe. Pressure testing is a leak location technique during which helium tracer gas is introduced under pressure into the test object and is detected as it is emitted from a leak. The detector or sampling probe used in pressure testing is designed to collect tracer gas from a restricted area of the test object and feed it to the leak detector. The detector probe test, as used with a helium mass spectrometer detector, differs from sampling with other types of detectors primarily in that the mass spectrometer responds specifically to the helium tracer gas and is relatively uninfluenced by atmospheric contaminants such as paint fumes, solvents or cigarette smoke.

Techniques for Pressure Testing with Helium Leak Detector

With many types of vessels, it is necessary to use an internal pressure rather than a vacuum for conducting a leak test. This technique of testing is referred to as pressure testing. Such tests require introducing a tracer gas within the test object or using a mixture of the tracer gas and some other gas such as nitrogen, under a pressure greater than atmospheric. Under these conditions, the tracer gas will then issue from existing leaks. Detection of the leakage of tracer gas may then be accomplished by either detector probe or accumulation techniques. In the detector probe test, exterior areas suspected of leaking are explored for traces of helium with a sampling probe attached to the leak detector with a flexible hose, as shown in Fig. 11. The detector probe technique is for leakage location only.

This detector probe technique may be applied to vessels of any size or configuration. In most instances, it can be used on equipment during normal operations. Figure 11 shows an optimum connection of the sampling probe. Vinyl tubing or flexible metal tubing may be used to connect the detector probe to the helium leak detector. Rubber tubing should be avoided because it adsorbs helium and releases it over a prolonged time period, causing helium hang up. The probe hose should be as large in diameter as practical and as short as possible. The length of a vinyl probe hose should not exceed 4 m (12 ft) for optimum speed of response and cleanup. Special metal probe hoses may be longer. When large objects are pressure tested, it is preferable to use a short hose and to move the leak detector rather than to use a long hose and a stationary leak detector (see Fig. 12). Pumped probes with viscous flow permeable membranes coupling to the spectrometer tube can have hoses 15 m (50 ft) or longer.

Technique for Detector Probing for Leaks in Pressurized Vessels

After pressurizing a vessel under test, suspected exterior areas are then explored for traces of helium in the atmospheric air with a detector or sampling probe. The probe continuously samples the atmosphere adjacent to the external surface of the vessel and admits the sample to the leak detector. Minute leaks may be detected with exceptional precision by slowly moving the probe along suspected areas, such as welds, soldered joints or gasket connections.

Probing Procedure for Detector Probe Tests

After the probe line has been evacuated and valved into the leak detector, the operator proceeds to test by passing the detector probe slowly over suspected points of leakage. When sampling, technique is very important. Some of the factors that can affect the helium detector probe technique are listed as follows.

1. The sensitivity of the test will depend on the linear speed of the probe, on the distance of the probe tip from the surface being tested and on the pressure of tracer gas in the vessel. Degradation of leak testing sensitivity due to probing speed and distance are shown in Fig. 13. The sensitivity shown in Fig. 13 should be considered

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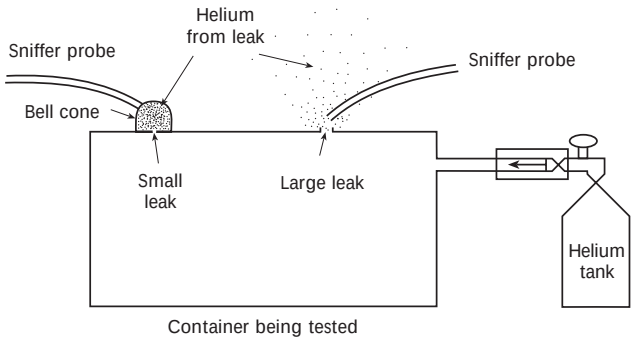
as only typical, because of variations in instruments, operator technique and other factors.

2. As in all leak testing, a nonreproducible leak indication may be due to a large leak in another location.
3. Advantage should be taken of the fact that the tracer gas concentration will build up around a leak. In view of this, the test object should be kept out of drafts.
4. The gas flow through the detector probe is very small. Nevertheless, increased sensitivity is obtained by restricting the slight vacuum to the immediate surface area under test. This can be done by putting a vinyl or hard rubber cone fitting on the end of the detector probe to act as a suction cup over the leak (see Fig. 23). In many cases, a small piece of tubing, just long enough to project past the end of the detector probe to enclose the test surface, will help considerably. Keep in mind that a long tubing connected to the detector probe in a conventional leak detector can introduce a long response time if the tracer gas is pumped into the detector probe by the flow through it. Time constants for detector probe tests with various lengths of 13 mm (0.5 in.) inside diameter tubing may be obtained from Fig. 12.
5. When the leak detector is tuned to a tracer gas in the atmosphere, a background indication will most likely be present when probing in air. In the case of helium, this reading is due to the normal concentration of helium in air (about 5 $\mu L \cdot L^{-1}$). This may be a magnitude that will decrease the ability to detect small leaks. Nulling out of the background signal due to atmospheric helium may be advantageous to achieve maximum detection sensitivity.

FIGURE 23. Confined small leak gives higher helium concentration than unconfined large leak.



Sensitivity of Detector Probing (Sniffing) Test

The sensitivity of pressure testing with a sampling probe and helium tracer gas will depend on the linear speed of the probe and on the distance of the probe tip from the surface being tested, as shown in Fig. 13. If the linear probing speed is 10 $mm \cdot s^{-1}$ (2 $ft \cdot min^{-1}$) and the tip of the probe is held 3 mm (0.125 in.) from the surface being tested, then the minimum leakage rate detectable would perhaps be in the order of 10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-6} std $cm^3 \cdot s^{-1}$). This, of course, is under ideal conditions and does not take into consideration such factors as air movement and varying temperatures. Fundamentally, the sensitivity of pressure testing with a sample probe is not as good as vacuum testing; the ratio is about 100 to 1 or higher. This is caused by dilution of the leaking helium by the atmospheric air. In vacuum testing no such dilution occurs. It must be understood that detector probe pressure testing is a qualitative leak test at best.

Pressurizing the Test Object for Helium Detector Probe Leak Tests

When probing, the detector probe test sensitivity may be improved by increasing the internal pressure of the tracer gas. This thereby increases the tracer gas out-leakage and results in an increase in the minimum detectable leak that can be found with a sample probe. The increase in out-leakage by viscous flow may be expressed by the relationship:

$$\textbf{(10)} \quad \frac{Q_2}{Q_1} = \frac{P_3^2 - P_2^2}{P_1^2 - P_2^2}$$

where Q_1 is out-leakage at lower differential pressure ΔP across leak opening; Q_2 is out-leakage at higher ΔP; P_1 is internal pressure inside vessel (at lowest ΔP); P_2 is total ambient pressure outside vessel; P_3 is internal pressure inside vessel (at highest ΔP).

If it is desired to use a pure helium internal pressure of several atmospheres and the test object is large, considerable amounts of helium will be used up in the test. This loss of helium can be avoided by pressurizing with a mixture of both helium and some other gas such as nitrogen. The test sensitivity will vary directly with the concentration of helium in the pressurizing gas. Thus, if a 10 percent helium and 90 percent nitrogen mixture is used, the test sensitivity will be 10 percent of that when pure helium is used at the same working pressure.

Fortunately, test sensitivity varies approximately as the square of the absolute pressure, for viscous flow leaks. It

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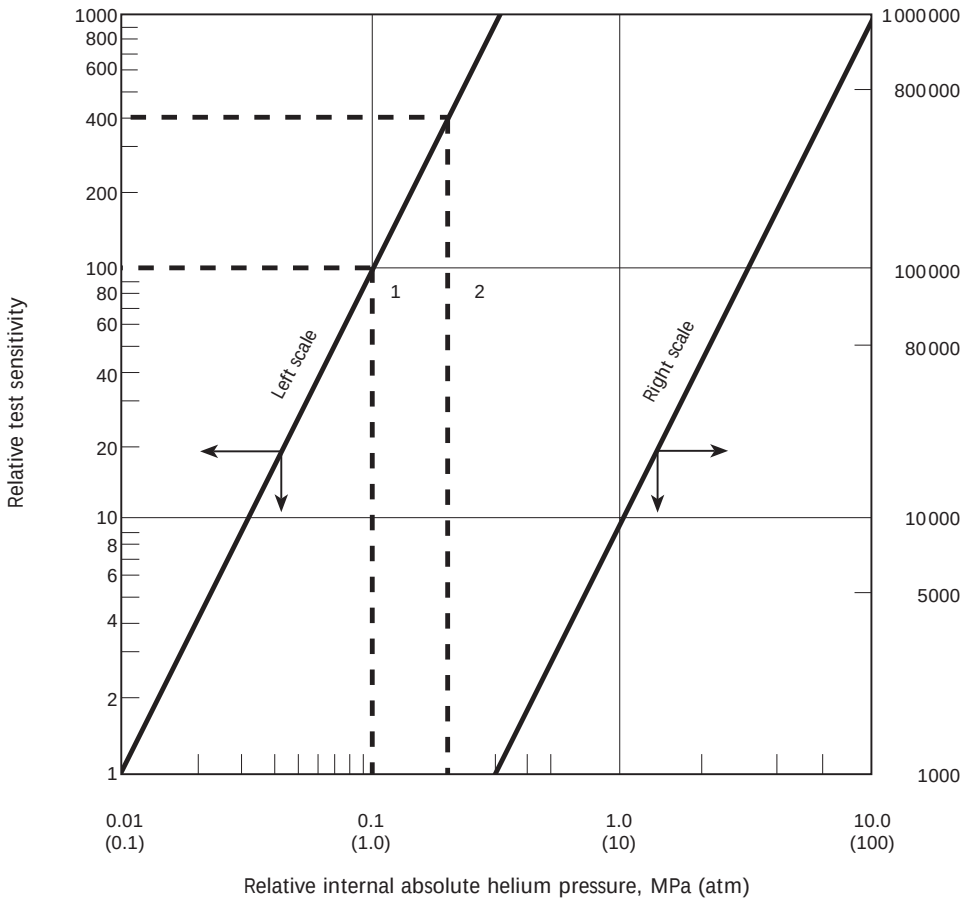
is possible to use this fact to great advantage, when pressurizing vessels, by working at reduced helium concentration and higher pressures. Because the helium consumed is a linear function of pressure whereas sensitivity is a square function, it is obviously more economical to use reduced helium concentration and increased total gas pressure.

Figure 24 illustrates the advantage of higher pressure. Consider the difference in test sensitivity of the probe to a 100 percent helium mixture at 0.2 MPa (30 $lb_f \cdot in.^{-2}$ absolute) to that of 10 percent helium and 90 percent nitrogen mixture at 0.9 MPa (130 $lb_f \cdot in.^{-2}$ absolute). The test advantage in sensitivity according to the curve is about 20 to 1 for 100 percent helium. But because a mixture of 10 percent helium is being used, the actual test sensitivity advantage will be 10 percent of 20, or 2 to 1. The test sensitivity has been doubled by using much less helium and raising the internal pressure of the vessel being tested.

System Calibration of Detector Probe Probe Helium Leak Test

The calibration of the helium leak detector for the detector probe test is extremely difficult to perform in a field application. As a consequence it is recommended that the leak detector be checked to ensure that the basic instrument is functioning properly, with the standard leak attached to the inlet manifold. After this has been accomplished, the detector probe and line may be attached. It is desirable to have available a source of helium to which the probe can be applied. A capsule capillary leak calibrated for atmospheric leakage works very well. An even better system calibration setup is the attachment of a capillary standard leak to the test system before pressurizing. With this, the detector probe can be checked to reassure the operator that it has adequate sensitivity.

FIGURE 24. Relative leak testing sensitivity in bell jar testing (pressure-vacuum leak testing) as a function of internal absolute atmospheres of 100 percent helium pressure in test objects, when outside of test object is placed in a high vacuum bell jar environment (SI units).



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Practical Capabilities and Limitations of Helium Detector Probe Leak Tests

The sensitivity of the helium detector probe leak test technique in a normal shop or field environment will enable detection of leakage rates in the range of 10^{-3} to 10^{-4} Pa·m^3·s^{-1} (10^{-2} to 10^{-3} std cm^3·s^{-1}) at a differential pressure of 100 kPa (1 atm) using helium mixtures of 2 to 5 percent by volume. Using the accumulation technique of bagging with polyethylene and rigidly controlling the factors affecting sensitivity, it is possible at a differential pressure of 100 kPa (1 atm) to detect much smaller leakage rates of 1×10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) using helium mixtures of 2 to 5 percent by volume. The increase of sensitivity obtainable by bagging and accumulation depends directly on time of accumulation. Quantitative sensitivity (total leakage rate) for an entire large test system is very difficult to determine with any accuracy by this test technique.

Leakage Sensitivity Attainable with Helium Detector Probes

The detector probe system responds essentially to changes in the ambient concentration of helium in the immediate vicinity of the probe. The original concentration of helium in the atmosphere with no leak is about 0.0005 percent. Commercial leak detectors are generally capable of detecting a change in helium concentration at this level of at least 0.0005 percent (by zeroing out the background). This sets the lower limit of detectability.

Once the helium concentration in the vicinity of the probe produces a 100 percent or full scale leak indication, no further increase in signal will be seen on the output meter. Quantitative calibration of the detector probe technique is therefore very difficult. It is apparent that even a small leak, leaking into a relatively small and confined volume, can produce as high a helium concentration as a much larger leak leaking into an unconfined volume (Fig. 23). Of course, if test conditions are identical, the rate of concentration increase will be faster with the large leak. It is also apparent that drafts disperse the leaking helium and dilute it even close to its source. This makes it difficult to detect large leaks and impossible to find small ones when operating in drafty environments.

Scanning Speed and Accumulation Times for Helium Detector Probes

The mass spectrometer instrument's response time is usually the determining factor in setting probe speed. A typical continuous scanning probe speed for helium leaking to atmospheric air is 1 cm·s^{-1} (2 ft·min^{-1}). A more sensitive accumulation technique requires the intermittent motion of a detector probe whose collecting tip penetrates inside a small rubber bell placed against the test surface (Fig. 23). After the probe has been held in a fixed location for an accumulation period of 30 s to 15 min, the probe assembly is moved to cover another test area, overlapping the first. The difference in sensitivity between leakage of helium into the open air and leakage of helium into a small enclosure containing the detector probe can be as great as 100 000 to 1.

Variables Influencing Sensitivity of Helium Detector Probe Tests

Basically, helium mass spectrometers are designed for leak testing under vacuum conditions. When used as detector probe instruments for leak testing in air, the sensitivity is much less than their vacuum leak detection sensitivity due to the following variable factors involved in helium detector probe tests: (1) technique and experience of the operator; (2) differential pressure across the test boundary; (3) helium gas concentration within the test boundary; (4) linear scanning speed of the detector probe; (5) distance the detector probe is held from the test surface; (6) length and diameter of flexible detector probe hose; (7) pressure in the sensing element of the helium mass spectrometer; (8) helium background in the air caused by buildup from excessively large helium tracer gas leaks in the test boundary; (9) weather conditions that adversely affect test results, i.e., strong winds that dilute and disperse the helium tracer gas passing from leaks in the test boundary; (10) conventional versus counterflow leak detector; and (11) pumped versus nonpumped detector probe.

Determining Pressure and Volume Factors Affecting Sensitivity of Helium Detector Probe Tests

The following information shows the relationship between the first seven factors in the preceding list of variables that affect the sensitivity of the helium detector probe leak test.

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Determining Helium Concentration or Partial Pressure. To determine helium concentration or helium partial pressure when either one of these two values and the test pressure are known or specified, use the following:

$$\textbf{(11)} \quad \%C_p = \frac{P_h}{P} \times 100$$

$$\textbf{(12)} \quad P_h = \frac{\%\, C_p\, P}{100}$$

where percent C_p is helium concentration in percent by volume; P_h is helium partial pressure (kilopascal or $lb_f \cdot in.^{-2}$ absolute); P is absolute total test pressure (kilopascal or pound per square inch absolute).

For example, a vessel specification requires a 10 percent by volume helium concentration for a 300 kPa (50 $lb_f \cdot in.^{-2}$ gage) helium detector probe test. What helium partial pressure is required to attain this required helium concentration? Using Eq. 11 and assuming atmospheric pressure is 100 kPa absolute (14.7 $lb_f \cdot in.^{-2}$ absolute), then helium partial pressure is:

$$P_h = \frac{(10)(300 + 100)}{100} = 40 \text{ kPa}$$

Determining Helium Quantity Required When Concentration and Volume Are Known. To determine the quantity of helium required when helium concentration and test boundary volume are known and/or specified, use Eq. 13:

$$\textbf{(13)} \quad V_h = \frac{\%C_p V}{100} \frac{P}{P_a}$$

where V_h is volume of helium (cubic meter or cubic foot); V is test boundary volume (cubic meter or cubic foot); P_a is atmospheric pressure (kilopascal); P is test gas absolute pressure (kilopascal); percent C_p is helium concentration (percent) by volume.

For example, what is the quantity of helium required to attain a 5 percent by volume concentration of tracer for a helium detector probe test of a 500 m^3 (2×10^4 ft^3) vessel at 200 kPa (30 $lb_f \cdot in.^{-2}$ gage)? By using Eq. 13 and assuming atmospheric pressure = 100 kPa (14.7 $lb_f \cdot in.^{-2}$ absolute), it is found that the helium volume is:

$$V_h = \frac{(5)(500)}{100} \frac{(200 + 100)}{100} = 75 \text{ m}^3$$

$$\left(= \frac{(5)(20\,000)}{100} \frac{(30 + 14.7)}{14.7} = 3040 \text{ ft}^2 \text{ gage} \right)$$

To determine the quantity of helium required when helium partial pressure P_h and test boundary volume V and atmospheric pressure P_a are known and/or specified, use Eq. 14:

$$\textbf{(14)} \quad V_h = \frac{P_h}{P_a} V$$

For example, what quantity of helium is required to pressurize a 300 m^3 (1×10^4 ft^3) vessel to 70 kPa (10 $lb_f \cdot in.^{-2}$ gage)? Using Eq. 14 and assuming atmospheric pressure = 100 kPa (14.7 $lb_f \cdot in.^{-2}$ absolute), the helium volume is:

$$V_h = \frac{(70)(300)}{100} = 210 \text{ m}^3$$

$$\left(= \frac{10}{14.7}(10\,000) = 6800 \text{ ft}^3 \right)$$

Note that, because atmospheric pressure is 100 kPa (1 atm), partial pressures can be considered as percentages. In this case, 70 percent of 300 = 210 m^3 (275 yd^3).

Effect of Increasing Length of Detector Probe Hose

When the detector probe hose is increased in length, the signal response time increases proportionately. Because of the increased volume, the sensitivity decreases approximately in inverse proportion to increase in hose length l.

$$\textbf{(15)} \quad (\text{sensitivity})_2 = \frac{l_1}{l_2} (\text{sensitivity})_1$$

Effect of Adding Carrier Gas in Detector Probe Line

The principle involved in a fast response probe is simple. A carrier gas, specifically carbon dioxide, is injected into the line near the detector probe inlet, thus increasing the pressure in the connecting line. This increased pressure changes the nature of flow from molecular (or transitional) to viscous. This drastically increases the conductance of the line from the detector probe inlet to the leak

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detector. The carrier gas is selectively pumped by the liquid nitrogen trap in the mass spectrometer leak detector at a rate of several thousand $L \cdot s^{-1}$ so that the pressure in the mass spectrometer is 1000 times less than in the detector probe line. In addition, the carrier gas also acts to sweep the line, thereby eliminating helium hangup and high background caused by prior testing. The system permits long lengths of line without sacrificing response time or cleanup time.

Selection of Optimum Gas Pressure in Sensing Element of Spectrometer

For conventional leak detectors an instrument sensing element pressure of 10 mPa (0.1 mtorr) should be used for maximum leak testing sensitivity. This mass spectrometer pressure can go as high as 25 mPa (0.2 mtorr); however, higher pressure in the sensing element for sustained periods of helium detector probe testing shortens filament life considerably, necessitating frequent filament replacements and delays. Lowering the sensing element pressure will reduce sensitivity because sensitivity varies directly with sensing element pressure. Variation in this relationship can exist due to inaccuracy in the sensing element pressure gage.

In counterflow leak detectors, pumping speed at the leak detector test port connection is constant and does not affect spectrometer sensing element pressure unless the maximum forepressure of the diffusion pump exceeds the manufacturer's specification.

Response Time of Helium Mass Spectrometer Leak Detectors

An additional serious consideration is the response time of the helium leak detector. Response time is the time for a leak detector or leak testing system to yield a signal output equal to 63 percent of the maximum signal attained when tracer gas is applied for an indefinitely long period to the detector probe. The total response time is determined by a combination of (1) response time of the circuitry of the mass spectrometer (usually 0.5 to 2 s); (2) mass spectrometer vacuum system response time (which decreases with increasing pumping speed and increases with greater lengths of detector probe hose); and (3) response time related to flow of tracer gas through the detector probe capillary orifice and tubing.
The net response time for a conventional leak detector with 3 m (10 ft) of hose may be 5 to 15 s but as low as 2.55 for a counterflow leak detector.

Counterflow leak detectors with a pumped 15 m (50 ft) hose can exhibit response times of less than 5 s. This allows the testing of very large vessels that do not permit use of short probe hoses.

Response Time of Actual Leaks in Test Object

An additional response time to consider is that of the leak itself. The time one must wait after pressurization or evacuation before searching for the leak is a function of the nature of the leak. With a direct leak passage such as a scratch or a hair across a gasket, the waiting time will be short. However, the delay may be very long if the leakage path consists of a tiny passage leading to a cavity and then to the other side or of multiple cavities, as shown in Fig. 6. This is often the case in castings or in joints welded on both sides.

Example Procedure for Helium Leak Testing by Remote Sampling

Remote sampling is a useful supplement to the helium detector probe test where test areas are hard to get at or cramped and can only be probed using an excessively long flexible hose between the sampling probe and the helium mass spectrometer. If properly performed, this sampling technique makes it possible to test these areas to optimum sensitivity without additional hose for the sampling probe.

Assume that a helium detector probe test is being conducted with the helium mass spectrometer in operation and the vessel pressurized with a helium-air mixture. The test area where leaks are suspected can be enclosed in a polyethylene or plastic bag (squeeze as much as from the bag as possible before sealing). The bag is then left intact for a sufficient time to allow buildup of helium concentration (from possible leaks) within the sealed bag. This provides an accumulation technique to increase the partial pressure of helium in the plastic bag.

A sample of gas is then removed from the sealed bag using either a hypodermic needle or a small evacuated valved container (such as a piece of pipe capped on both ends with a valved pipe nipple in one end). If a hypodermic needle is used with the plunger in, pierce the bag and withdraw the plunger to remove the gas sample. If a small evacuated container is used, insert the container connection through a hole cut in the bag and open the valve to remove the gas sample.

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The gas sample is then inserted into the helium mass spectrometer system as follows. If a hypodermic needle is used, the sampling probe hose should be removed from the instrument manifold (follow necessary steps with instrument) and the end of the manifold sealed with a tight rubber diaphragm. When the manifold has once again been evacuated and the instrument is ready for testing, the rubber diaphragm should be pierced with the hypodermic needle. The sample in the needle will be slowly pulled into the system and any helium in the sample will be indicated by the instrument. If a small evacuated container is used, connect it to the instrument manifold with a very short length adapter. After the manifold is evacuated, keep a check on the instrument sensing element pressure and crack the sample container valve slightly to allow the sample to be slowly pulled into the instrument where any helium present in the leakage sample will be indicated.

Whenever a sample is first introduced to the system, the instrument indicator reading will rise suddenly due to the sudden slight increase in pressure caused by the initial intake of the sample. If helium is present, the instrument indicator should return to its initial reading or become steady within a few minutes.

Direct Probing of Leaks to Atmosphere

The direct probing technique (see Fig. 25) is the simplest test and may be used on parts of any size. It requires only that a tracer gas pressure be created across the area to be tested and the searching of the atmospheric side of the area with the detector probe. This technique detects leakage and locates leaks. Experience has shown that probe testing in factory environments will usually be satisfactory to 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}), if reasonable precautions against releasing gas like the tracer gas in the test area are observed and if the effects of other interference are considered.

Apparatus and Materials Required for Direct Probing Technique

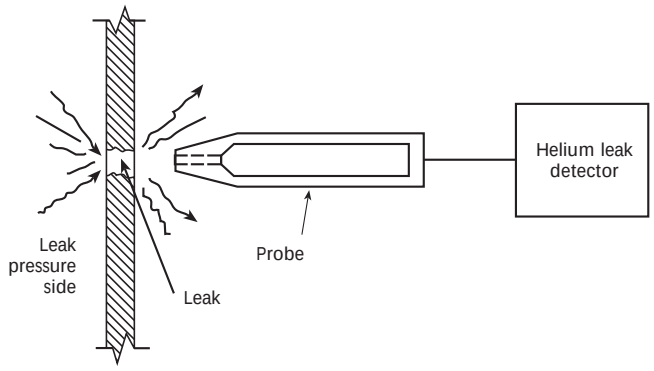
The following items constitute the equipment and materials required for testing of helium pressurized test objects leaking to air at atmospheric pressure (see Fig. 25): (1) test specification; (2) helium leak detector, with atmospheric detector or sampling detector probe; (3) helium leak standard, discharge to atmosphere — size equal to or as near as possible to helium content of maximum leakage rate per specification, taking helium concentration into account; (4) helium leak standard, discharge to vacuum — size: anywhere between 1×10^{-7} Pa·m^3·s^{-1} (1×10^{-6} std cm^3·s^{-1}) and 1×10^{-10} Pa·m^3·s^{-1} (1×10^{-9} std cm^3·s^{-1}), unless otherwise specified by the maker of the leak detector; (5) test gas at or above specification pressure; (6) pressure gages, valves and piping for introducing test gas and, if required, vacuum pump for evacuating device; and (7) liquid nitrogen if required.

Procedure

The following steps constitute the leak testing procedure with helium pressurized test objects leaking to air at atmospheric pressure (see Fig. 25).

1. Set helium leak standard leakage rate to maximum allowable per specification. For example, if maximum leak rate is 1×10^{-5} Pa·m^3·s^{-1} (1×10^{-4} std cm^3·s^{-1}) and test gas is 1 percent helium in air, set standard at $1 \times 10^{-5} \times 0.01 = 1 \times 10^{-7}$ Pa·m^3·s^{-1} (1×10^{-6} std cm^3·s^{-1}).
2. Start detector and adjust in accordance with a manufacturer's instructions.
3. Attach atmospheric detector probe to detector sample port in place of leak standard and open valve of detector probe, if adjustable type is being used, to maximum flow rate under which a detector will operate properly.
4. Rezero detector to compensate for atmospheric helium.
5. With orifice of leak standard in a horizontal position, hold the tip of the detector probe directly in line with and 1.5 ± 0.5 mm (0.06 ± 0.02 in.) away from the end of the orifice and observe reading while scanning past the orifice at a normal rate of about 2 cm·s^{-1} (4 ft·min^{-1}). If necessary to obtain a reasonable instrument

FIGURE 25. Direct probing technique with sampling probe or sniffer on test objects leaking to air at atmospheric pressure. Note that probe does not detect all of the leakage.



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deflection, adjust range, rezero if necessary and again apply sampling probe to leak standard.
6. Remove probe from standard leak and note minimum and maximum readings due to atmospheric helium variations or other instabilities.
7. If atmospheric helium variation is larger than 30 percent of standard leak indicator, take steps to reduce the helium added to the atmosphere or to eliminate other causes of instability. If this cannot be done, testing at this level of sensitivity may not be practical.
8. Evacuate (if required) and apply test gas to device at specified pressure.
9. Probe areas suspected of leaking. Probe should be held on or not more than 1 mm (0.04 in.) from the surface of the device and moved not faster than 20 mm·s^{-1} (0.8 in.·s^{-1}). If leaks are located that cause a reject leakage indication when the probe is held over 1 mm (0.04 in.) from the apparent leak source, repair all such leaks before making final acceptance test.
10. Maintain an orderly, bottom-to-top procedure in probing the required areas, preferably identifying them as tested and plainly indicating points of leakage.
11. After the test, evacuate or purge test gas from the device, if required.
12. Write a test report or otherwise indicate test results as required.

Interfering Effects

The atmosphere contains about 5 µL·L^{-1} of helium, which is being continuously drawn in by the detector probe. This helium background must be zeroed out before leak testing using helium tracer gas can proceed. Successful leak testing is contingent on the ability of the detector to discriminate between normal atmospheric helium, which is very constant, and an increase in helium due to a leak. If the normally stable atmospheric helium level is increased by release of helium in the test area, the reference level becomes unstable, making leak testing more difficult.

Helium absorbed in various nonmetallic materials (such as rubber or plastics) may be released during the test. If the rate and magnitude of the amount released approaches the amount released from the leak, the reliability of the test is decreased. The amount of such materials or their exposure to helium must then be reduced to obtain a meaningful test.

To evaluate leakage accurately, the test gas in all parts of the device must contain substantially the same amount of tracer gas. When the device contains air before test gas is introduced or when an inert gas and a tracer gas are added separately, this may not be true. Devices in which the effective diameter and length are not greatly different (such as tanks) may be tested satisfactorily by simply adding tracer gas. However, when long or restricted systems are to be tested, more uniform tracer distribution will be obtained by first evacuating to about 1 kPa (several torr) and then filling with the test gas. The test gas must be premixed if not 100 percent tracer.

As the orifice in the detector probe is very small, the parts being tested should be clean and dry to avoid plugging of the detector probe orifice. Reference should be frequently made to a standard leak to ascertain that this has not happened. However, plugging causes the pressure in the sensing element of the helium leak detector to decrease significantly, which should alert test operators to the possibility of plugging.

Test Apparatus Required for Helium Leak Testing in Detector Probe Mode

Test apparatus for helium leak testing in the detector probe mode includes a helium mass spectrometer analyzer, calibrated leaks and test fixtures. The helium leak detector should be equipped with an atmospheric detector probe and be adjusted for testing with helium. The helium leak detector should meet the following minimum requirements.

The sensor mass analyzer should have a panel instrument or digital readout and a sensitivity on its most sensitive range of 1×10^{-8} Pa·m^3·s^{-1} (1×10^{-7} std cm^3·s^{-1}) full scale. The response time should be 3 s or less. The required instrument stability and sensitivity should result in maximum variation not exceeding ± 5 percent of full scale on most sensitive range while the probe is active and only sensing atmospheric helium. A maximum variation of ± 2 percent of full scale on other ranges should be attained over a period of 1 min. The instrument should provide a range control preferably in steps of about 3× and a zero control having sufficient range to null out atmospheric helium background signals.

Requirements for Leak Standards for Helium Leak Testing

To perform leak tests two types of helium leak standards are used that should meet the following minimum requirements.

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1. The helium leak standard calibrated for discharge to atmosphere should have ranges of 1×10^{-3} to 1×10^{-7} Pa·m^3·s^{-1} (1×10^{-2} to 1×10^{-6} std cm^3·s^{-1}).
2. The helium leak standard calibrated for discharge to vacuum should have ranges from 1×10^{-7} to 1×10^{-10} Pa·m^3·s^{-1} (1×10^{-6} to 1×10^{-9} std cm^3·s^{-1}).
3. Accuracies of helium leak standards should be ± 10 percent.
4. Adjustable leak standards are convenient but not mandatory.
5. The temperature coefficient of leak standards should be stated by the manufacturer.

Requirements for Tracer Gas and Gas Mixtures

To be satisfactory, the test gas should be nontoxic, nonflammable, inexpensive and not detrimental to common materials. Helium meets the requirements, as does helium mixed with air or nitrogen, or helium mixed with some other suitable inert gas. If the test specification for maximum allowable leakage is 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}) or more or if large vessels are to be tested, consideration should be given to diluting the tracer gas with another gas such as dry air or nitrogen. This will avoid excessive helium input to the sensor and save tracer gas expense in the case of large vessels. When a vessel is not evacuated before adding test gas, the gas mixture is automatically diluted by 100 kPa (1 atm) of air.

Producing Premixed Test Gas

If the volume of the device or the quantity to be tested is small, premixed gases in cylinders can be obtained conveniently. Continuous gas mixing using calibrated orifices is another simple and convenient technique when the test pressure does not exceed 50 percent of the tracer gas source pressure available.

Calibration during Helium Leak Testing

The leak detectors used in leak testing with helium tracer gas are not calibrated in the sense that they are taken to the standards laboratory, calibrated and then returned to the job. Rather, the leak detector is calibrated to a standard leak for reference and is then used to measure the unknown leak. However, the sensitivity of the leak detector is checked and adjusted on the job so that a leak of specified size will give a readily observable, but not offscale reading. More specific details are given next under the test technique being used. To verify sensitivity, leak test equipment should be checked with a standard leak before and after a prolonged leak test. When rapid repetitive testing of many items is required, the leak standard is referred to often enough to ensure that desired test sensitivity is maintained.

Specifications for Helium Leak Testing

A testing specification should be in hand. This specification should include (1) the gas pressure on the high side of the device to be tested, also on the low side if it needs to differ from atmospheric pressure; (2) the test gas composition, if there is need to specify it; (3) the maximum allowable leakage rate in Pa·m^3·s^{-1} (std cm^3·s^{-1}); (4) whether the leakage rate is for each leak or for total leakage of the device; and (5) whether or not surface areas other than seams, joints and fittings need to be tested.

Safety Factor in Specified Leakage Rates

Where feasible, it should be ascertained that a reasonable safety factor has been allowed between the actual operational requirements of the device and the maximum specified for testing. Experience indicates that a safety factor of at least 10 should be used when possible. For example, if a maximum total leakage rate for satisfactory operation of a device is 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}), the leak test requirement should be 1×10^{-7} Pa·m^3·s^{-1} (1×10^{-6} std cm^3·s^{-1}).

Disposition or Recovery of Test Gas

Test gas should never be released into the test area if further testing is planned. It should be vented outdoors or recovered for reuse if the volume to be used makes this worthwhile.

Detrimental Effects of Helium Tracer Gas

Helium tracer gas is quite inert and seldom causes any problems with most materials, particularly when used in gaseous form for leak testing and then removed. When there is a question as to the compatibility of the tracer with a particular material, an authority on the material should be consulted. This is particularly true when helium is sealed in contact with glass or other barriers that it may permeate.

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Correlation of Test Gas Leakage with Leakage of Other Gases or Liquids at Different Operating Pressures

Given the normal variation in leak geometry, accurate correlation between a measured leakage rate and that for other gases or pressure is impossible. However, if a safety factor of 10 or more is allowed, adequate correlation for gas leakage within these limits can usually be obtained by assuming viscous flow of gas and using the relation:

$$\textbf{(16)} \quad Q_2 = Q_1 \frac{n_1}{n_2} \frac{P_2^2 - P_1^2}{P_4^2 - P_3^2}$$

where Q_2 is test leakage rate, $Pa \cdot m^3 \cdot s^{-1}$ (or std $cm^3 \cdot s^{-1}$); Q_1 is operational leakage rate, $Pa \cdot m^3 \cdot s^{-1}$ (or std $cm^3 \cdot s^{-1}$); n_2 is viscosity of the test gas, n_1 is viscosity of operational gas; P_2, P_1 are absolute pressure on high and low sides at test; P_4, P_3 are absolute pressures on high and low sides in operation.

Viscosity differences between gases are a relatively minor effect and can be ignored if desired. Leakage increases at a rate considerably with pressure increase. For this reason, it is often desirable to increase the sensitivity of the test by testing at the maximum safe pressure for the part. Increased sensitivity may even be obtained with the same amount of helium by increasing the pressure with another less expensive gas, as when pressurizing with air.

Experience has shown that, at the same pressures, gas leaks smaller than 1×10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-5} std $cm^3 \cdot s^{-1}$) will not show visible leakage of a liquid, such as water, which evaporates fairly rapidly. For slowly evaporating liquids, such as lubricating oil, the gas leakage should be another order of magnitude smaller, 1×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-6} std $cm^3 \cdot s^{-1}$).

Helium Pressure Testing of Large Vessels with Detector Probe

With many types of vessels, it is necessary to use internal pressure for conducting a leak test. Such tests require pressurizing the vessel with helium or a mixture of helium and air. Suspected exterior areas such as welds and joints are then explored for traces of helium with a detector probe connected to the leak detector. The probe continuously sniffs the atmosphere adjacent to the external surface of the vessel near a suspected leak. Any helium leaking from the vessel is admitted, at optimum pressure, to the helium mass spectrometer leak detector. Minute leaks can be detected by this technique. The probe-to-leak-detector connection is made through a flexible vinyl hose. The length of the detector probe hose should not be greater than about 4 m (12 ft) or the resultant response and cleanup time for the system will be poor. This does not apply, however if a pumped probe is used with a counterflow leak detector

Effect of Probing Techniques on Sensitivity of Detector Probe Leak Testing

Two factors affecting the sensitivity of the leak test are the linear speed of the probe and the distance of the probe tip from the surface being tested (see Fig. 13). In general, the sampling probe should not be moved at speeds of more than 5 $mm \cdot s^{-1}$ (1 $ft \cdot min^{-1}$). The distance of the probe tip from the vessel surface can be maintained constant by using a small rubber sleeve over the probe tip. The end of the sleeve is held directly against the surface being tested. Another benefit resulting from this procedure is that the slight suction through the detector probe is restricted to the neighborhood of the joint under test.

To obtain maximum detector probe leak testing sensitivity, it is necessary to open the probe as far as possible. However, because of the hot filament used in the spectrometer tube, it is not wise to exceed a manifold pressure of 25 mPa (0.2 mtorr) in conventional mass spectrometer leak detectors.

This pressure limitation is not usually a problem with counterflow leak detectors because of their ability to test at pressures up to 5000 times greater than conventional leak detectors without exceeding the 25 mPa (0.2 mtorr) limit. Pressures much above 25 mPa (0.2 mtorr) can produce a nonlinear increase in sensitivity due to mean free path limitations. However, the sensitivity of pressure testing is not as good as the sensitivity in vacuum testing, the ratio being about 1000 to 1 or greater. This is because the outflowing helium is diluted by the atmospheric air (see Fig. 25). In vacuum testing no such dilution occurs.

Pressurizing Large Vessels with Helium Mixtures

Sensitivity varies directly with the concentration of helium in the pressurizing gas. Thus, if a 10 percent helium, 90 percent air mixture is used, the leak testing sensitivity will be 10 percent of that for pure helium at the same working pressure. Fortunately, sensitivity

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FIGURE 26. Relative test sensitivity in bell jar testing in pressure-vacuum leak testing as a function of internal 100 percent helium pressure in test object, when outside of part is at 100 Pa (1 atm) bell jar pressure.

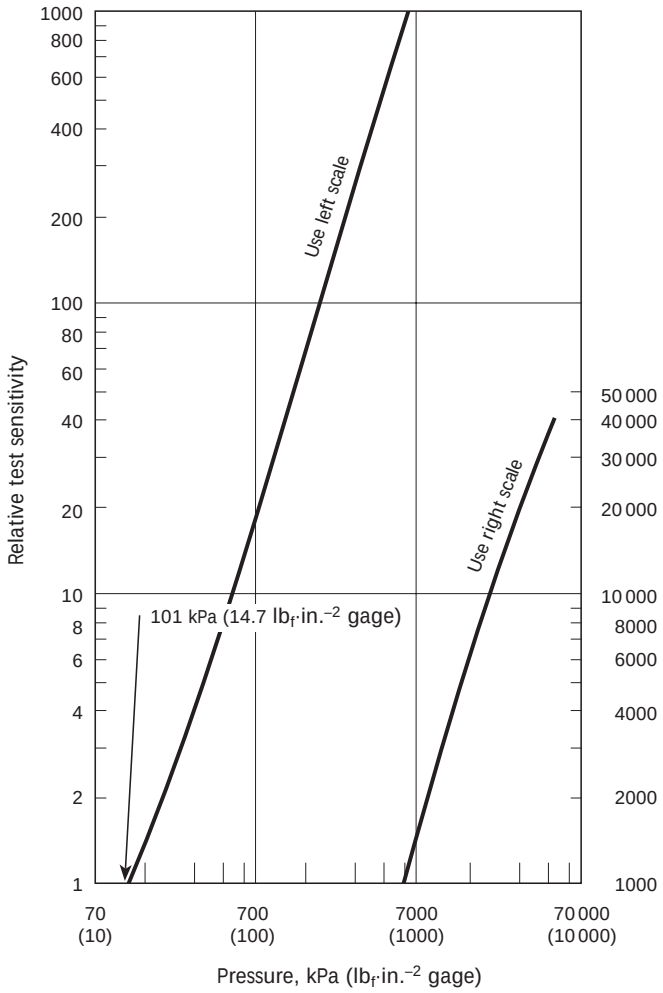
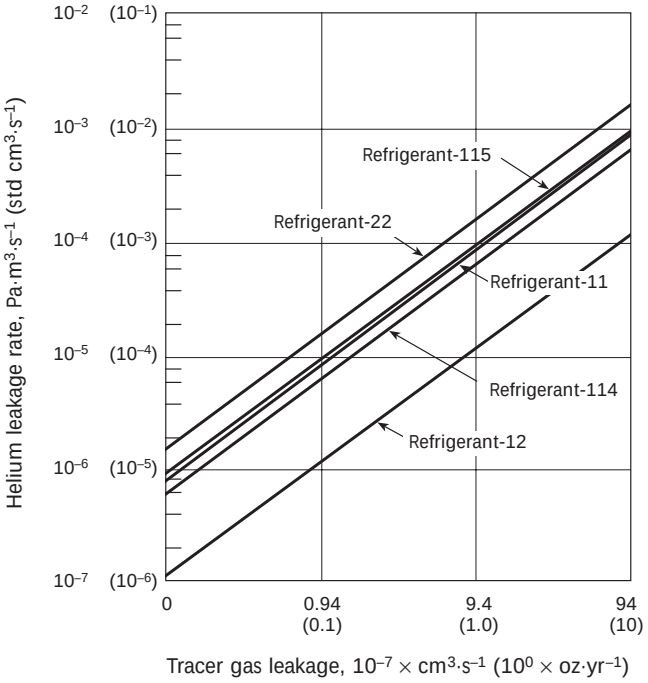


FIGURE 27. Conversion chart relating helium leakage rates to refrigerant gas tracer leakage rates in ounces per year.



also varies approximately as the square of the absolute pressure (with viscous flow above atmospheric pressure). This fact can be used to advantage by working with reduced helium concentrations and higher pressures. Thus, because the percentage of helium used is a linear function whereas sensitivity is a square function of pressure in the leak path, the desired sensitivity can be economically achieved by using reduced concentration and increased pressure.

Obviously, it is desirable to establish the approximate maximum leak that can be tolerated. Knowing this figure and assuming an operating pressure above equivalent to the designed working pressure of the vessel, the user of the sampling probe may then determine from the curves of Fig. 26 the concentration of helium needed in the pressurizing mixture for the required sensitivity.

Selection and Control of Leak Test Pressure

The device should be tested at its design operating pressure with the pressure drop in the normal direction, where practical. Precautions should be taken so that the device will not fail during pressurization and so that the operator is protected from the consequences of failure.

Detection of High Pressure Leaks in Large Welded Vessels

An additional advantage of pressurizing a vessel to the designed working pressure is the detection of high pressure leaks. Experience has shown that deformation in the walls of a fabricated vessel often causes the opening of leaks to expand under high pressure. These leaks may close again at lower pressures. Welded joints may contain leaks having very tortuous paths.

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Techniques for Helium Leak Testing in Refrigeration Equipment

Several techniques of leak testing using the helium mass spectrometer leak detector are used for testing refrigeration systems and components. Manufacturers of all sizes and configurations of refrigeration and air conditioning components and systems have stipulated that refrigerant gas leakage should not exceed 1 oz in 10 years. This leakage rate, when converted to an equivalent helium leakage, is approximately 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) under similar pressure conditions. Figure 27 is a chart for converting helium leakage rates to equivalent rates of refrigerant gas leakage in ounces per year.

The detector probe technique can be used to locate individual leaks without using enclosures. It requires pressurization of the component or system with helium, or mixing it with another gas such as nitrogen or even air in known quantities. Care should be taken, however, to ensure that the concentration of helium is at least 100 times greater than the abundance of helium in the air.

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PART 6. Bell Jar Technique for Leak Testing of Pressurized Objects[5]

Bell Jar Technique for Leak Testing of Sealed Components Containing Helium

A very sensitive helium leak test can be performed when testing relatively small sealed units for overall leakage. Components to be tested are filled under slight pressure with helium or a mixture of helium and another gas. They are then sealed and placed in a vacuum chamber (bell jar) that is evacuated by an auxiliary pump system to which the leak detector is connected (see Fig. 4). Helium leaking from the sealed units into the evacuated chamber is detected almost immediately. This type of testing has proved very satisfactory when leak testing hermetically sealed components such as relays, switches and integrated circuit packages.

Technique for Helium Leak Testing of Sealed Components

Leak testing of sealed components pressurized with helium involves a combination of pressure and vacuum testing. Hermetically sealed devices whose enclosures are filled with helium or with a tracer gas containing some percentage of helium can be rapidly leak tested with high sensitivity by placing them within a bell jar as sketched in Fig. 4 or within a metallic enclosure as shown in Fig. 15. The bell jar or other enclosure is then evacuated and its interior volume is connected to the helium mass spectrometer leak detector. If a helium indication results it is evidence that a leak exists in the sealed device under test.

Equipment for Bell Jar Leak Testing of Sealed Components

For helium leak testing of sealed components, the bell jar or test enclosure should have a minimum free volume so as to shorten the pumpdown time. Frequently, specially built metallic enclosures are preferable to standard glass bell jars. The seal between the bell jar or cover unit and the vacuum plate is usually made by means of a gasket. When sealing a bell jar to a vacuum plate, a minimum amount of vacuum grease should be used on the seal, or helium hangup will result when the grease first absorbs the helium it entraps and then later releases it.

An automatic test port may be used in the bell jar technique of testing. This type of test is simple and straightforward. With a flick of the switch the operator can read the total leakage of the part being tested for a go/no-go test. All parts that fail this go/no-go test may subsequently be subjected to probing, so that the leaks may be pinpointed and repaired.

Leak Testing Sensitivity in Bell Jar Testing

In bell jar testing, just as in pressure testing, an increase in test sensitivity may be achieved by increasing the internal pressure of the part under test. The test sensitivity will vary directly with the concentration of helium in the pressurizing gas. Figures 26 and 28 show the advantage of this procedure in bell jar testing. Compare the leak test sensitivity with 100 percent helium at 100 kPa (1 atm) with that of 100 percent helium at 200 kPa (2 atm) pressure. The test sensitivity advantage, according to the curve on Fig. 24, is 4 to 1. With molecular flow leaks, the advantage would be only 2 to 1. It can be seen that the test sensitivity has increased by the square of the tracer gas pressure, for the case of viscous flow leaks only.

Now consider the difference in the test sensitivity of 100 percent helium at 100 kPa (1 atm) pressure and a 10 percent helium and 90 percent nitrogen mixture at 500 kPa (5 atm) pressure. The leak test sensitivity advantage for viscous flow leaks due to the increase in pressure, according to Fig. 24, is about 25 to 1 for 100 percent helium gas. However, when use is made of a mixture of 10 percent helium, the actual test sensitivity advantage will be 10 percent of 25, or 2.5 to 1. It can be seen that the test sensitivity has been more than doubled by using less helium and raising the internal pressure of the object being tested. Figure 24 applies when the bell jar is highly evacuated. Figure 26 applies if the interior of the bell jar is at atmospheric pressure (100 kPa).

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Pumpdown Time for Helium Bell Jar Leak Testing

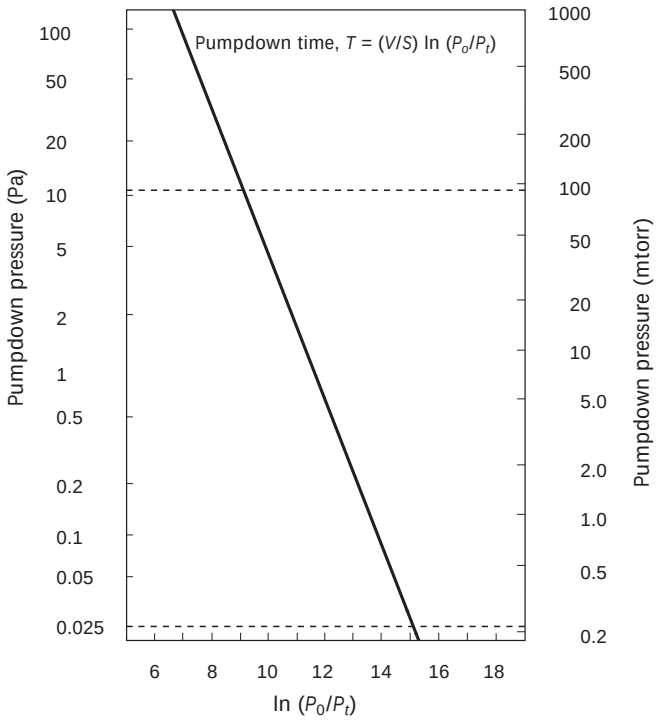
When setting up a bell jar test, one important consideration is the time required to evacuate the bell jar to a pressure low enough to allow it to be connected to the leak detector. Pumpdown time may be determined from the graph of Fig. 28, showing the pressure to which the bell jar must be pumped down as a function of the natural logarithm of the pressure ratio P_o/P_t. The pumpdown equation gives pumpdown time:

$$\textbf{(17)} \quad T = \frac{V}{S} \ln \frac{P_o}{P_t}$$

where V is the free volume of the bell jar and S is the effective pumping speed on the volume, assuming the pumping speed is constant. Usually the desired time is the time required to pump down to 25 mPa (0.2 mtorr) because that is the maximum operating pressure of the conventional flow mass spectrometer leak detector. From Fig. 28, it can be seen that the time will be close to 15.2(V/S). Another handy relationship is that the pressure will drop by a factor of 10 in 2.3(V/S) units of time.

The counterflow leak detector can tolerate testing pressures of at least 10 Pa (0.1 torr). The pumpdown time will be much shorter.

FIGURE 28. Pressure to which bell jar must be pumped down as a function of pressure ratio, in vacuum leak testing.



Response Time for Helium Bell Jar Leak Testing

In bell jar testing, helium flow through a leak is usually well established during the time required to evacuate the bell jar, so that a steady state indication is present in the helium leak detector as soon as the valve to the leak detector is opened. However, the operator should always be aware that a long path leak, such as in a gasket or a threaded seal, can have a time constant longer than the pumpdown time (see Fig. 14).

Technique for Helium Bombing of Hermetically Sealed Components (Bombing Technique)

Leak tests are often required for quantities of small, hermetically sealed test objects that have an internal cavity, such as transistors, diodes and small relays. These components can be leak tested by subjecting them to an environment of high helium pressure before leak testing them in a small bell jar test fixture on a test port or leak detector. This technique is usually referred to as bombing, or more specifically helium bombing, because the test objects are bombed with high helium pressure. The logic behind this technique is as follows. If leaks are present in the test objects, the high pressure will force some helium into the part through the leaks. When these parts are subsequently subjected to the bell jar test, the helium will then issue from the leaks and be detected. The technique has the major disadvantage that gross leaks will not be found because all the helium will be quickly pumped out.

Transient Response to Pressure Cycle during Helium Bombing

The curves in Fig. 10 show, in a general way, the amount of helium that will leak into a part as a function of the bombing time duration. The left curve assumes the gas flow to be molecular in nature and illustrates that after five time constants, the internal pressure of helium in the test component will be equal to the applied helium bombing pressure. The time constant is the product of internal volume of the part and the inverse of the conductance of the leak. A leak ten times larger will reach the full pressure in a much shorter period whereas a leak ten times smaller will reach only 39 percent of the bombing pressure in the same time. In practice a bombing time of five time constants is adequate because it will produce an internal pressure that is 99 percent of the bombing pressure.

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Storage Cycle of Test Parts after Helium Bombing

The right curve of Fig. 10 shows problems in correlating measured leakage rates to actual leakage rates in helium bombing leak testing. When parts are removed from the high pressure helium bombing chamber, any helium that has leaked in begins immediately to leak out. This out-leakage rate depends on the internal pressure and the conductance of the 1.0× leak. If a part exhibiting the rate of Fig. 10 is stored for one time constant, the internal helium pressure (and leakage rate of helium) will be only 37 percent of the starting value. After five time constants, helium leakage will be reduced to less than 1 percent (effectively zero). It is interesting to note that there is a time at about three time constants after storage where the 0.1× leak begins to give a larger helium flow than the 1.0× leak.

Experimental Determination of Accuracy of Helium Bomb Leak Test

To make the helium bombing leak test technique accurate, correlation studies must be made on the parts to be tested to correlate actual leakage rates to the helium leakage rate detected after bombing.

In this type of correlation study, samples of production parts identical to those to be bombed have short lengths of tubing attached to their internal volumes, through which the parts can be subjected to vacuum or pressure testing to determine their actual leakage rates. These parts are then sealed and subjected to various helium bombings of different durations and pressures. After each bombing they are tested by the bell jar technique and the leakage rate noted. A graph can then be made of actual leakage, as a function of detectable leakage, for various bombing parameters. A correlation study is required for accurate determination of leakage whenever a different part with a different internal volume is to be leak tested with the helium bombing technique.

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PART 7. Accumulation Technique for Leak Testing of Pressurized Objects

Technique for Accumulation Pressure Testing with Helium Leak Detector

In some instances, the total out-leakage of a large pressurized system must be accurately measured. The pressure technique used to measure this type of leakage with a mass spectrometer leak detector is called accumulation testing. The vessel to be tested is pressurized with a tracer gas and placed in a sealed room with a leak detector and probe, or the instrument can be outside and the probe connected to the chamber through a port or opening (see Fig. 29). Any leakage of tracer gas from the pressurized vessel will then be picked up by the leak detector. Because the concentration of tracer gas in the room will be increased with time, the output reading will also increase with time. The accumulation technique may be applied to vessels of any size or configuration that can be pressurized at greater than atmospheric pressure.

Preparing for Accumulating Testing (Parts-per-Million Testing)

For accumulation tests, the free volume surrounding the test object within the test chamber should be minimized where possible. This is recommended to reduce the time required to accumulate sufficient tracer gas in the free volume for detection. For the purpose of estimating the quantity of gas that accumulates the relationship of Eq. 18 may be used:

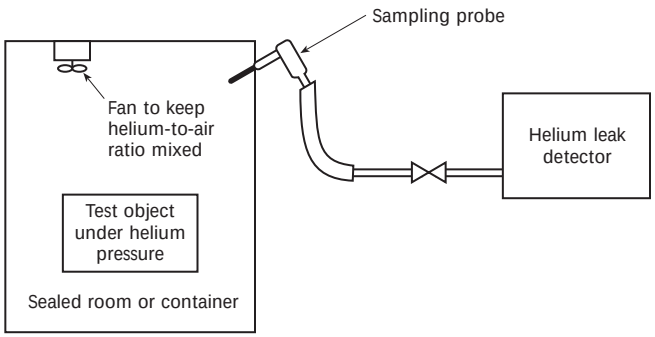
$$\Delta P = \frac{Qt}{V} \quad (18)$$

where Q is leakage rate of gas into free volume given by leakage rate specification; V is free volume; ΔP is pressure change in free volume; t is elapsed time of gas leakage Q into free volume.

Steps preceding accumulation testing are as follows.

1. Connect a suitable length of flexible hose to the sample probe and attach to the leak detector inlet.
2. The test object, as shown in Fig. 30a, should be in a sealed enclosure but not yet pressurized with the tracer gas. The enclosure may be a room or chamber, or it could be formed by blanketing a test object with a plastic sheet and sealing with tape. It is important that the free volume (chamber volume less test vessel volume) be held to a minimum where possible.
3. Place the leak detector and probe in the test chamber free volume or insert the probe through a port or opening into the free volume and note the background level signal. If desired, the background signal may be nulled out.

FIGURE 29. Parts-per-million testing by accumulation of helium tracer gas and sampling probe test. Normal percentage of helium in air is 0.0005 percent or 5 μL·L helium.



Procedure for Calibration When Accumulation Helium Detector Probe Testing

A standard helium leak is connected in a manner that will allow the gas to leak into the free volume of the closed room or accumulation chamber, for calibration as sketched in Fig. 30a. The following steps calibrate the leak detector.

1. The leak detector output is recorded as a function of time for the standard leak helium inflow rate, to obtain a calibration curve (see Fig. 30b). From this calibration curve, the unknown leakage rate can be compared and calculated.
2. After the calibration data have been acquired, the operator can close or remove the standard leak from the system, purge the free volume of tracer

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gas if necessary and pressurize the test vessel with tracer gas.

3. The leak detector output signal during leakage accumulation from the test vessel is recorded as a function of time for the item under test. When these data have been secured, the total out-leakage may be compared with and/or calculated from the standard calibration, as indicated in Fig. 30 or from Eq. 19.

$$\textbf{(19)} \quad t_1 \times \frac{Q_1}{X_1} = t_2 \times \frac{Q_2}{X_2}$$

or

$$Q_2 = Q_1 \frac{X_2}{X_1} \frac{t_1}{t_2}$$

where t_1 is accumulation time with calibrated leak (second); t_2 is accumulation time with unknown leak (second); Q_1 is known calibrated leakage rate, Pa·m^3·s^{-1} (or std cm^3·s^{-1}); Q_2 is unknown leakage rate, Pa·m^3·s^{-1} (or std cm^3·s^{-1}); X_1 is leak detector signal with calibrated leak (any unit); and X_2 is leak detector signal with unknown leak (same unit).

Equation 19 assumes that the helium leak detector panel signal meter has a linear scale with uniform divisions.

Sensitivity of Accumulation Helium Detector Probe Test

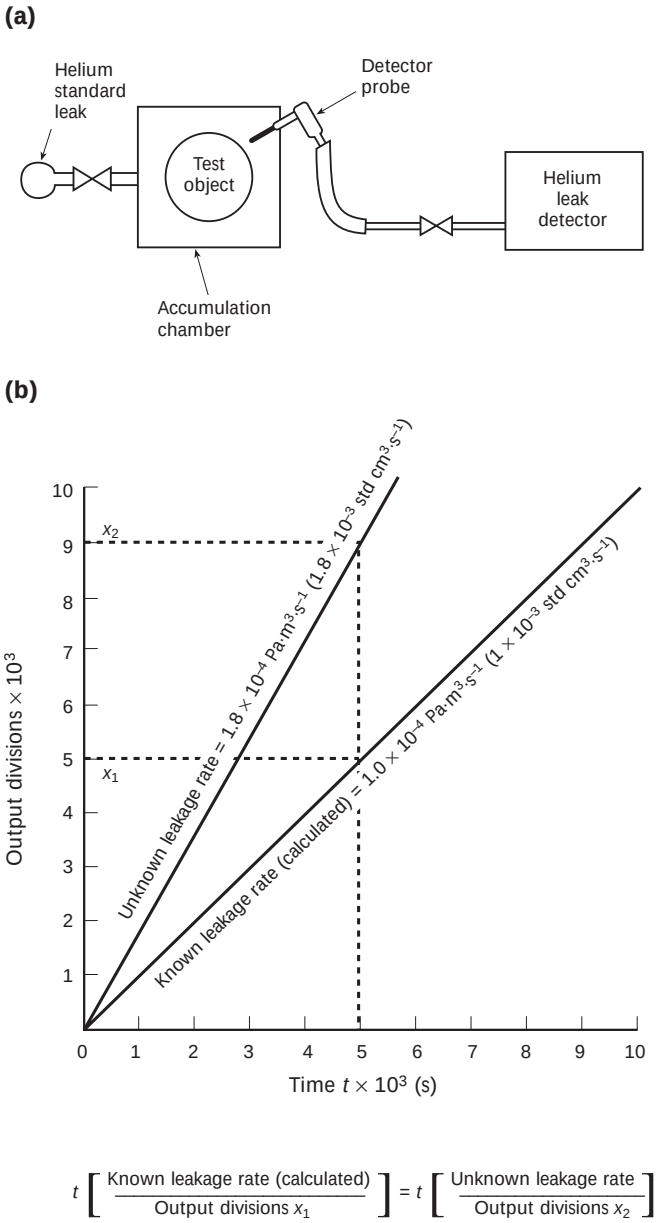
The sensitivity of an accumulation type test cannot be stated without knowledge of the sensitivity of the leak detector with probe attached, free volume of a particular test arrangement, time of accumulation and potential leakage rate of the part. Because it is difficult in many applications to determine several of these factors accurately, calibration of the system as shown in Fig. 30 is recommended when an estimate or quantitative measurement of total out-leakage is required.

In some applications where less accuracy is required, approximation of the factors that affect the system sensitivity may be made to conduct a leakage test. This involves (1) estimating the free volume, (2) knowledge of the leak detector partial pressure sensitivity for the tracer gas being used and (3) determining an appropriate accumulation time. When these factors are known, Eq. 20 may be used to determine the sensitivity Q_s for a given test arrangement:

$$\textbf{(20)} \quad Q_s = P_s \frac{V}{t}$$

where Q_s is minimum detectable out-leakage rate per division meter deflection for given test configuration, Pa·m^3·s^{-1} per division (or std cm^3·s^{-1} per division); P_s is tracer gas partial pressure sensitivity per division meter deflection of leak detector with probe, pascal per division; V is estimated free volume, cubic meter or cubic centimeter; t is accumulation time, second.

FIGURE 30. Calibration of helium accumulation out-leakage test: (a) parts-per-million test; (b) test with calibrated (standard reference) leak.



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Technique for Parts-per-Million Leak Testing with Helium Tracer Gas

In some cases the total out-leakage of a large pressurized system, such as a fuel system, must be accurately measured. The technique used to measure this type of leakage with a mass spectrometer leak detector is called the parts-per-million test. In this technique, the vessel to be tested is pressurized with helium and placed in a sealed room with a leak detector, or the instrument can be outside and connected to the room by a test line as shown in Fig. 29. A sampling probe is attached to the leak detector and adjusted according to manufacturer's instructions. The proportion of helium in air is 5 $\mu L \cdot L^{-1}$. The leak detector will indicate a constant, readable output reflecting this helium concentration, and the sensitivity of the instrument can be determined in $\mu L \cdot L^{-1}$ per division. To avoid confusion, it may be desirable to zero this reading out of the system after calibration and before leak testing the vessel. Then any leakage of helium from the pressurized vessel will be picked up by the leak detector. Because the concentration of helium in the enclosure room will be increasing with time, due to the leakage of helium from the vessel, the output reading will also increase with time. This can be converted to a leakage rate atmospheric $\mu L \cdot L^{-1}$ per unit time using the calculated instrument sensitivity.

If desired, the leakage can be converted to leakage rate units, i.e., $Pa \cdot m^3 \cdot s^{-1}$ or std $cm^3 \cdot s^{-1}$. To make this conversion, a knowledge of the room or chamber volume, less vessel volume, is necessary. The leakage rate of the vessel under test can be found by multiplying the percent helium concentration increase per unit time by the free volume of the room. For example, let the increase in helium content of the room be 8 $\mu L \cdot L^{-1}$ (which is an increase of 0.0008 percent) with a 1 h test time. Let the free volume of the room be 1 m^3 (35 ft^3); then the leakage rate would be $(8 \times 10^{-6}) \times 1\ m^3 \cdot h^{-1}$ or $(8 \times 10^{-6})/3600 = 2.2 \times 10^{-9}\ Pa \cdot m^{-3} \cdot s^{-1}$ (2.2×10^{-8} std $cm^3 \cdot s^{-1}$).

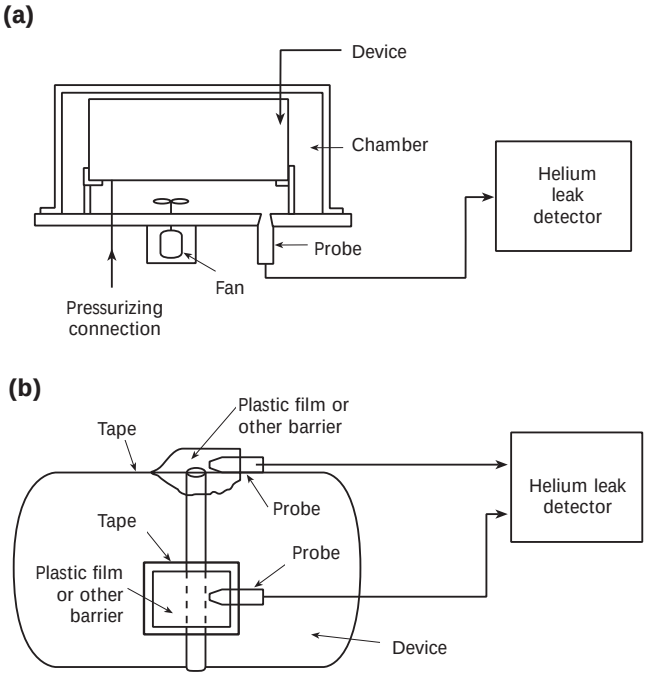
Accumulation Leak Testing

The accumulation test (see Fig. 31) provides for the testing of parts up to several cubic meter in volume as in Fig. 31a or in portions of larger objects as in Fig. 31b. This is accomplished by allowing the leakage to accumulate in the chamber for a fixed period while keeping it well mixed with a fan and then by testing the internal atmosphere for an increase in tracer gas content with the detector probe. The practical leak sensitivity attainable with this technique depends primarily on (1) the volume between the chamber and the object, (2) time available for testing and (3) the amount of outgassing of tracer gas produced by the object. Thus, a part having considerable exposed rubber, plastic, blind cavities or threads cannot be tested with the sensitivity of a smooth metallic part. The time in which a leak can be detected is directly proportional to the volume between the chamber and the part. In theory, extremely small leaks can be detected by the accumulation technique. However, the time required and the effects of other interferences limit the practical sensitivity of this technique to about $1 \times 10^{-9}\ Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-8} std $cm^3 \cdot s^{-1}$) for small parts, but only $1 \times 10^{-4}\ Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-3} std $cm^3 \cdot s^{-1}$) for volumes of several cubic meter.

Procedure for Accumulation Test with Chamber or Shroud

The accumulation test procedure is the same as the first steps of the direct probing technique. However, somewhat larger variations in atmospheric helium can be tolerated due to the isolation of the part during test. In general, it will be

FIGURE 31. Accumulation testing techniques with detector probe: (a) accumulation leak test, complete device in chamber; (b) accumulation leak test, flexible shroud over a small portion of device.



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advantageous to use the maximum stable sensitivity attainable with leak detector to reduce the accumulation time to a minimum. Steps in the accumulation test procedure are as follows.

1. Insert these three items in the enclosing chamber or shroud: the part to be tested (unpressurized), the leak standard for discharge to atmosphere and the detector probe.
2. Note the rate of increase of detector indication.
3. Remove the leak standard, pressurize the part with test gas and again note rate of rise, if any. If Step 3 exceeds Step 2, reject part.
4. Remove the part from the enclosure and purge out any accumulated helium.
5. Evacuate or purge test gas from the part, if required.
6. Write a test report or otherwise indicate test results as required.

Helium Leak Testing of Large, Complex Systems in Enclosures

Helium leak testing within enclosures provides the advantages of speed and accuracy, frequently of major concern in the leak testing of large systems. The procedural steps for helium leak testing by the accumulation of tracer gas within a test enclosure are as follows:

1. The system under test is pressurized with pure helium to the expected operational pressure.
2. The system under test is enclosed in a suitable leaktight bag, tent or other convenient enclosure within which leaking helium gas is collected.
3. The gaseous contents within the enclosure are sensed with a detector probe and a helium mass spectrometer to determine the rate of increase in helium concentration.
4. The overall leakage rate from the test object is computed by calibrating the enclosure with a known leakage or by an addition of a known quantity of helium tracer gas.

Three techniques of leak testing based on the technique used to calibrate the enclosure are (1) the injection technique, (2) the comparison technique and (3) the superposition technique.

In all three techniques, the object to be tested is pressurized with helium and the helium leakage is determined.

Preparation for Helium Leak Testing with Enclosures

A step-by-step description of the procedure used in common for the first three steps of the injection, comparison and superposition techniques of helium leak testing follows.

Pressurization of the Test Object with Helium

The object to be leak tested is pressurized with pure helium. The most advisable pressure for the leak test is the pressure that will actually be used during operation of the test object. Due to the dependence of leak structure on pressure, pressurization to a lower pressure and then extrapolating over large ranges will yield results of questionable accuracy. Furthermore, the pressure functionality of the leakage rate will depend on the type of leak. In most cases, such knowledge will be unavailable without a detailed study.

Enclosing the Test Object

The test object is next enclosed by a suitable leak testing hood or tent. The closer the conformation of the tent to the shape of the object, the smaller will be the free volume and the faster test results can be obtained. It is possible to reduce the free space volume within the tent through inert volumes such as balloons (preferably not of rubber, which absorbs helium). Tent permeability, not including gross leaks, has been shown to be negligible for most materials of reasonable thickness. Polyvinyl chloride of 0.5 mm (0.02 in.) thickness has proven to be a durable tent material.

Air Circulation in the Test Enclosure

The regular size office fan is usually adequate for a circulation within an enclosure with a volume of up to 6 m^3 (210 ft^3). However, if the enclosure volume is larger or the test object is such as not to allow adequate ventilation, a second fan may be desirable.

External Connections to Detector Probes and Standard Leaks

Detector probes and calibrated leak connections are made by means of bulkhead fittings. This is not the only way and certainly simpler connections can be made. Provision for power lines into and out of the enclosure need not be elaborate. Simple duct sealant or tape proves convenient.

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Detecting Helium Leakage from Test Object within Enclosure

After the test object is enclosed in a reasonably tight tent, the fan is turned on and the detector probe is connected to the leak detector. Adjustment of leak detector sensitivity will hinge on a knowledge of the leak range to be expected and also a familiarity of results obtained with the given volume and leak settings. Experiments in which the detector was started from a shutdown state resulted in noticeable drift in mass spectrometer readings. Newer models and designs typically require less time for warmup. Hence, it is highly advisable to keep the detector in a standby condition for a period of an hour or so, depending on the type of machine and the cleanliness of the sensing equipment.

Leak Detector Zeroing for Sampling Techniques

The leak detector is zeroed by immersing the probe in a nitrogen atmosphere. Any drift from zero may be detected after a period of use by reimmersing in nitrogen. Two types of sampling techniques are available: continuous and discrete. Continuous sampling is performed by zeroing at the start and then checking the zero at the finish of the run, perhaps 2 to 4 h later. In the discrete sampling approach, a zero is taken before each sample. Samples are taken at intervals of 20 to 30 min.

Experimental results indicate little difference in accuracy between the continuous and discrete techniques. Because continuous sampling is more convenient, it is recommended technique. However, continuous sampling admits more contamination to the mass spectrometer leak detector system. Constancy of pressure in the mass spectrometer tube is found to be very important in maintaining steady leak testing sensitivity.

Calibration of Helium Leak Tests with Test Object Enclosures

Three calibration procedures used to determine quantitative leakage rates when conducting helium leak tests by the test object enclosure technique are described next. The injection technique of calibration is probably the most rapid and simplest technique for helium leak testing in enclosures. After the unknown leakage rate has been established with the mass spectrometer in terms of leak detector readings, a known quantity of helium is injected into the enclosure. From the change in leak detector signal resulting from the injection, it is possible to evaluate the sensitivity of the leak detector for the given conditions of enclosure volume and leak detector settings. The injection technique is simple and rapid because it does not require calibrated leaks or the time necessary to perform calibration runs.

In the comparison technique of calibration, a satisfactory graph is first obtained showing helium content in the enclosure as a function of accumulation time. Then, the bag or enclosure volume is ventilated to remove all helium tracer gas. The test system is depressurized of helium and enclosed again by the bag. However, now a known leak (as close in value as may be estimated to the test system leak) is introduced into the enclosure. The graphs for the test and known leaks are then compared. This procedure may have to be repeated several times to ensure reproducibility or degree of variation between test runs.

The comparison technique of calibration is perhaps the most fundamental because it can be used when application of the other enclosure leak testing techniques might be questionable. This approach hinges on obtaining a curve relating helium leakage to time for the unknown leak and comparing it with curves for known leaks in the same system. Permeability is not a hindrance because it will presumably be identical for the unknown and calibrated leaks. This calibration technique, however, requires the greatest time to perform.

In the superposition technique of calibration, the leak test object is allowed to leak into the enclosure. After obtaining a satisfactory graph of enclosure helium content as a function of accumulation time for the test object, a known leak at least twice the estimated value of the leak is introduced into the enclosure. The unknown test object leakage is then computed from the slope of the helium content as a function of time for (1) the test leak alone and (2) the sum of the test leak and known leak.

Time Required for Leak Testing with Calibrated Enclosures

The speed of the calibrated enclosure leak testing techniques will depend on (1) enclosure volume, (2) test leakage rate and (3) detector sensitivity. Leakage rates ranging from 4×10^{-4} to 3×10^{-3} Pa·m^3·s^{-1} (4×10^{-3} to 3×10^{-2} std cm^3·s^{-1}) in a 10 m^3 (350 ft^3) enclosure are estimated to within 10 percent accuracy by the

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injection technique. Testing times are of the order of 2 to 4 h.

Example of Linearity of Helium Leak Testing System with Enclosure

The linearity of enclosure leak testing was studied by repeatedly injecting fixed portions of helium into a 6.5 m^3 (230 ft^3) enclosure (fan on) and noting the increment in reading throughout the span of the scale. Table 1 shows successive increments in mass spectrometer leak signals for the 1× and 10× scales. The difference between the greatest and least increment reading for the 1× and 10× scales was 5.32 percent and 8.83 percent. These figures indicate fairly reasonable linearity over the entire detection range. Although the results suggest the 1× scale should be better for linearity, it was found that there tended to be greater drift problems compared to the 10× scale.

Stability of Leak Test System with Enclosure

The stability of the system is important in obtaining accurate results. In general, fairly good stability over a period of several hours can be obtained with the bag enclosure system. Without question, the leak detector is the major key to achieving system stability. Close attention must be paid to the mass spectrometer manifold and analyzer tube pressure readings. If these pressures are kept at less than maximum limits, sensitivity is stable.

Effect of Detector Probe Position in Enclosure

Because of the relative densities of helium and air, intuition would lead one to believe that the concentration of helium would be somewhat greater in the upper portion of the enclosure. Following this reasoning, tests with the fan operating were conducted with the probe relatively high (2.2 m above the floor) and the calibrating leak relatively low (1 m above the floor). Tests were performed in which the probe and calibrating leak positions were interchanged. Hence the probe was now low whereas the leak was relatively high. No significant effect of position interchange on the results could be detected.

TABLE 1. Linearity and reproducibility of helium leak detector indications on 1× and 10× sensitivity scales shown by increments in scale readings after successive injections of fixed amounts of helium into 6.5 m^3 (230 ft^3) test enclosure, with circulating fan operating continuously.

1× Scale Reading Change after Injection of 10 std cm^3 (0.6 std $in.^3$) (arbitrary scale division)	10× Scale Reading Change after Injection of 50 std cm^3 (3.0 std $in.^3$) (arbitrary scale division)
5.8	11
5.4	11.6
5.9	11.2
5.8	11.5
5.6	11.1
5.5	

Design and Construction of Atmospheric Pressure Flexible Enclosures for Helium Leak Tests

The enclosure for helium testing for leaks from a pressurized system to a flexible bag or enclosure held at atmospheric pressure is designed to attain four main goals: (1) minimum gas leakage, (2) volume constancy, (3) ease of assembly, handling and shipping and (4) low cost of manufacture.

Each of these goals introduces particular considerations, some of which may be of prime importance in establishing the final design whereas others are compromised for practical reasons.

Selecting Materials for Flexible Leak Testing Enclosures

The rigid frame requirements of strength, rigidity, toughness, dimensional stability, fire resistance and ease of fabrication together with size availability suggest extruded polycarbonate or rigid polyvinyl chloride sheet. Polycarbonate is preferred as having significantly greater toughness and dimensional stability, particularly at elevated temperatures.

Enclosure sheet thickness is selected to provide a balance between rigidity required for structural support and flexibility needed to allow rolling sheets into a tubular package shape. With plastic sheet materials having flexural moduli between 2.1 and 2.8 GPa (3×10^5 and 4×10^5 $lb_f \cdot in.^{-2}$), a thickness is the range of 2 to 2.5 mm (0.08 to 0.10 in.) is satisfactory.

Reinforcing rings at each end of the cylindrical frame may be of any material that can provide the necessary rigidity. Considerations of weight and ease of fabrication may narrow the choice to fiberglass reinforced plastic (FRP), aluminum alloys or magnesium alloys. Steel parts could also be used if the design were simplified somewhat to lessen fabrication costs.

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Considerations in choosing material for the flexible bag enclosure are durability, availability in wide stock and adaptability to suitable techniques of fabricating large bags. Although some upper limit on the tolerable specific permeability must exist, in practice the degree of porosity of leaky seals is more critical. Plasticized polyvinyl chloride has a specific permeability to helium gas of about 5×10^{-17} $Pa \cdot m^3 \cdot s^{-1} \cdot m^{-2} \cdot m^{-1}$ (5×10^{-16} std $cm^3 \cdot s^{-1} \cdot m^{-2} \cdot m^{-1}$). Although other films, notably those incorporating a lamination or coating of polyvinylidene chloride, have lower permeabilities by factors of perhaps 100 to 1000, such films are not available in forms that have the necessary durability for this application. In most cases, maximum thickness produced commercially is 0.05 to 0.1 mm (0.002 to 0.004 in.), because such films find application almost exclusively in food and drug packaging.

Laminated films containing aluminum foils together with polyethylene for heat sealability and sometimes a woven fabric for strength are also available. Some of these have quite high strength, but lack the elasticity required in leak testing enclosures.

Procedure for Sealing Seams between Flexible Enclosure Sheets

For most seals, either tent-to-tent or tent-to-floor, plastic adhesive tape serves rather well. The floor on which sheets are laid out for sealing should be reasonably smooth and clean to form good seals with the tape. If the condition of the floor is such as to make achieving of good seals questionable, it is recommended that a plastic sheet be placed on the floor and sealing be made from the enclosure to the plastic floor.

Care must be exercised in forming the seals. If arching or sloping off of the graphs of helium concentration with time is observed, this is taken as evidence of poor seals. Although the sloping-off effect may appear at a glance as being small, it could easily mean a difference of 10 percent in accuracy. However, the initial leak detector response in the first 15 min or so may yield a curve that might be interpreted as sloping off due to leakage. Therefore, it is desirable to allow the detector to equilibrate by taking readings for 15 min or so before seriously considering the readings as part of the curve relating helium concentration to accumulation time.

Refrigerant System Leakage Measurement by Accumulation of Helium in Enclosure

The enclosure technique uses pressurization of the component or system with helium. The unit, after pressurization, is enclosed in a tight container having a minimum free volume. Leakage of helium from the unit is allowed to accumulate within the free volume for a measured period of time. A simple probe is then inserted into the free volume. The corresponding output indication of the helium leak detector is used to compute the rate of total leakage.

Production leak testing rates depend mainly on the time for accumulation of enough gas to give an adequate signal. Operator skill is not a production limiting factor in this technique because all parameters are fixed. Decision by the operator to reject or not is virtually eliminated by having the no-go point indicated by an audio alarm.

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CHAPTER 9

Mass Spectrometer Instrumentation for Leak Testing

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PART 1. Principles of Detection of Helium Gas by Mass Spectrometers

Characteristics of Helium As a Tracer Gas for Leak Testing

Helium, the tracer gas most commonly used for leak testing, is the lightest chemically inert gas. It is monomolecular with a relative atomic mass A_r(He) of only 4 unified atomic mass units (u). At any specific temperature, helium molecules have higher particle velocities than those of any other gas except hydrogen, so that helium penetrates through leaks more rapidly than most other tracer gases. Helium is chemically inert and is a noble gas that does not corrode or damage metallic materials. It is also an ideal tracer gas in terms of its detectability in air or gas mixtures by means of the mass spectrometer, which responds even to the five parts per million (5 $\mu L \cdot L^{-1}$) of helium present in the normal earth's atmosphere. Helium is nontoxic, nonflammable and nonhazardous unless, if it collects in portions of closed vessels or enclosures, it completely displaces air or oxygen needed for human respiration.

Although gases other than helium have been used for some applications, helium has the following outstanding qualifications for the task. (1) Helium is nontoxic and environmentally safe. (2) Helium is nonreactive with chemical processes and is noncontaminating. (3) Helium has a high mobility, so it diffuses quickly and thoroughly within a vacuum apparatus. (4) The detection of helium (with the mass spectrometer) is unambiguous. (5) The background (ambient) helium concentration is low and stable. (6) Helium's low atomic weight lets it flow through a leak (if in the molecular flow regime) at a higher rate than any other gas except hydrogen.

Terminology for Mass Spectrometer Helium Detector Components

The mass spectrometer is the preferred detector for helium tracer gas used in leak testing. The largest application of mass spectrometers is the location and measurement of extremely fine leaks. The versatility of mass spectrometer instruments has led to an extremely wide variety of applications. Although it is true that the mass spectrometer leak tester is occasionally misapplied, the greater problem presently lies in the frequent misuse of mass spectrometer leak detection equipment because of lack of understanding of its basic principles.

Various terms are used interchangeably to identify the helium separation and sensing components of helium mass spectrometers, including (1) *helium leak detector* or *sensor*, (2) *helium mass spectrometer sensor* (or simply *sensor*), (3) *helium analyzer tube* (or *analyzer* or *tube*), (4) *helium ion source* or *source tube*, (5) *helium ion analyzer tube* (or *spectrometer tube* or *sector tube*), (6) *helium magnetic analyzer* or *tube* and (7) *source* or *mass spectrometer tube*.

Figure 1 shows the numerous components and physical systems that function together to sense and indicate the partial pressure of helium within the mass spectrometer sensing element. The functions of each component and the physical principles on which each operates are described in detail below.

Sensitivity of Helium Mass Spectrometer

The helium mass spectrometer leak detector can detect 0.1 $\mu L \cdot L^{-1}$ of helium gas in air. With a highly sensitive helium leak detector, it is possible to detect and measure minimum helium leakage rates in the range of 5×10^{-12} Pa·m^3·s^{-1} (5×10^{-11} std cm^3·s^{-1}). This amount of leakage is so small that it would take more than 1000 yr for 1 cm^3 of air to leak from a vessel pressurized at 100 kPa (gage pressure) or about double the normal atmospheric pressure, to air at atmospheric pressure. Basically, the helium mass spectrometer can be used to detect and indicate a range of helium leakage rates from 1×10^0 to 5×10^{-12} Pa m^3·s^{-1} (or 1×10^1 to 5×10^{-11} std cm^3·s^{-1}).

In special leak testing applications that require sensing the normal partial pressure of helium in atmospheric air, the helium mass spectrometer leak detector can be used to detect air leakage rates as large or larger than 0.1 Pa·m^3·s^{-1} (1 std cm^3·s^{-1}). When operating in the detector probe detection mode, where helium

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tracer gas (often mixed with inert gas such as nitrogen) leaks into the atmosphere, the 5 $\mu L \cdot L^{-1}$ helium content of the normal atmosphere may establish a threshold level. The helium leak detector's sensitivity to leakage rates that increase the helium content of tracer gas input to the detector probe is limited by fluctuations in the threshold level to two or three times the residual helium content of the atmosphere. This limits the minimum leakage rate to about 10^{-8} Pa·m^3·s^{-1} (10^{-7} std cm^3·s^{-1}).

A helium mass spectrometer leak detector is a complete system for locating and/or measuring the size of leaks into or out of a device or a container. In use, this technique of leak detection is initiated when a tracer gas, helium, is introduced to a test part that is connected to the helium mass spectrometer leak detector system. The helium leaking from the test part diffuses through the detector system, its partial pressure is measured and results are displayed on a meter. The mass spectrometer leak detector operating principle consists of ionization of gases in a vacuum and acceleration of the various ions through electrical and magnetic fields. The helium ions are separated and collected and the resulting ion current is amplified and indicated on an indicating device. Modern leak detector meters are often calibrated in std cm^3·s^{-1} despite the fact that the actual parameter being measured is helium partial pressure within the spectrometer tube. This is made possible when the leak detector pumping speed is known and is constant.

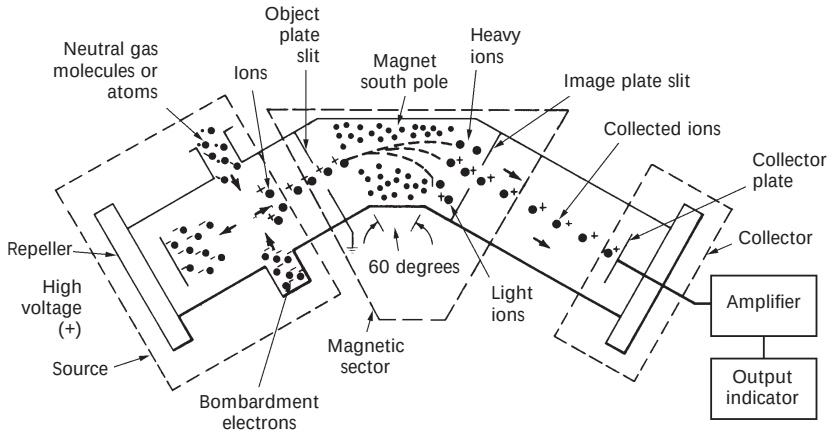
A helium mass spectrometer leak detector consists of a spectrometer tube, quantitatively sensitive to the presence of helium; a vacuum system, to maintain adequately low operating pressure in the spectrometer tube; mechanical pump(s), to evacuate the part to be tested; valves, to transfer the connection of the evacuated part from the mechanical roughing system to the spectrometer vacuum system; amplifier and readout instrumentation, to monitor the spectrometer tube output signal; electrical power supplies and controls, for valve sequencing, protective circuits etc.; and fixturing, for attachment to the part to be leak tested.

Applications of Helium Mass Spectrometer Leak Detectors

The mass spectrometer leak detector is presently the most satisfactory and versatile means for performing rapid nondestructive leak tests with helium tracer gas in certain types of industries requiring minimal leakage rates. By using helium tracer gas and the ultrasensitive helium mass spectrometer, one can achieve a greater assurance of leak tightness in both large and small test objects and systems than with most other leak testing techniques.

A mass spectrometer helium leak detector can provide an immediate indication of (1) the existence of leakage, (2) the locations of leaks and (3) the rates of leakage. Reproducible leak testing

FIGURE 1. Arrangement of 60 degree magnetic sector mass spectrometer.



Legend

- $\bullet^{\cdot}$ = neutral gas atom or molecule
- $\bullet^{-}$ = electron
- $\bullet^{+}$ = positive ion

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indications can be obtained by personnel with a normal degree of training in leak testing and in operation of the spectrometer, using various leak testing techniques. Many helium leak testing mass spectrometer instruments are portable and can be used to detect leaks in almost any test object or system — in the laboratory, on a production line or during construction in the shop or field. Industrial or laboratory equipment, high pressure systems, compressor units, glass-to-metal seals, hermetically sealed components, space capsules, large and small dewars and valves (including those in service) are but a few of the products routinely tested with helium mass spectrometer leak detectors.

Specific examples of applications of leak testing with helium tracer gas and mass spectrometer leak detectors to detect minute leaks include (1) miniature semiconductor and integrated circuit devices; (2) small hermetically sealed electrical and electronic components; (3) cryogenic and vacuum equipment; (4) large refrigeration equipment and heat exchangers; (5) large chambers used to simulate space environments during testing; (6) nuclear reactor pressure vessels, piping and enclosures; and (7) high vacuum sections of large high energy particle accelerators.

Versatility of Helium Mass Spectrometer for Leak Testing

The high sensitivity, dependability, versatility and ease of operation of the helium mass spectrometer leak detector have made this instrument the unparalleled standard of high sensitivity nondestructive testing for leaks. In addition to the leak testing applications already described, the helium leak detector can be used to determine the helium content of any gaseous mixture, to study the diffusion rate of helium through various materials or to ascertain the sealing quality of materials proposed for vacuum seals. Leak tests can be conducted on either evacuated or pressurized equipment or on pressure boundaries with above-atmospheric pressure on one side and vacuum on the other side. Sealed components subjected to high external pressure of helium, as during helium bombing, can then be tested by detection of out-leakage of the helium from within a bell jar enclosure.

The helium mass spectrometer has found use in environmental testing to study leakage effects resulting from pressure, heat, vibration or shock. The helium leak detector has also been used in process control and setup operations for production welding of hermetically sealed parts or fabricated assemblies. For example, it was used to check welds on bellows assemblies to aid in determining the optimum resistance welding machine settings and welding schedules. This contributed to a lower final rejection rate of bellows assemblies, where rejection was based on unacceptable levels of leakage.

Direct Flow and Counterflow Leak Detectors

To understand the discussion of mass spectrometer vacuum system design and operation, a brief review of the differences between the older direct flow and the newer and more commonly used counterflow mass spectrometer may be useful (see Fig. 2).

Direct Flow Leak Detector

Before 1970, all leak detectors used direct flow (Fig. 2c), requiring the use of a liquid nitrogen chilled cold trap. The cold trap was necessary because the maximum pressure allowed in the helium sensor, often called the *spectrometer tube,* was usually 25 to 40 mPa (0.2 to 0.3 mtorr). This limits the throughput of the leak detector vacuum system to about 0.006 $Pa \cdot m^3 \cdot s^{-1}$ (0.06 std $cm^3 \cdot s^{-1}$). This mass flow of gas is given by the product of total pressure times the effective pump speed at the pressure of the diffusion pump evacuating the helium sensor. This requires the test object to be evacuated with an auxiliary rough pump to as low as most mechanical pumps can possibly attain, before exposing the remaining gas load directly to the helium sensor.

Because a large part of this gas load is water vapor, the cold trap effectively condensed or pumped it quite well, but only if the total gas load remaining in the test object was less than the throughput of the high vacuum system. Unfortunately, this was usually not true. As a result, the remaining gas flow from the test object had to be carefully throttled into the helium sensor with a variable valve, without overpressuring the helium sensor. The remaining gas flow had to be bypassed to the roughing pump. This bypassing results in a loss of sensitivity, because much of the helium tracer also was bypassed.

In Fig. 2c, the test valve would be partially opened and the roughing valve would be fully opened. However, if the total gas load and tracer can be tolerated through the test valve, leakage as slow as 2×10^{-12} $Pa \cdot m^3 \cdot s^{-1}$ (2×10^{-11} std $cm^3 \cdot s^{-1}$)

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can be detected in this type of leak detector. This leakage is about ten times slower than that detected by counterflow mass spectrometer leak detectors of the mid-1990s. Few applications require such high sensitivity.

One advantage of the direct flow leak detector is that it can be used to leak test high or ultrahigh vacuum systems directly without contaminating them with oil vapor backstreaming from the leak detector into the system being tested. Counterflow leak detectors using dry (oil free) mechanical pumps can also be used for this same application safely.

Counterflow Leak Detector

Since the counterflow mass spectrometer leak detector was introduced in the early 1970s, most manufacturers sell far more counterflow than direct flow units. In counterflow systems (Figs. 2a and 2b), after rough pumping the test object to 13 Pa (0.1 torr), the test valve is opened, exposing the remaining gas and tracer to the foreline instead of to the helium sensor. The forepump now must continue to keep the test object at or below this maximum tolerable forepressure during testing.

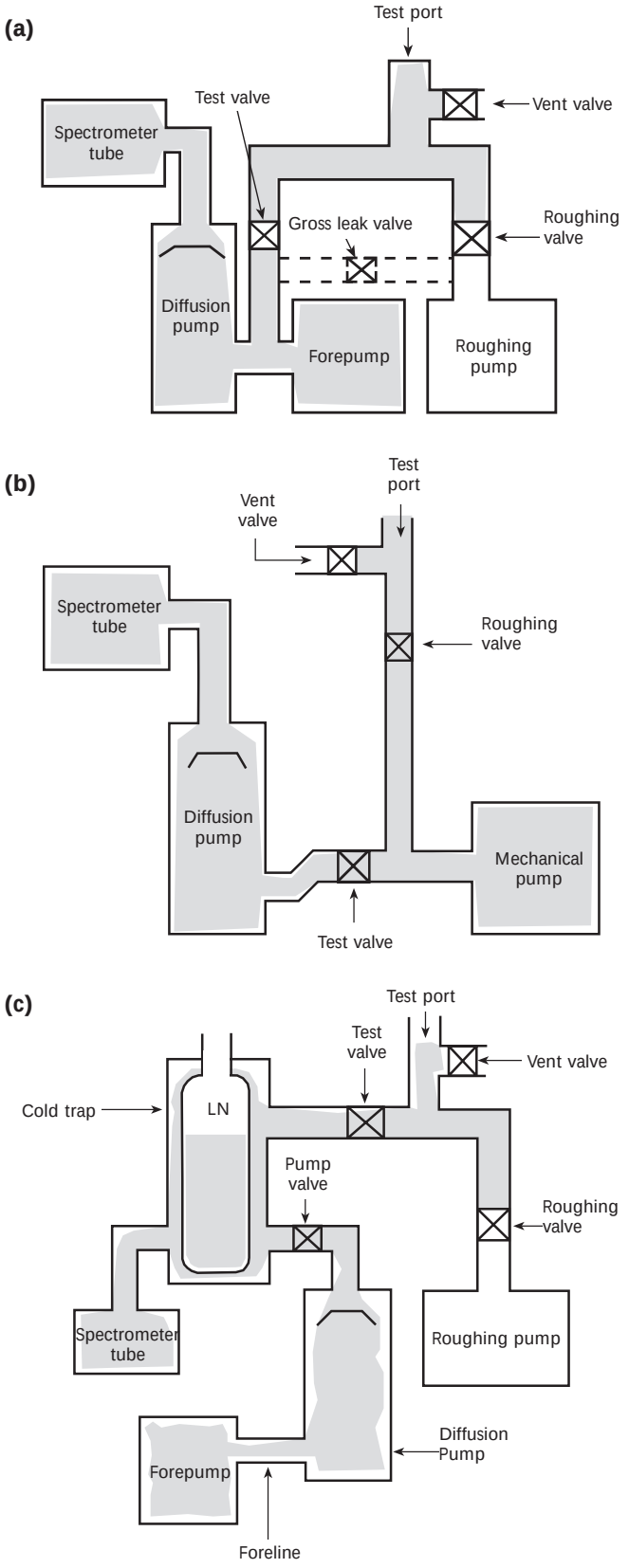
When helium reaches this injection point, a fixed proportion flows backwards through the diffusion pump to the helium sensor without affecting this pump's ability to compress the heavier gases toward the forepump. As the level of tracer in the foreline rises and falls, a similar effect takes place in the helium sensor. An improvement to this design for testing at pressures as high as 700 Pa (5 torr) is possible if a turbomolecular drag pump is used.

Advantages of Counterflow Principle

Advantages of the counterflow principle include the elimination of liquid nitrogen, thus saving cost and removing a potentially hazardous material. In addition, pump times are reduced, as tests can be made at pressures ranging from atmosphere to high vacuum without adverse affect on the diffusion pump. In a direct flow helium leak detector, the diffusion pump must be protected from exposure to pressures above 10 Pa (0.1 torr). Particular advantages are achieved with counterflow systems when testing large systems that cannot be evacuated to low pressures. This type of leak detector is available as an automatic cabinet system (Fig. 2a) or as a portable unit (Fig. 2b).

A disadvantage is that the test port is never at a high vacuum and normally cannot be connected to any piece that is at a high vacuum. Also a disadvantage is the possibility of contaminating the parts

FIGURE 2. Leak detector vacuum system configurations: (a) counterflow system with dual primary pumps; (b) portable counterflow system with single primary pump; (c) direct flow system with dual primary pumps and liquid nitrogen cold trap.



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to be leak tested with mechanical pump vapors. The best approach for controlling or eliminating this possibility is to use oil free (dry) mechanical pumps. In direct flow leak detectors, the sensitivity decreases at higher pressures, because the admittance of test gas to the detector must be restricted by throttling valves.

The counterflow leak detector, on the other hand, shows an almost constant sensitivity, independent of total pressure. The operation of the counterflow leak detector can be, of course, extended to still higher pressures by using a throttling valve after the tolerable forepressure of the diffusion pump is approached. In this case, the advantage over the conventional detector is preserved, the two response curves continuing parallel to each other toward higher pressures.

Thus, under carefully controlled conditions, the sensitivity of the counterflow leak detector can be as high as the direct flow unit (for the same spectrometer and electronics). However, the main advantage is the simplicity of operation and the higher sensitivity at the higher test pressures. The advantage is particularly great when the gas producing the high pressure is noncondensable.

Beyond the conveniences of equipment design, simplicity and perhaps cost of operation, the question remains: What are the basic advantages and disadvantages of the two techniques? Or which technique is suited for which applications?

For precise measurements (repeatability and accuracy) of very small helium flow rates, the direct flow leak detector has an overall advantage because of the promise of better linearity under molecular flow conditions. An example of such an application would be permeability measurements of helium through "porous" solids. For applications in which the primary object is to find leaks, particularly with systems and objects that are difficult to pump into the high vacuum range, the counterflow technique provides a very useful and more sensitive technique.

Totally Dry Leak Testing

In some of the more sophisticated vacuum system requirements, contamination by hydrocarbons (although minimal) can pose serious problems. As a result, vacuum pump and semiconductor system manufacturers have introduced oil free versions of their equipment, eliminating hydrocarbon vapors that can diffuse into the processing system, thus improving device yield.

High throughput, dry, scroll or diaphragm type roughing or backing pumps, combined with oil free turbomolecular pumps, provide leak testing totally without contamination. There is a benefit to the cleanliness of the part or system under test; moreover, the spectrometer tube stays extremely clean, reducing maintenance in the most critical part of the leak detector. In addition, dry vacuum pumping systems have a key advantage in not having to add or dispose of oil for the roughing and backing pumps, saving significant operating costs compared to conventionally pumped leak detection systems.

Basic Operation of Mass Spectrometer Helium Leak Detector

A mass spectrometer (see Fig. 1) is basically a device for electromagnetic sorting of charged gaseous particles by their species in accordance with their molecular weights. More precisely, the analyzer tube of the mass spectrometer divides mixtures of charged gaseous ions into different curved paths that depend on the mass-to-charge ratios for each individual species of particles. Baffles containing narrow slits are then used to obstruct all but the desired species of gaseous ions from reaching the collector. Helium ions with their positive charge are allowed to reach the collector in helium leak detectors. The number of helium ions that reach the collector per unit time constitutes an electrical current signal proportional to the concentration of helium atoms in the incoming gaseous mixtures. Typically, the signal current is shown as an amplified voltage on a leakage rate display.

Helium or Other Tracer Gas

For helium leak testing applications, the mass spectrometer design factors are optimized to produce a mass spectrometer with great sensitivity to helium gas alone. Other mass spectrometers have been tuned to detect only argon as a tracer gas. Actually, if an analytical mass spectrometer is used for leak testing, almost any specific gas over a wide range of molecular weights can be used as a tracer.

The output signal of the mass spectrometer leak detector used only for detecting of helium tracer gas leakage is a digital or analog display indication that may be supplemented by visible or audible alarms. This signal magnitude is proportional to the absolute partial pressure of helium gas atoms in the analyzer tube of the mass spectrometer. Vacuum pumps within the leak detector serve to move tracer gas from leaks into the mass spectrometer. They also create

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the necessary vacuum (typically below 0.1 Pa or 1 mtorr) within the analyzer tube to allow ions to follow the desired paths without frequent collisions with other gaseous particles.

Causes of False Leakage Signals on Mass Spectrometer Leak Detector

Care is usually required to ensure that helium from other sources does not influence the response of the mass spectrometer so as to produce false or misleading leakage signals. Other sources of helium producing false leakage signals include leaks in the mass spectrometer vacuum system itself or outgassing of helium absorbed on contaminated surfaces internal to the vacuum system. Rubber and certain other materials, as well as grease or oil, can serve as reservoirs for storage of helium. When these sources continue to emit helium into the mass spectrometer source chamber, a continuing false signal can occur as a result of this helium hangup.

Another false signal may be caused by diffusion of atmospheric helium backwards through the exhaust opening of the forepump. This is more troublesome with dry pumps but can be reduced by ducting forepump gases to outside atmosphere areas.

Capabilities and Limitations of Helium Mass Spectrometer Leak Detector

The helium mass spectrometer leak detector can be used either as an instrument to measure helium concentration in a gas mixture or as a flow meter for a gas mixture containing helium. Either the total pressure or the effective pumping speed can be held constant during the operation of the helium mass spectrometer leak detector. During the leak detection, the output signal of the mass spectrometer is directly proportional to the number of helium atoms within the sensing element, without regard to the total gas pressure within the sensing element (when operating below the maximum recommended operating pressure). However, the number of helium atoms within the sensing element is proportional to the concentration of helium atoms in the gas mixture, so the number of helium atoms is proportional to the total pressure of the gas mixture in the source chamber of the spectrometer.

In more functional terms, the output signal of the helium mass spectrometer leak detector is proportional in magnitude to the partial pressure of helium within the sensing element. The partial pressure of helium is proportional to the total gas pressure (in a gas mixture with a specific percentage concentration of helium atoms). For maximum leak testing sensitivity, the total gas pressure within the sensing element of the spectrometer should approach (but not exceed) the maximum recommended working pressure of about 25 to 40 mPa (0.2 to 0.3 mtorr).

Measuring Helium Concentration with Mass Spectrometer Leak Detector

If the total pressure within the sensing element of the helium mass spectrometer is held constant, the output signal indicated by the mass spectrometer meter deflection is proportional to the partial pressure of helium. This helium partial pressure is itself proportional to the concentration of helium. In fact, the helium partial pressure P_{He} is equal to the fractional concentration of helium C multiplied by the total gas pressure P_t:

$$P_{He} = CP_t \quad (1)$$

where P_{He} is partial pressure of helium (pascal or torr); C is concentration of helium (fraction by volume); P_t is total pressure of gas mixture (pascal or torr). Similar pressure units (pascal or torr) must be used for both pressure terms.

Principles of Operation of Mass Spectrometer Instrument

The mass spectrometer instrument sketched in Fig. 1 produces a beam of positive ions from a sample of tracer gas being investigated, sorts these ions into a spectrum of mass-to-charge ratios and records or indicates the relative abundance of each species of ion present. In mass spectrometers, the ion currents of specific ion species are detected electrically. The signal is usually amplified electronically before being displayed or recorded. The primary functions of a mass spectrometer instrument are to be sufficiently sensitive to detect all desired ion currents and to be able to resolve or separate completely the ion currents due to different ion species. Common functions of commercially available mass

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spectrometer leak detector instruments are (1) pumping tracer gas samples from leaks in test objects into the vacuum of the instrument, (2) ionization of gas sample molecules by electron impact and (3) sorting and identification of positive ions according to their mass-to-charge ratios.

Hot tungsten, iridium or rhenium filaments are used as sources of electrons for ionization of the molecules of tracer gas pumped into the vacuum of the mass spectrometer. The resultant monoenergetic positive ion beams produced in the source chamber are then accelerated electrostatically and passed through an analyzing magnetic field that serves as a momentum filter. Within this magnetic field, the ion beam of the tracer gas is deflected through angles of 60, 90 or 180 degrees in various types of commercially available mass spectrometer instruments. The combination of the monoenergetic ions and momentum filtering provides mass separation of ions. After separation, one or more distinct species of ions can be passed through separating slits and collected on a target plate connected to an electrometer (charge detector). The output electrical signal from the electrometer is amplified and typically displayed on a multirange leak signal meter.

Because a vacuum is necessary for the operation of the mass spectrometer, leak detection spectrometers are equipped with vacuum pumps and operate internally as high vacuum systems. Liquid nitrogen traps, oil diffusion pumps, inlet throttle valves and turbomolecular pumps are used to attain this vacuum. The components being leak tested do not necessarily have to be within this vacuum, although sensitivity is decreased when test objects are not leaking into a vacuum environment. An alternative is to pass the main flow from the leak through a large mechanical pump while the rest of the flow goes into the leak detector through a throttle valve set so that the leak detector maintains high vacuum. Another alternative is to use a counterflow helium leak detector.

Sensitivity of Helium Mass Spectrometer Leak Detectors

Mass spectrometer leak detectors have typical leak sensitivities of 1×10^{-11} to 5×10^{-12} Pa $m^3 \cdot s^{-1}$ (1×10^{-10} to 5×10^{-11} std $cm^3 \cdot s^{-1}$) for helium tracer gas. Fundamental sensitivity of the helium mass spectrometer leak detector is about 0.1 $\mu L \cdot L^{-1}$ of air. When this instrument is used in the dynamic operation mode, this fundamental sensitivity implies a leak testing sensitivity of 5×10^{-12} Pa $m^3 \cdot s^{-1}$ (5×10^{-11} std $cm^3 \cdot s^{-1}$). This sensitivity is reduced if additional pumps must be used in leak testing or if the mass spectrometer is used in the detector probe (sniffing) mode. If a counterflow detector is used, the sensitivity is about 1×10^{-11} Pa $\cdot m^3 \cdot s^{-1}$ (1×10^{-10} std $cm^3 \cdot s^{-1}$).

Interpretation of Leak Detector Sensitivity with Mass Spectrometer

For helium mass spectrometer leak detection systems, the term *leak detector sensitivity* is specified and interpreted in two ways:

1. By the smallest detectable tracer gas concentration in air (formerly expressed in parts per million but now expressed in SI units as $\mu L \cdot L^{-1}$). This leak sensitivity value for the helium mass spectrometer leak detector is about 0.1 $\mu L \cdot L^{-1}$.
2. By the minimum partial pressure of helium that would produce the minimum detectable leakage indication. This minimum detectable leakage signal is often taken as a leak signal magnitude three times the magnitude of the random noise signal associated with the leak test.

The second of these definitions is commonly applied to helium leak detectors. Manufacturers of leak detection mass spectrometers often use another form of this second definition, namely the smallest helium leak that can be detected at a specified tracer gas source pressure. This specified pressure at the inlet port of the mass spectrometer leak detector is usually atmospheric pressure and is stated under specified leak testing conditions. This last definition is often called the smallest leak detectable and is given in units of leakage such as pascal cubic meter per second (Pa $\cdot m^3 \cdot s^{-1}$), standard cubic centimeter per second (std $cm^3 \cdot s^{-1}$), torr liter per second (torr $L \cdot s^{-1}$). For the helium mass spectrometer leak detector, this sensitivity value is about 5×10^{-12} Pa $m^3 \cdot s^{-1}$ (5×10^{-11} std $cm^3 \cdot s^{-1}$).

Another term often used is the minimum detectable leak, defined as the smallest leakage that can be clearly detected in the presence of noise signals or tracer gas contamination of the air in the leak testing area. An alternative definition of this minimum detectable leak is the product of the minimum detectable pressure change and the pumping speed at the detector. These definitions of leak sensitivity are used interchangeably in this discussion, with the same types of units.

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In interpreting sensitivity claims for mass spectrometer leak detectors, it must be remembered that helium flows through a leak more readily than air. The sensitivities of most commercial helium leak detectors are expressed in terms of 100 percent helium tracer gas. Some misleading advertisements state the minimum detectable leakage as the air leakage, even though the leakage measurements were performed with helium gas. The advantage of stating leakage in terms of air units is that the value for the minimum detectable leak will appear 2.7 times smaller than the equivalent helium leak. The difference in specification for helium mass spectrometer sensitivities should be evaluated carefully when comparing advertised sensitivities of various helium leak testing systems.

Analogy between Mass Spectrometer and Visible Light Spectrometer

The mass spectrometer can be understood through an analogy with a visible light spectrometer. Figure 3 shows the arrangement of components of a visible light spectrometer that has a prism to refract different colors or wavelengths of light at different angles. In the light spectrometer, a narrow beam of white light (containing many wavelengths) is formed by a slit. The light beam enters the prism where various colors (individual wavelengths) are refracted at characteristic angles. The intensity of any specific refracted wavelength band of color could be measured by placing a light detector in its portion of the light spectrum formed by the prism. The output electrical signal from the detector could be amplified and displayed by means of a panel meter. Alternatively, by moving the light detector across the spectral plane and recording the light intensity signals as a function of light wavelength, the intensities of spectral bands could be plotted as a function of light wavelengths (see Fig. 4).

Figure 5 shows the analogous components of a single sector Nier mass spectrometer analyzer tube. Tracer gas such as helium from a leak in a test object, together with air, nitrogen or other gases, enters the upper portion of the chamber and is ionized by electron bombardment. The resultant monoenergetic gaseous ion beam containing ions of many different gaseous elements is accelerated through a slit and enters the magnetic deflection field of the mass spectrometer tube. Here, ions with different ratios of mass to charge are refracted (deflected) at different angles.

The magnetic field is analogous in its action to the prism of the light spectrometer. The specific tracer gas ions (such as those of helium) can be selected and separated from all other gaseous ions by a slit arrangement, placed in the focal

FIGURE 3. Components of visible light prism spectral analyzer.

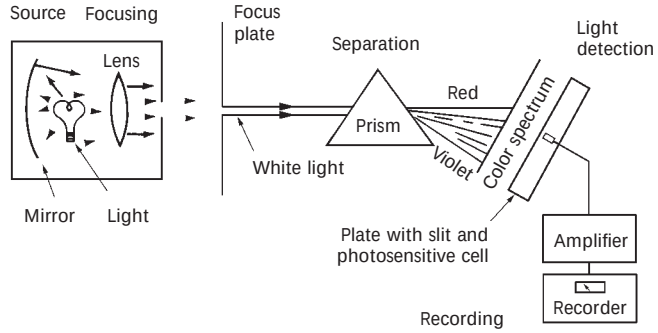
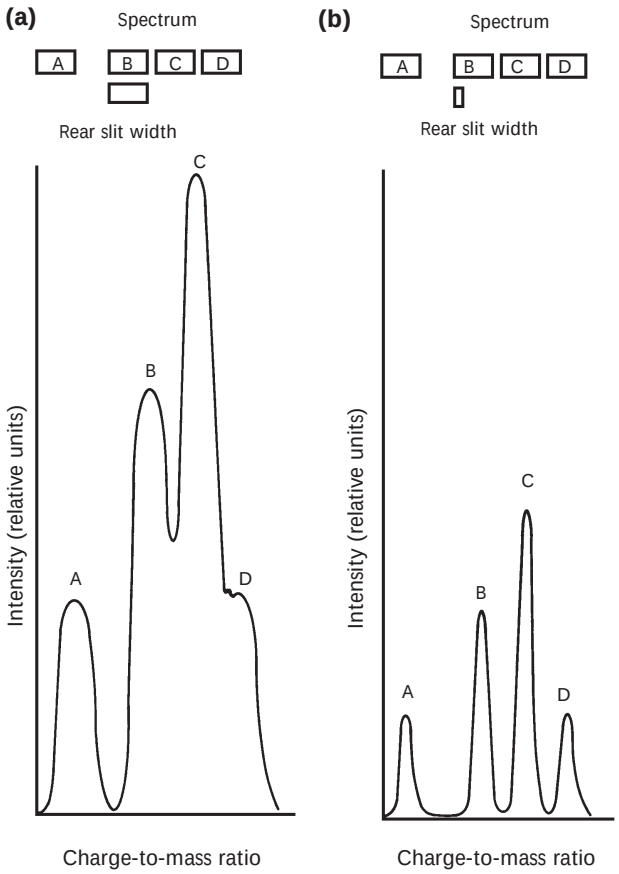


FIGURE 4. Spectrum scan recording using wide and narrow scan with rear slit widths, constant source slit: (a) scan with wide slit; (b) scan with narrow slit. This example is for a small radius mass spectrometer used in leak detectors.



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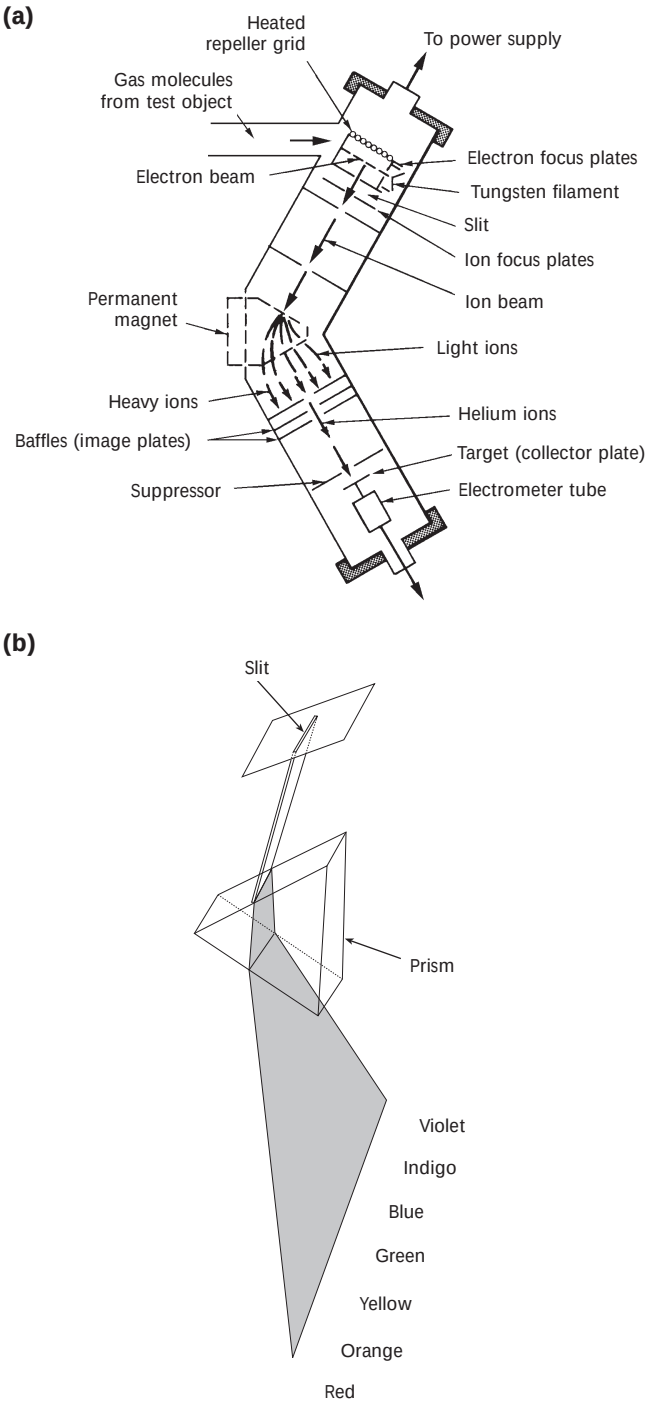
plane of the mass-to-charge spectrum formed by the magnetic field of the mass spectrometer. The intensity (relative abundance) of any specific ion species can be measured by collecting these positively charged ions on a target electrode. The resultant electrical signal is amplified by an electrometer circuit with high charge sensitivity and is displayed on a panel meter. If desired, the mass spectrometer can be designed to move the ion spectrum across the separating slit system, so that the signal current can be recorded as a function of the ion mass-to-charge ratio. This permits the relative abundance of different species of ions to be plotted as a function of mass-to-charge ratios, as sketched in Fig. 4.

FIGURE 5. Operating principles of a mass spectrometer tube and its analogy to the prism light spectrometer: (a) mass spectrometer tube; (b) analogous separation of light into a spectrum of wavelengths by a slit-and-prism optical spectrometer.



Effect of Small Scanning Slit Width on Spectral Sensitivity and Resolution

If a scanning slit width equal to one fifth of a single bandwidth is used in analysis of the light spectrum, the scan recording would look like that of Fig. 4b. Bands B and C are now almost completely separated, with only a slight signal contribution from adjacent bands to the signal level in the valley between them. The same is true for bands C and D. The peak signal heights in the spectral scan recording are smaller in Fig. 4b than those obtained with the larger slit width used during the record of Fig. 4a. This indicates that the smaller slit width reduces the sensitivity of the spectral measurement apparatus. However, the smaller slit results in better resolution in spectral measurements because of the reduction in signal contributions from adjacent frequency bands to the signal level in the valleys between these bands. As the slit width is reduced, measurement sensitivity is traded off for improved resolution.

Functions of Analogous Components in Light and Mass Spectrum Analyzers

Figure 6 illustrates the analogous functions of individual components of the light spectrum analyzer of Fig. 3 and the mass spectrum analyzer of Fig. 5. Each system involves five basic functional steps: (1) providing a source for the beam whose spectrum is to be analyzed; (2) focusing or directing the beam to concentrate as much of the beam intensity as possible onto the collimating slit that establishes scanning beam size and intensity; (3) selective angular deflection of the collimated beam in accordance with characteristics of individual species that make up the beam and with separation of these dispersed bands of species by means of a slit that transmits only one specific species from the original beam; (4) providing means for detection and amplification of the spectral signal corresponding to a single species selected from the entire spectrum of species included in the original beam;

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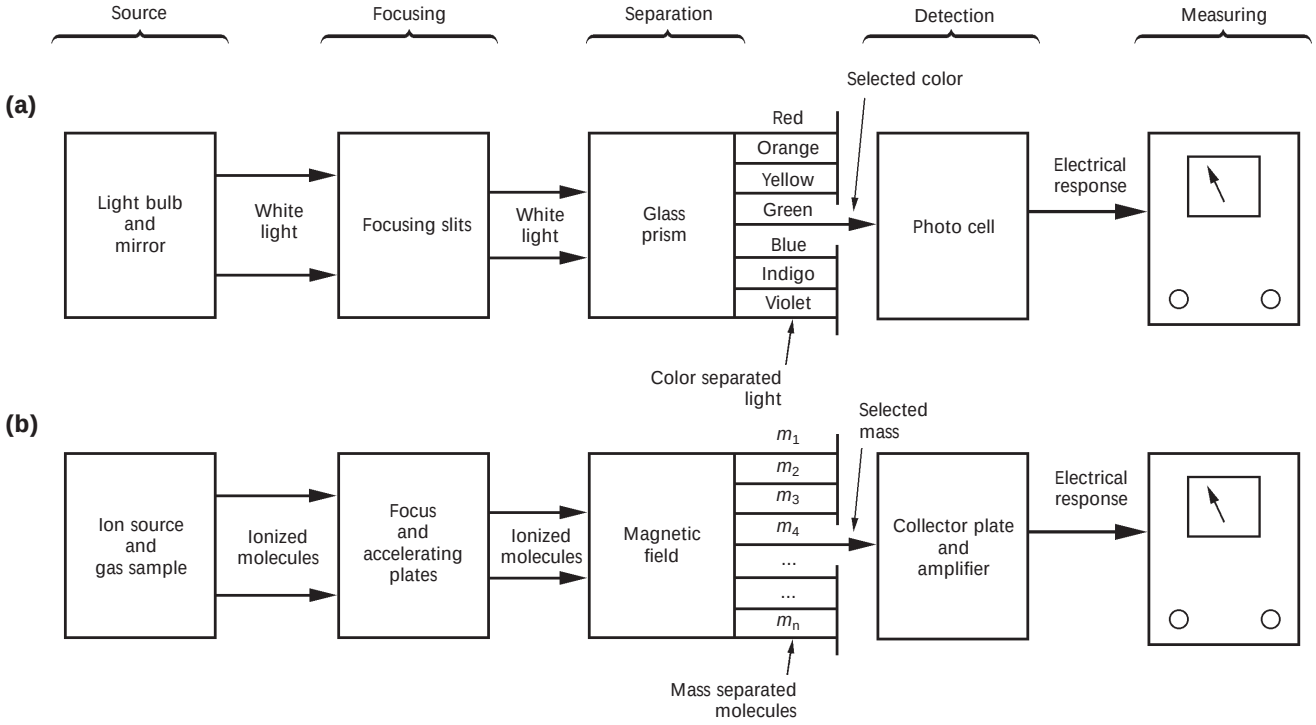
FIGURE 6. Functional similarity of spectrometers: (a) light spectrometer; (b) mass spectrometer.



and (5) displaying or recording quantitative output signals that are proportional in magnitude to the energy or number of discrete particles of the specific species passing through the separating slit at any instant.

Mass spectrometer instruments capable of resolving a gas sample into its individual gaseous constituents are analogous to light spectrometers. Functional similarities of each stage or segment in the light spectrometer and the mass spectrometer are shown in each vertical column of Fig. 6. The primary difference between these two analyzer systems is the nature of the beam analyzed. The light spectrometer analyzes the wavelengths of photons in the beam. The mass spectrometer analyzes the masses (or charge-to-mass ratios) of ionized gas particles in the beam.

Ion Beam Formation and Deflection in the Mass Spectrometer Tube

When tracer gases from leaks first enter the chamber of the mass spectrometer tube of Fig. 1 or Fig. 5a, the gas molecules are uncharged. These neutral gas molecules must first be ionized before they can be effectively controlled with electric or magnetic fields. Within the ion source, randomly moving gas molecules are ionized by bombardment with electrons emitted from a heated filament. The ion source floats at positive potential. The ionized gas molecules are then accelerated toward a grounded plate. As the positive ions pass through a collimating slit in the focusing plate, they are formed into a narrow beam with an energy determined by the ion source voltage. This beam is analogous to the beam of white light in the light spectrometer (see Fig. 3) in that the mass spectrometer beam contains ions of different masses. Separation of differing species of ionized gas molecules is accomplished by directing the ion beam through a magnetic sector. The magnetic field exerts forces on the charged ions that deflect the gaseous ions into circular paths. The path radius of each ion species will depend on the mass of the specific ions. The larger the ion mass, the larger will be the radius of its path within the magnetic field. Ions with the heaviest masses will therefore be deflected least. Ions with the lightest masses will be deflected most and have the smallest radii of path curvature in the magnetic field (see Fig. 5a).

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Magnetic Separation of Ionized Gas Molecules in Mass Spectrometer

Different species of ionized gas molecules within the ion beam of the mass spectrometer are separated by the transverse magnetic field of the magnetic sector tube into a number of discrete circular paths. Each path with a specific radius of curvature contains ionized gas molecules of only one mass. During magnetic separation, each ion beam follows a curved path whose radius is mass dependent. The relative abundance of each spectral mass band in the gas sample is determined by collecting the ions of each band individually, at the collector plate. The desired ion mass is selected by means of the slit in the image plate of the spectrometer. The ions passing through the slit are directed to the collector plate, where each one accepts an electron and becomes a neutral molecule again. The flow of electrons to the plate is then amplified and displayed as visual leak indications on a panel meter. The same current, when its magnitude rises to a preselected high value, can actuate a relay that sounds an audible alarm.

Electrical Scanning of Spectrum of Mass-to-Charge Ratios in Spectrometer

In the light spectrometer sketched in Fig. 3, the distribution of light spectral intensities could be recorded by moving a photocell across the spectrum. This type of mechanical scanning is not possible in the mass spectrometer sketched in Fig. 1 and Fig. 5a. Instead, the collector plate is fixed in position within the mass spectrometer tube and the spectrum is fanned across the slit in the image plate. This sidewise movement of the ion beam spectrum across the image slit is accomplished by varying the voltage applied to the first beam accelerating plate within the spectrometer.

Increasing the accelerating voltage increases the ion energy and produces higher ion velocities in the beam within the magnetic field. Ions passing through the magnetic field at higher velocities are not deflected as much as slower speed ions. The higher speed ions therefore follow curved paths of greater radii. Decreasing the accelerating voltage has the opposite effect; the ions follow paths of smaller radii. Varying the accelerating voltage causes the mass spectrum to be fanned across the stationary image slit and collector target plate.

Permanent Magnet Ion Sorting Systems in Leak Detector Spectrometer

In analytical mass spectrometers, it is not customary to vary the electric accelerating field because this tends to produce so-called mass discrimination effects. Instead, the mass spectrum is scanned by varying the transverse magnetic field intensity with an electromagnet. However, a mass spectrometer expressly designed for leak testing does not have to be capable of scanning a mass spectrum. The leak testing mass spectrometer can be tuned for use with a particular tracer gas by adjusting the electric accelerating field, as discussed previously. In this case, a permanent magnet can be used to provide the magnetic field used to deflect the positive ions into circular paths.

Resolving Power of Helium Mass Spectrometer Leak Detector

Mass spectrometer resolving power that provides clear separation of signals from different gaseous constituents is a critical factor in accurate determination of leakage rate. A leak detector must clearly resolve helium (4 unified atomic mass units [u]) from adjacent hydrogen (2 and 3 u) or carbon (6 u). Hydrogen is usually the most abundant residual gas in vacuum systems and results from dissociation of water vapor (H_2O) by the heated filament. An increase in hydrogen gas levels is typically due to surface outgassing and moisture within the evacuated systems. If the helium leak detector has poor resolution, erroneous signals due to hydrogen can make the leak detector inaccurate in measurements of leakage rates. Therefore, resolving power is a critical feature in mass spectrometer leak detectors where accurate quantitative data are required.

Requirements for Ion Separation by Mass-to-Charge Ratios in Spectrometers

In the following discussion, the term *m/q* indicates the ratio of the molecular mass in kilogram (kg) to the electrical charge in coulomb (C). The fundamental operation

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required of all mass spectrometers is the sorting and identification of positive ions according to their ratios of *m/q*. Three conditions must be achieved for this sorting to be possible.

1. The chamber of the mass spectrometer tube must be evacuated to such a level that the mean free path of gaseous particles is significantly longer than the distance individual particles must travel within the tube. If particles can collide with other gaseous particles, the ion beam would be dispersed and the ions attempting to follow circular orbits would be deflected into random paths, reducing both resolution and sensitivity of the mass spectrometer.
2. A portion of the gas molecules must be ionized to permit (a) the accelerating field to bring the ions to the proper velocity (remembering that the ion beam must be monoenergetic) and (b) the magnetic field to apply deflecting forces to cause the ion particles to travel in circular orbits. Electron bombardment should result in single ionization of nearly all gaseous particles. This single ionization occurs when only one valence electron is knocked out of the orbital electron cloud of the atom or molecules. This leaves each positive ion with a charge of the same magnitude, equal (but opposite in polarity) to the charge on one electron, 1.6×10^{-19} C. Only with single ionization can determination of the mass-to-charge ratio *m/q* serve to separate ions of gaseous atoms in accordance with the atomic masses *m* of the individual species.
3. To provide clear cut separation of each gaseous species in the ion beam of the mass spectrometer, each charged ion should have been accelerated through the same electrical potential drop *V* so as to form a well defined, monoenergetic ion beam. In addition, this ion beam should have particle velocities at right angles to the direction of the uniform magnetic field, in order that the ions be forced to follow true circular paths during magnetic separation of gaseous species.
4. To attain adequate resolution of ions, the ion beam should be well collimated, be shaped in a narrow slit and pass through additional slits and baffles so that only ions of the desired tracer gas can pass through slits to the ion collector to form output signals. However, scattering and bouncing off other gaseous ions may permit some ions of other gases to get past these baffles. For this reason, some manufacturers of leak testing mass spectrometer equipment use a second magnetic deflection field identical to the first magnetic field, to again deflect the desired species of charged ions through a second slit and baffle system to further reject ions of undesired gases

Radial Force Makes Ions Follow Circular Path in Magnetic Field

Singly charged positive ions, like any moving mass, travel in straight lines if no forces act on them. When these ions are passing through the magnetic field of the mass spectrometer, they are subject to a force that will bend their path into a circular sector. Because the force is acting toward the center of the circle, it is called the centripetal (or inward) force. The following two equations may be used to calculate the path of an ion. Equation 2 gives force for inward acceleration of mass and Eq. 3 gives electromagnetic force:

$$F_1 = \frac{mv^2}{r} \quad (2)$$

$$F_2 = Bvq \quad (3)$$

where q is ion positive charge, equal to $+1.6 \times 10^{-19}$ C; m is ion mass, typically atomic mass (kilogram); r is radius of ion path (meter); v is ion velocity after acceleration in electric field ($m \cdot s^{-1}$); and B is magnetic flux density in magnetic field ($Wb \cdot m^{-2}$). Equation 4 follows from $F_1 = F_2$:

$$\frac{mv^2}{r} = Bvq \quad (4)$$

Solving Eq. 4 for the ratio of ion charge q to ion mass m results in Eq. 5 for the separation of ions by their charge-to-mass ratio q/m (coulomb per kilogram):

$$\frac{q}{m} = \frac{v}{rB} \quad (5)$$

Alternatively, Eq. 4 could be solved for the radius r of the ion path in magnetic field (meter):

$$r = \frac{mv}{Bq} \quad (6)$$

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Velocity of Positive Ions after Electrical Acceleration in Mass Spectrometer

As a singly ionized positive ion enters the accelerating electrical field of the mass spectrometer sector tube, it gains kinetic energy as it accelerates through the potential drop V of the electric field. Equating these two energy changes results in Eq. 7 for ion energy balance during acceleration ($m \cdot s^{-1} \cdot s^{-1}$):

$$\textbf{(7)} \quad Vq = \frac{mv^2}{2}$$

Solving Eq. 7 for the ion velocity v leads to Eq. 8, for ion velocity ($m \cdot s^{-1}$) after acceleration:

$$\textbf{(8)} \quad v = \sqrt{\frac{2Vq}{m}}$$

Radius of Ion Path in Magnetic Field of Mass Spectrometer

If the value for ion velocity from Eq. 8 is substituted for v in Eq. 6, the radius r of the ion path in the magnetic field is given by Eq. 9 for radius of ion path in magnetic field (meter):

$$\textbf{(9)} \quad r = \frac{\sqrt{\frac{2}{q}}\sqrt{mV}}{B} = \left(3.53 \times 10^9\right)\frac{\sqrt{mV}}{B}$$

Ratio of Mass-to-Charge for Spectrometer Tube with Fixed Radius

In a mass spectrometer tube with fixed geometry (commonly used for leak testing), the ion path radius at which ions traveling along the circular path will strike the collector plate is a fixed radius r_o. In this case, the equation for the ratio m/q of ion mass to charge is derived from Eq. 9 in the form of Eq. 11 (kilogram per coulomb):

$$\textbf{(10)} \quad \frac{m}{q} = \frac{r_0^2 B^2}{2V}$$

Equation 10 indicates that the particular mass of a singly ionized particle striking the collector plate depends on the intensity of the magnetic field B and the accelerating voltage V. From Eq. 10, it can be seen that a mass spectrometer can be tuned for a particular ion mass by varying the magnetic field B alone, the accelerating voltage V alone or both B and V.

Simplified Equation for Fixed Geometry and Magnetic Field Spectrometers

For a mass spectrometer with a fixed magnetic field intensity B, Eq. 11 gives the ion mass-to-charge ratio (u per electron charge):

$$\textbf{(11)} \quad \frac{M}{e} = \frac{K_{ms}}{V}$$

In Eq. 11, the mass M of the positive ion is given in atomic mass units (u). The charge on the positive ion is given in units equal in magnitude to the charge on one electron, 1.6×10^{-19} C. The term K_{ms} is a characteristic constant of the particular mass spectrometer (with fixed geometry and magnetic field intensity that is selected for use). With this type of instrument, the ion mass that strikes the target depends only on the accelerating voltage V. To determine the existence of ions with different masses in the leak testing tracer gas sample, one could simply adjust the accelerating voltage V of the mass spectrometer. For example, if a mass spectrum scan were being made with an instrument whose constant $K_{ms} = 1200$ and a signal peak had appeared at a voltage $V = 300$ V, then from Eq. 11 the ratio of ion mass M in atomic mass units (u) to ion charge in units of electron charge e is $M/e = 4$. In this case, helium tracer gas would have been detected.

Conversion of Ion Beam into Electrical Leakage Signals[1]

After their separation in the magnetic field of the mass spectrometer, ions of a specific mass (or, more exactly, ions with a specific mass-to-charge ratio) can be selected to strike a target or ion collector located beyond the slit (Fig. 1 or 5). Each singly ionized ion that strikes the collector plate carries a net positive charge (because it lacks one electron in its orbital

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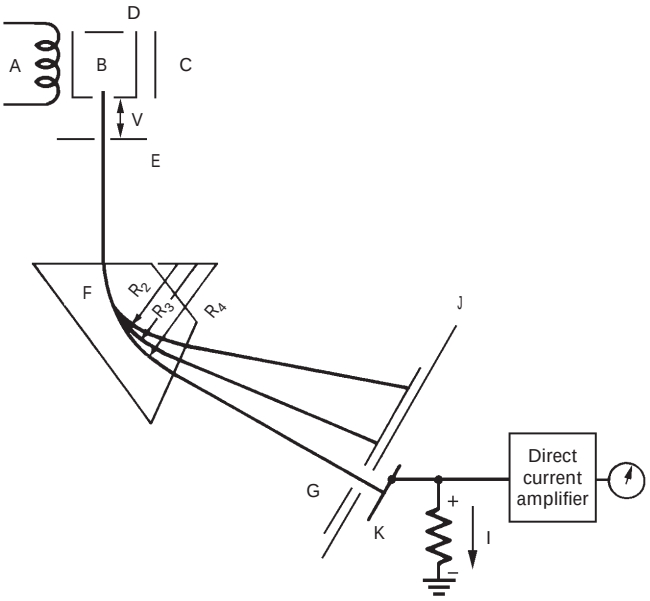
FIGURE 7. Mass spectrometer leak detector, capable of integration into production line.

FIGURE 8. Schematic of ion beam path during mass spectrometer tube operation.



Legend

A = Heated electron source
B = Ionization chamber
C = Anode
D = Repelling plate
E = Grounded plate
F = Magnetic field
G = Ion separating plate
I = Electrical current
J = Suppressor plate
K = Ion collector plate
R = Circular orbit of ions
V = Ion accelerating voltage

electron cloud). When the positive ion strikes the collector plate, an electron is removed from this target to neutralize the charge on the arriving positive ion. Each arriving positive ion then causes a minute current to flow to the collector. This collector current is then amplified by the electrometer stage that is often placed within the high vacuum enclosure to ensure signal stability, minimize the electrical time constant and reduce the stray noise pickup.

This amplified signal current is then typically displayed on a readout display of the mass spectrometer leak testing instrument. Figure 7 shows a mass spectrometer that can be integrated into the vacuum system of a high speed production line, small parts system. The system enables the repetitive testing of mass produced parts in as little as several seconds per part.

Operation of Ion Beam Gun in Mass Spectrometer Tube

The source for the ion beam is a chamber within the spectrometer tube; the chamber is exposed to helium and other gases at a low pressure. This permits ions and electrons to travel considerable distances before collisions with other gaseous particles or ions. A beam of electrons of stabilized intensity emitted from a heated filament (A in Fig. 8) is attracted by a potential difference of a few volts toward an ionization chamber B. The electron beam, passing through this chamber, collides with neutral gas molecules or atoms, ionizing them by knocking an orbital electron out of the atomic field. The surviving electrons are collected on a positively charged electrode, anode C. The mass spectrometer unit receives air or other gases from the component being leak tested, as well as helium tracer gas. Therefore, the electrons, on their way through the ionization chamber, produce positive ions from each of the types of gas molecules, including the tracer gas.

A repelling plate D covers one end of the ionization chamber B (see Fig. 8). This plate D carries a positive charge and repels the positive ions toward the gun orifice. Ions escaping from the ionization chamber B through this orifice are accelerated further by the ion accelerating voltage *V* applied between plates B and E. The ions that pass through the collimating slit in the grounded plate E emerge in the form of a narrow ion beam. This beam typically contains ions of many species of gaseous particles in air, contaminant gases and (when leakage is detected) the tracer gas.

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Deflection and Sorting of Ion Beam in Magnetic Field of Sector Tube

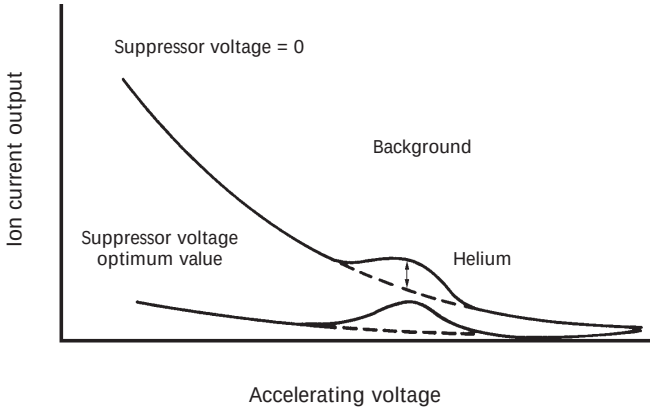
Mounted on the mass spectrometer tube chamber, a permanent magnet (shown as a trapezoid F in Fig. 8) separates ions by mass-to-charge ratio. Under the influence of this magnetic field of intensity *H*, the positive ions in the beam are made to follow circular orbits, such as R_2, R_3 and R_4 of Fig. 8. After leaving the magnetic field, the positive ions again follow straight line paths at the angle to which they were deflected in the magnetic field. They eventually encounter an ion separating plate G containing a slit that selects only singly ionized helium atoms to pass to the collector plate K. The centers of slits G and E (of Fig. 8) lie at opposite ends of a 60 degree sector of 40 mm radius. The magnetic field intensity H is about 19 $A \cdot m^{-1}$. When the accelerating voltage is about 440 V (in this specific instrument), singly charged helium ions with mass of 4 u can pass through the slit G. Behind the slit G lies a suppressor plate J, also equipped with a slit, and the collector plate K. Helium ions arriving at this collector plate constitute the leakage signal of the mass spectrometer. The electric charge carried to the collector by the helium ions produces the output electrical current that actuates the leak detector's indicating instrument.

Amplification of Electrical Signal in Electrometer of Leak Detector

The electric current output from the collector plate K of Fig. 8 is conducted to ground through a 100 GΩ resistor. The signal voltage developed across this high resistance is applied to the input terminal of an electrometer tube or semiconductor circuit. Both the electrometer and the high value resistor are mounted in the vacuum system of the collector assembly. The output signal voltage from the electrometer is then amplified further and produces a deflection on the leakage rate meter or digital indicator. This meter indication is proportional to the partial pressure of helium within the mass spectrometer system, if helium tracer gas is used and only helium ions reach the collector plate K.

Mounting the first stage of the signal amplifier inside the mass spectrometer tube keeps the high resistance and the electrical connections dry to minimize leakage currents. It also makes the lead wires as short as possible from the collector plate to the grid of the first (electrometer) tube and provides electrostatic shielding for the input stage. The remainder of the electrical signal amplifier is mounted in the control panel of the leak detector.

FIGURE 9. Effect of suppressor voltage on ion current output.



Suppression of Ions Causing Background Electrical Signals in Spectrometer[1]

A suppressor plate J is placed just in front of the collector plate K, in the mass spectrometer tube sketched in Fig. 8. This suppressor plate is operated at a potential near that of the ionization chamber so that those ions that lose energy by collision cannot pass through the slit in the suppressor plate to reach the collector plate. Without the suppressor voltage, a mass spectrometer instrument operating with a small concentration of helium in air would give a mass spectrum in the region of helium such as that shown by the upper curve in Fig. 9. The sloping background of the upper curve of Fig. 9 is due to ions that have been slowed down or deflected by collisions with other gaseous particles during transit from the ion source and that by chance happen to pass through the collector slit. All such scattered ions have less than normal kinetic energy because each collision results in a loss of the energy of the accelerated ions. A potential barrier to stop scattered ions can be created by applying a positive suppressor potential, essentially equal to the ion accelerating voltage V, on the suppressor plate J. When this is done, the resultant spectrum of ion energies is similar to that shown by the lower curve of Fig. 9. With the much reduced background signal, the presence of helium is much more easily discerned than in the mass spectrum of the upper curve.

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PART 2. Sensitivity and Resolution of Mass Spectrometer Helium Leak Detectors

Factors Controlling Leak Detector Sensitivity and Resolution

Many design and operating factors can influence the sensitivity and resolution of helium mass spectrometer leak detectors when used in industry. Leak testing reliability and minimum detectable leakages are determined by the mass spectrometer instrument capabilities and the conditions under which it is operated. The gas pressure that exists in the vacuum system of the spectrometer and the purity of the gas that enters the mass spectrometer ionization chamber are controlling factors that must be understood and reproduced during leak testing operations.

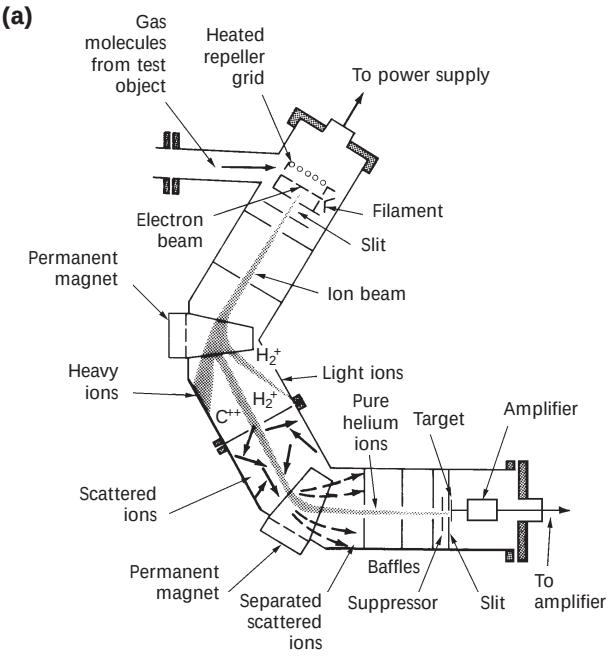
Effects of Excessive Gas Pressure in Mass Spectrometer Tube

Excessively high pressure in the mass spectrometer tube gives rise to spurious signals due to scattering of the separated ions back into the ion collector. At a pressure of 10 mPa (0.1 mtorr), the mean free path (the average distance a gas molecule will travel before colliding with another gas molecule) is about 0.5 m (20 in.). Even with no tracer gas present, the amplifier will show a signal due to scattered ions. The signal due to ion scattering at this pressure is likely to limit the minimum detectable leakage of 60 degree magnetic sector commercial mass spectrometer instruments to about 5×10^{-11} to 1×10^{-10} Pa·m^3·s^{-1} (5×10^{-10} to 1×10^{-9} std cm^3·s^{-1}). This problem can be surmounted through two stages of separation in the mass spectrometer (Figs. 10 and 11) and has been surmounted by deflecting the ions in a magnetic sector of 90 degrees or more in (Fig. 12). Resolving power can be measured in accordance with an American Vacuum Society practice.[2]

Effect of Ion Source Pressure on Sensitivity of Helium Mass Spectrometer

The sensitivity of any particular mass spectrometer leak detector is a function of (1) total gas pressure and (2) helium concentration in the detector tube. Leak detector sensitivity can be improved by lowering the pressure in the vacuum system including the mass spectrometer tube. Depending on the individual instrument, the sensitivity tends to become much more nearly constant

FIGURE 10. Mass spectrometer leak testing system using two stages of magnetic separation of gaseous ions to reduce background noise and improve minimum detectable leakage rate and resolution: (a) schematic; (b) photograph.



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below a pressure in the range of 0.1 Pa (1 mtorr). If excessive outgassing or leaks in the system being tested raise the system pressure above a critical value in the region of 1 Pa (10 mtorr), leak sensitivity worsens rapidly. In such situations, auxiliary pumping (in addition to vacuum pumping by the leak detector) can be used to reduce pressure in the test system and mass spectrometer tube. However, such auxiliary pumping will also reduce leak test sensitivity.

If a detector probe has too large an inlet orifice or if the pressure is too high in a vacuum system to which the instrument is connected, pressure in the mass spectrometer leak detector will rise to the 1 Pa (10 mtorr) region. Leak test sensitivity will then decrease rapidly. Thus, a compromise must be sought between an excessively large and an inadequate size of detector probe opening.

Cold Trap to Avoid Contamination of Ion Source in Spectrometer

In a direct flow leak detector, the purpose of using a cold trap in the inlet to the vacuum system of the direct flow mass spectrometer is to condense vapors such as water and oil and to entrap these vapors by condensation onto a cold surface. This reduces the vapor pressure of these constituents to a negligible value. Cryogenic pumping is desirable. Because it is not a pump for helium tracer gas, but is used only for condensables, it is a very selective pump source. For example, the vapor pressure of water at 20 °C is 2.3 kPa (17.5 torr). However, water vapor pressure is about 10^{-13} Pa (10^{-15} torr) at the temperature of –196 °C (–320 °F) for liquid nitrogen. The lower the temperature of the cold trap, the more effective it is in reducing pumpdown time and assuring a vapor free vacuum system.

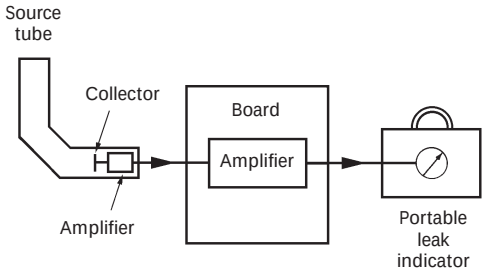
Good practice requires that the refrigerant liquid in the cold trap be kept at a reasonable level. If the cold trap is neglected and allowed to go dry, the most noticeable immediate effect would be the inability of the mass spectrometer system to maintain adequately low pressures during leak testing operations. However, such neglect should not become a routine operating practice because the operating efficiency of the mass spectrometer system is considerably reduced. With inadequate cold trap decontamination of inlet gases, the direct flow mass spectrometer system will exhibit a high signal background and short filament life.

The ion sources of the mass spectrometer analyzer tubes become dirty due to decomposition products of organic molecules that migrate into the ionization chambers. These are then readily decomposed by electron bombardment. The primary need for the cold trap is to prevent this migration of contaminants from the atmosphere or the systems being leak tested into the mass spectrometer tube. Certain commercial instruments use a platinum clad ion repeller that may be cleaned after system shutdown by immersion in a soft flame. Manufacturers' instructions for maintenance and cleaning of spectrometer tubes should be followed routinely as good practice. In the counterflow leak detector, operation without a cold trap reduces the possibility of contamination.

Effects of Atmospheric Leakage into Mass Spectrometer Tube

Leakage of atmospheric air into the mass spectrometer system can result in high pressure within the mass spectrometer tube and, in addition, can give rise to a helium background signal. Atmospheric air with 1 part helium in 200 000 parts of air (5 $\mu L \cdot L^{-1}$) can, at 10 mPa (0.1 mtorr) pressure in the vacuum system, produce a signal on the helium leak detector that can be perhaps 10 to 100 times larger than the minimum detectable leak signal of the instrument. This is one of the basic limitations of the helium detector probe technique. Few gases are rarer in normal atmosphere than helium. However, if the leak test uses argon tracer gas, the effect would be much more serious because the normal argon concentration in air is 1 percent.

FIGURE 11. System diagram of helium mass spectrometer leak detector.



Construction of Mass Spectrometer Tube for Portable Leak Detector

Figure 12 shows a schematic diagram of the design of a mass spectrometer tube designed for use in a portable helium leak detector. The components of the spectrometer tube system are combined in

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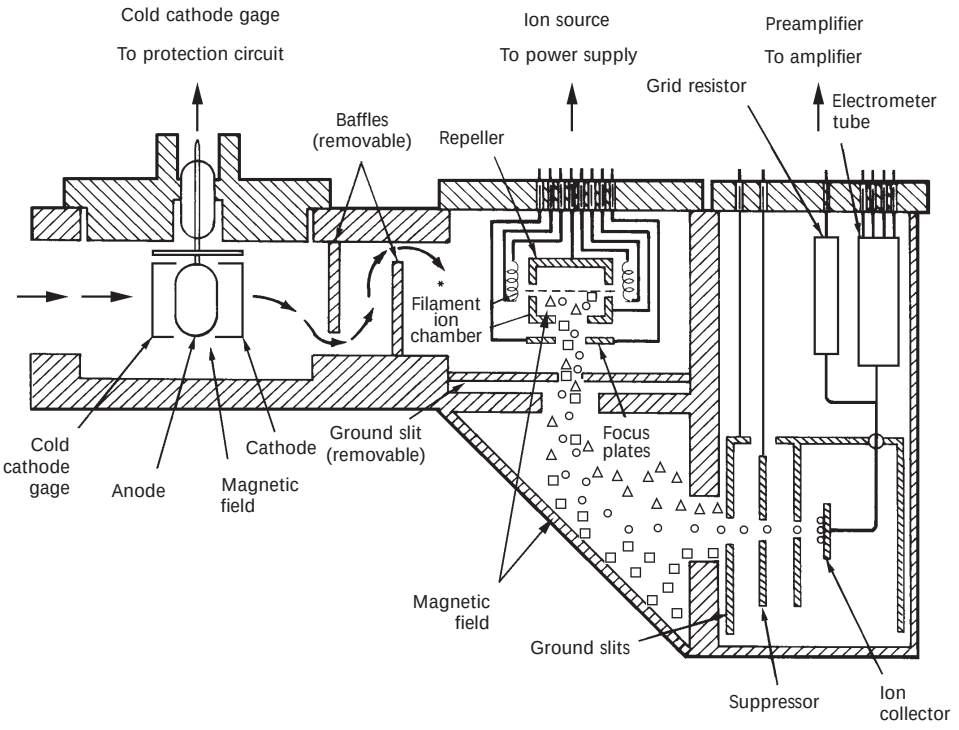
a single assembly. This spectrometer tube is attached by means of an integral O-ring coupling at the inlet to the diffusion pump of the leak tester. It contains an electron source, an ion gun, an ion collector and a preamplifier within its single, compact assembly. The magnetic field for this spectrometer tube is provided by inserting a permanent magnet into the tube with O-ring seals.

Ion Source for Portable Mass Spectrometer

The ion source for the spectrometer shown in Fig. 12 is a one piece expendable unit consisting of the following parts: (1) two permanently aligned tungsten filaments that can be used alternately and that provide a source for ionizing electrons; (2) an ionization chamber in which electrons are beamed and in which gas molecules are struck by electrons and become positive ions; (3) repeller electrode that repels the positive ions so they escape through the beam forming slit in the ionization chamber; and (4) two focus plates that direct the ion beam toward the exit slit, which is at ground potential.

These parts of the ion source are welded to eight rods that extend through individual glass seals in a round flange to form the male portion of a standard octal connector. A clamp and O-ring are used to seal the assembly into the spectrometer tube. This construction permits easy servicing of the spectrometer tube. The spare tungsten filament allows leak testing to continue after one filament burns out. In addition, no cleaning or disassembly of the ion source is necessary. The ion source is inexpensive and easily replaced. All parts of the source unit are prealigned and the unit itself is keyed to the spectrometer tube so that no special skill is required to replace it. Rotatable external eccentric magnetic poles on each side of the ion source allow adjustment of the electron (ionizing) beam direction for optimum ionization and sensitivity.

FIGURE 12. Compact mass spectrometer tube assembly for portable helium leak detector. Note cold cathode pressure gage and electrometer signal amplifier tube included in single replaceable tube assembly. Filaments are shown 90 degrees out of place for clarity.



Legend

△ = Low mass ions (hydrogen)
○ = Helium ions
□ = High mass ions (O_2, N_2, CO, CO_2 etc.)

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Magnetic Deflection System for Spectrometer

The ion beam deflection into circular paths occurs after the ions pass through the ground potential exit slit of the ionization chamber and travel between the magnetic pole pieces. When the spectrometer is properly tuned, helium ions with mass of 4 u are deflected through 90 degrees and pass through ion separating slits in the baffle plates. Heavier ions are deflected through angles less than 90 degrees; hence separation of helium ions is accomplished.

Ion Collector and Preamplifier in Spectrometer

In the spectrometer tube shown in Fig. 12, the preamplifier consists of the ion collector, solid state amplifier and resistor, together with two ground slits for ion separation and a suppressor slit. This preamplifier assembly is mounted as a unit on eight rods that extend through individual glass seals in a round flange to form the male portion of the preamplifier section connector. This unit is prealigned in the factory and is of all welded construction. It is sealed in place within the spectrometer tube with a clamp and O-ring. Removal and replacement, when necessary, is quick and easy.

The cold cathode ionization gage of the electrometer shown in Fig. 12 consists of two magnetic pole pieces, a liner that forms the cathode and a nickel chrome loop that forms the anode. This assembly is mounted on a single ceramic insulator. A ceramic disk shield prevents sputtered conductive deposits from causing leakage paths across the anode lead-through insulator. The ionization gage assembly seals in place in the spectrometer tube assembly with an O-ring. The magnetic field of the cold cathode ionization gage is provided by the magnet, which serves also to create the ion beam deflection field and the magnetic field applied to the ionization chamber.

Function and Operating Ranges of Pressure Gages Used on Spectrometers

Mass spectrometer leak detectors usually use two types of gages for measuring vacuums.

1. Pirani or thermocouple gages are used for low vacuum measurement, from atmospheric pressure down to 0.1 Pa (1 mtorr).
2. Ionization gages are used for measurement of pressures in the high vacuum range (less than 10 mPa or 0.1 mtorr).

The Pirani or thermocouple gage indicates pressure in the test port. This indicates when the diffusion pump line may be safely opened. The ionization gage, if used, is usually a cold cathode discharge gage. The ionization gage is more rugged than a hot filament ionization gage and does not contain a hot filament that could burn out if exposed to atmosphere.

Principle of Operation and Automatic Control by Cold Cathode Gage

In the cold cathode ionization gage, the discharge current results from the application of high voltage between anode and cathode. The discharge current magnitude is a function of the gaseous pressure within the gage chamber. The external permanent magnet facilitates ionization by forcing the electrons into a spiral path between the two electrodes. The discharge current is displayed on a meter on the control panel and usually is monitored by a filament protection circuit. This protective circuit senses when the pressure exceeds safe operating levels and then instantaneously removes the heater power from the filament in the ionization chamber of the spectrometer. In some automatically operated leak detectors, the protective circuit will shut valves of the mass spectrometer leak detector if the spectrometer tube pressure rises above a safe operating level.

Design and Performance Characteristics of Leak Signal Amplifier

The need for maximum overall sensitivity suggests the most sensitive available means of ion current detection. However, the signal amplifier must also meet the important requirements of ruggedness, portability and simplicity of operation. The mass spectrometer signal amplifier, when used with an input resistance of 10 GΩ and an output indicating instrument rated for 5 V full scale deflection, can measure ion current magnitudes of the order of 10^{-14} A. This first stage is then followed by additional stages of voltage gain. The output voltage operates a bar graph display or can be processed by a computer.

Effect of Signal Noise, Drift and Signal-to-Noise Ratio on Sensitivity

The maximum sensitivity of mass spectrometer leak detectors is limited by fluctuations in the helium background in the spectrometer tube and the characteristics of the electronic circuitry (usually in the amplifiers) used to measure

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the helium ion current. This shows up as a random fluctuation in the electronic signal output and is indicated by an erratic motion of the detector output meter. Drift shows up as a gradual wandering of the output meter. The drift for most commercial mass spectrometer leak detectors is usually less than 0.5 percent of full scale per minute, on the output meter. This type of drift usually introduces no particular problem, although it may become necessary to occasionally zero the output meter while locating leaks.

The combination of the effects of noise and drift determines the minimum detectable leak signal, the smallest readable deflection (in terms of meter scale divisions). This minimum detectable leak signal is specified for the most sensitive scale setting of the leak detector output meter. Often, this smallest detectable signal is arbitrarily taken as a deflection three times as large as the mean peak-to-peak noise fluctuations of the output meter, averaged over ten successive fluctuations. In this case, the signal-to-noise ratio is 3:1.

Interpreting Significance of Signal Indications of Helium Leak Detector

The visible light flashes or audible loudspeaker signals of the mass spectrometer leak detector indicate only the presence or location of leaks and not quantitative leakage rates. On the other hand, the meter indications obtained from the positive ion current signal of the mass spectrometer do permit quantitative calculation of leakage rates, when correlated with standard leak signals. However, the significant signals originate from helium atoms that enter the source chamber of the mass spectrometer from the inlet hose of a detector probe or through direct connections to the interior volumes of a system under test. Significant signals due to leakage of helium tracer gases through test object pressure boundaries must be differentiated from nonsignificant meter deflections caused by electronic noise, drift, valve systems or varying background helium concentrations in the test area.

The minimum detectable leakage rate is defined as that leakage rate that produces an output meter signal that can be unambiguously interpreted as due to a leak. Drift and noise are the most common causes of ambiguous signals. Background helium molecules in the leak testing area can also lead to confusion in interpretation of leak test signals. When such ambiguous signals have been identified and their sources eliminated, the remaining valid leakage signals must still be interpreted with caution. The output meter of the mass spectrometer leak detector merely indicates the magnitude of an electrical signal proportional to the partial pressure of helium tracer gas present in the source chamber of the mass spectrometer tube. This helium partial pressure may or may not be proportional to leakage rate, depending on several test variables and operating conditions.

For example, the partial pressure of the helium tracer gas in the mass spectrometer source chamber is directly proportional to the leakage rate when all other test conditions are held constant. However, the partial pressure of helium varies in inverse proportion to the pumping speed of the high vacuum system. Changes in inlet orifice size, in length of hose between probe and source chamber or in helium concentration in the test vessel, could cause changes in meter indications. In-leakage of atmospheric air containing helium, or ingestion of high helium background contamination through the detector probe, could also lead to ambiguous or invalid leak indications on the output meter.

Establishing Quantitative Leakage Rate Calculations with Standard Helium Leaks

Comparison tests in which the mass spectrometer leak detector is checked and calibrated with a standard helium leak of known leakage rate are essential for quantitative interpretation of leakage rates. A leak standard is allowed to leak helium into the interior volume of the evacuated system under test, so that the leak standard controls the helium partial pressure within the source chamber of the mass spectrometer. Other adjustments of the leak detector system are held constant when the standard leak indication is compared with that for the unknown leak. When the system under leak test is filled with 100 percent helium tracer gas or a hood or outer chamber is filled with 100 percent helium gas that leaks into an evacuated test vessel, the meter indications will be proportional to standard helium leakage rates. If it is desired to estimate the equivalent leakage of air or of other gases, the precautions indicated in the discussion of mass spectrometer leak detector sensitivity must be considered.

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Enhancing Helium Leak Detector Sensitivity by Throttling of Pumping Speed

A higher helium partial pressure in the ion source of the mass spectrometer and a correspondingly greater signal deflection on the meter can be attained by the technique of throttling the vacuum pumping speed. The accumulation technique is usable only when dealing with very small leaks and well outgassed systems so that the leak detector vacuum system does not overpressure, because pumping speed is greatly reduced. If this technique is necessary, the leak detector manufacturer should be consulted because many designs have been used with varying degrees of success.

Reduction of Mass Spectrometer Leak Test Sensitivity at High Pumping Speed

The leak testing sensitivity of the helium mass spectrometer leak detector is reduced as system pumping speed increases. If the mass spectrometer tube alone were to be attached directly to the chamber of a large vessel where very high pumping speeds are involved, the leak rate sensitivity would be greatly reduced. For example, a mass spectrometer tube that could detect 1 pPa (0.01 ptorr) partial pressure of helium would, when attached to a space simulator with a pumping speed of 100 $m^3 \cdot s^{-1}$ (2.12×10^5 $ft^3 \cdot min^{-1}$), be able only to detect leaks larger than 10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-6} std $cm^3 \cdot s^{-1}$).

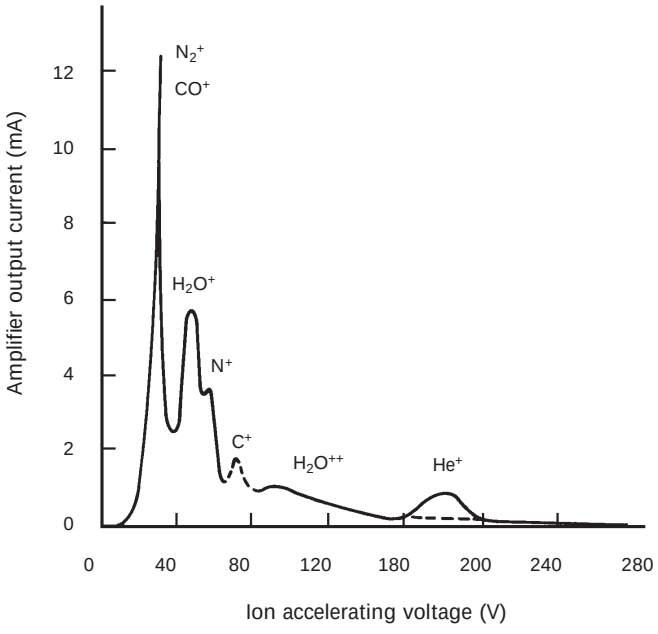
Pumping Capacity Limitations of Mass Spectrometer Vacuum System

The throughput of a vacuum system is a measure of the mass flow of gas being handled by the vacuum pumps. Throughput is equal to the product *PS* of the total pressure *P* and the volumetric pumping speed *S* at that operating pressure. Therefore, throughput is increased by operating at high pressure and with high pumping speed. However, the maximum pressure permitted by mass spectrometer limitations is usually in the range of 30 to 40 mPa (0.2 to 0.3 mtorr). This limits the throughput of the mass spectrometer leak detector to about 4×10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (3×10^{-3} torr $L \cdot s^{-1}$). If the diffusion pump is throttled as in dynamic testing, the gas handling capacity (or throughput) will decrease. If the gas load of the item under test is larger than the allowable throughput of the leak detector, the test item must be differentially pumped. This bypasses some of the gas load. This also results in loss of test sensitivity as some of the tracer gas is bypassed. However, loss of sensitivity is usually no problem under these circumstances if the leaks being sought are large enough to build up helium in the system. A sensitivity calibration on a differentially pumped system can be in large error unless the calibrated leak is mounted in the same position in the differential pumping arrangement as the leak being measured.

Spectral Characteristics of Helium Mass Spectrometer Output Signals

Figure 13 shows a typical mass spectrum recorded by sweeping the ion accelerating voltage to include helium and some of the heavier ions. If quantitative leakage measurement is required, the helium peak in the mass spectrum must be measured accurately and the background signal (shown by dashed line) must be subtracted from this peak signal. The background tail results from ions of other gases present within the mass spectrometer tube. This background signal becomes particularly large at high gas

FIGURE 13. Typical mass spectrum taken with a mass spectrometer leak detector.



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pressures within the spectrometer, 1 to 100 mPa (0.01 to 1 mtorr). Background is reduced somewhat by the suppressor plate, but rarely can it be entirely eliminated.

The usual practice when testing is to electronically zero out the background before exposing the leak detector to helium from the test object. However, if this background is unstable, it will be difficult to differentiate between these instabilities and a leak signal response.

Response Time of Helium Mass Spectrometer Leak Detectors

An additional factor that deserves serious consideration in estimating the sensitivity of helium mass spectrometer leak detectors is response time. Response time constant is the time required, after exposure to the source of tracer gas, for the leak detector or leak detection system to yield an output leak signal magnitude equal to 63 percent of the maximum signal attainable when the tracer gas is applied to the system under test for an indefinitely long period of time.

The factors controlling the response time of the leak detector include the following subsystem time constants, which act in combination to determine the overall time constant for the leak detection system: (1) the mass spectrometer's electronic signal system time constant and (2) the mass spectrometer's vacuum system time constant, which decreases with increasing pumping speed and increases with increasing lengths of detector probe hose. (The mass spectrometer's electronic time constant for both direct flow and counterflow is in the millisecond range and of no significance.)

The vacuum system time constant for a direct flow leak detector varies with the amount of throttling used but may be longer than several minutes. If the system is used in the detector probe mode, the vacuum system time could be as much as 30 s, depending on probe length. With a counterflow detector, time constants as short as 2 s can be achieved with probes 15 m (50 ft) long.

The instrument's response time is usually the determining factor in setting the scanning speed of the helium sampling or detector probe. A typical probe scanning speed is about 2 $cm \cdot s^{-1}$ (4 $ft \cdot min^{-1}$).

A far higher sensitivity is attainable when an accumulation test is performed with a detector probe whose collecting tip penetrates inside a small hollow rubber bell placed over the leak area of the test object. If accumulation of leaking helium tracer gas is carried out for a period of 0.5 to 15 min, the increase in leak testing sensitivity may exceed 100 000 to 1. Thereafter, during inspection, the bell detector probe assembly is moved to cover another area of the test object (such as a weld) and again held in a position overlapping the preceding accumulation test position. Note that an additional time constant that affects the feasible speed of inspection and potential sensitivity of the helium leak detection operation is the time constant of the physical leak itself.

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PART 3. Operation and Maintenance of Mass Spectrometer Vacuum System

Components of Vacuum System of Mass Spectrometer Leak Detector

The mass spectrometer tube operates under a high vacuum. The vacuum system of the direct flow mass spectrometer leak detector contains the following components to provide the vacuum necessary for operation of the spectrometer tube (Figs. 2c and 14).

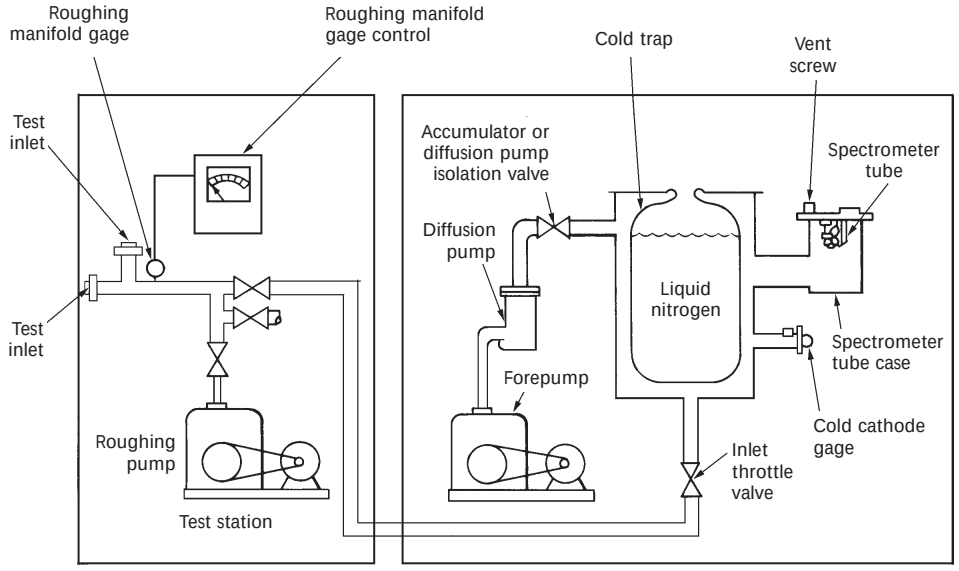
1. A vacuum pumping system evacuates the analyzer tube and associated vacuum lines for handling the sample gas collected from the leak.
2. A cold trap removes condensable vapors from the gas sample before it passes into the mass spectrometer tube (direct flow only).
3. Appropriate vacuum coupling connects a standard leak to the object to be tested.
4. Flanges connect the leak detector to test objects or systems to be leak tested.
5. Valves control evacuation of test objects (in direct flow systems, the throttle valve and diffusion pump isolation valve, also known as the accumulator valve, are used for special test techniques).
6. Vacuum gages provide information on pressure.
7. A leak detector pumping system is used to evacuate the test objects.

Counterflow Leak Detector

The counterflow leak detector (Figs. 2a and 2b) takes advantage of the differences in compression ratios (outlet pressure divided by inlet pressure) produced by the diffusion pump for gases of different molecular weights. For example, the maximum compression ratio for helium may be 10:1 or 100:1, whereas for oxygen, nitrogen and other gases contained in air, the ratios are normally far in excess of one million to one. This is typical of most mass spectrometers used in production in the 1990s.

The counterflow principle is implemented in the leak detector by introducing helium into the diffusion pump outlet (foreline) rather than into

FIGURE 14. Older mass spectrometer leak detector pumping system. Note belt driven pumps in 1970's design.



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the "normal" pump inlet, as in direct flow leak detectors. Helium, having a much lower maximum compression ratio than other gases contained in air, diffuses backwards through the diffusion pump to reach the spectrometer tube where it is detected in the normal manner. Although the mechanical pump is also attached to the foreline and removes all inlet gases, including helium, there is no appreciable loss of sensitivity in the counterflow leak detector. The main advantage of the counterflow leak detector is the ability to leak test at about 100 times higher test port pressure. The sensitivity of the automatic direct flow model is 10^{-12} Pa·m^3·s^{-1} (10^{-11} std cm^3·s^{-1}) whereas the sensitivity of the counterflow is 10^{-11} Pa·m^3·s^{-1} (10^{-10} std cm^3·s^{-1}). However, at test pressures greater than 1 Pa (10 mtorr), the counterflow leak detector will be more sensitive than the direct flow one.

Vacuum Pumping System of Mass Spectrometer System

A complete, self-contained vacuum pumping system must be provided for the proper operation of the mass spectrometer tube of the helium leak detector system. Two levels of vacuum pumping are used.

1. A mechanical pump for fast removal of large quantities of gas from smaller test objects and from the mass spectrometer. The mechanical pump is used for initial pump down operations in the pressure range from atmospheric down to 1 Pa (10 mtorr).
2. A diffusion pump for operation at absolute pressures from 1 Pa (10 mtorr) down to the high vacuums required for operation of the mass spectrometer tube. These pressures are typically a factor of 1000 times lower. The diffusion pump is turned on only after the forepump has reduced system pressures adequately, to 10 Pa (0.1 torr) or less for proper operation of the diffusion pump. The diffusion pump must also be valved off or turned off before exposing the leak detector to atmospheric pressures on the completion of operation under vacuum conditions.

Automatic Trapping Action

In the counterflow leak detector (Figs. 2a and 2b), the diffusion or turbomolecular pump, by optimizing the compression ratio for helium and the other gases of heavier molecular weights, acts as a filter that prevents the other gases and contamination, such as water vapor, from reaching the spectrometer tube. These gases and contaminants are introduced by the connection of the test piece to the leak detector. The filtering action of the diffusion pump eliminates the need for any cryogenic trapping.

A diffusion pump used in this fashion also acts as a buffer, protecting the spectrometer tube from pressure bursts that would normally endanger the mass spectrometer tube and trigger protective devices. Interruption of testing due to pressure bursts is less frequent and the unit can be used at higher pressures, up to about 70 Pa (0.5 torr), as high as atmosphere in the gross leak mode, allowing the measurement of gross leaks (leakage rates generally more than 10^{-4} Pa·m^3·s^{-1} or 10^{-3} std cm^3·s^{-1}) without need for special test techniques.

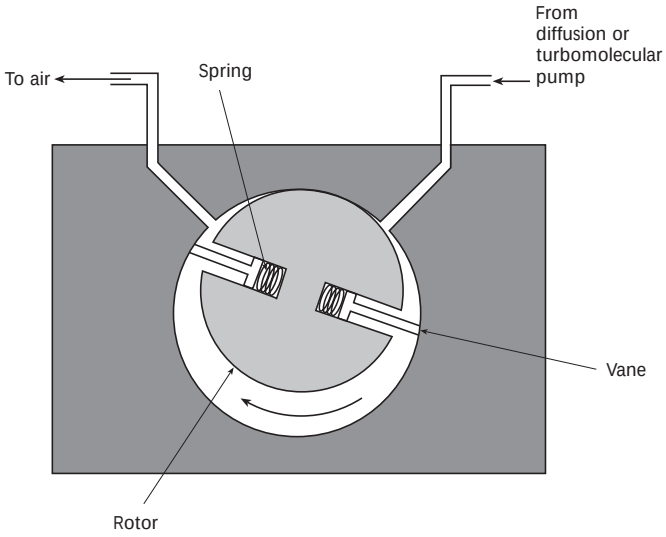
Adjustable Sensitivity

The compression ratio of helium can be varied by changing the pumping action of the diffusion pump. A control is provided to allow the variation of this compression ratio and thus increase or decrease the sensitivity of the leak detector as required by the application.

Operation of Mechanical Forepump of Mass Spectrometer

The mechanical forepump of the mass spectrometer (see Fig. 15) operates by means of an eccentrically mounted, oil sealed rotor, turned by means of an electric motor. As the rotor turns, it compresses gases from the test object and mass spectrometer connections into a smaller volume. This increases the gas pressure until it intermittently forces open

FIGURE 15. Schematic diagram of mechanical forepump used in mass spectrometer vacuum system.



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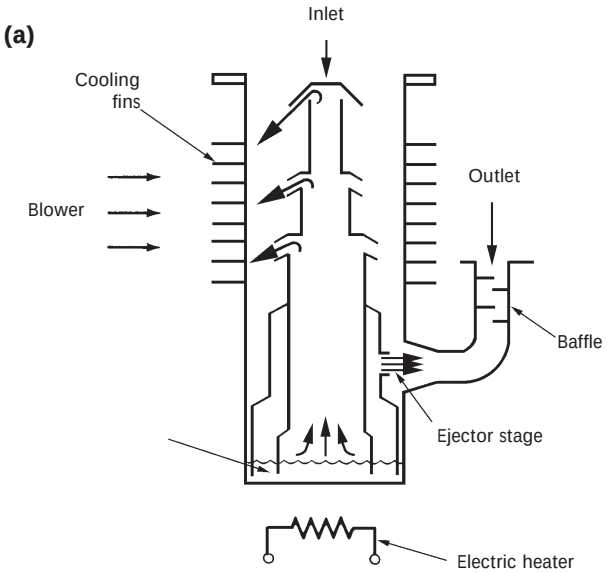
an oil seal valve and escapes to the atmosphere. Rotary mechanical vacuum pumps are capable of reducing the system pressure to less than 1 Pa (10 mtorr).

The rotary pump has two spring activated vanes in the annulus between the eccentric rotor and the stator of the pump. these vanes serve as seals to prevent back streaming of air or gases from higher pressure into lower pressure portions of the annular cavity. Oil within the annulus helps to seal the vanes to the cylindrical interior wall of the stator, to aid in preventing back leakage within the pump stages. The typical forepump is capable of reducing system pressure to about 3 Pa (20 mtorr). However, this low pressure may not be attainable in systems with large leaks. In this case, large leaks must be sealed before progressing to higher vacuum levels to search for small leaks with the mass spectrometer.

FIGURE 16. Diffusion pump used in mass spectrometer vacuum systems: (a) schematic; (b) exterior of oil diffusion pump.



Operation of Diffusion Pump of Mass Spectrometer

Because the vacuum produced by the forepump is not sufficient for proper operation of the mass spectrometer, an oil diffusion or turbomolecular pump is used in series with the forepump. In an oil diffusion pump, high speed jets of oil vapor impart to the gas molecules a momentum that acts to drive these molecules toward the outlet part of the pump (see Fig. 16). These vapor jets are produced by heating a pool of oil in the base of the pump. The vapors rise in a central column and are forced outward and downward into the annulus by jet guides. As these vapors flow downward against the walls of the pump, they are condensed and return to the oil pool for reheating and evaporation. The diffusion pump is usually air cooled by means of an electric blower. An oil diffusion pump cannot exhaust its pumped gases against atmospheric pressure. The forepump is therefore connected to the exhaust port of the diffusion pump. The mechanical forepump lowers pressures to less than 10 Pa (0.1 torr), into which the diffusion pump can exhaust its gases.

Oil Vapor Jets in Multistage Fractionating Pump Stack

Heating the oil in the reservoir at the base of the diffusion pump (see Fig. 16) produces an upward flow of oil vapor through the center of the diffusion pump. The jets deflect the oil vapor outward and downward to the outside wall, which is air cooled. Striking the cool wall, the oil vapor condenses and then flows back into the reservoir, where it is reevaporated. Pumping action is achieved when the molecules of gaseous constituents within the vacuum system are bombarded by the outward and downward stream of oil vapor. These gas molecules are forced lower and lower by each of the three succeeding jets. The fourth stage is an ejector jet that compresses the molecules into the foreline to be removed by the mechanical forepump. This fourth stage jet also cuts down drastically the back migration of the mechanical pump oil. An ejector stage also increases the compression factor of the pump to a considerable degree. This decreases the number of helium molecules that get back through the upper jets of the diffusion pump.

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The diffusion pump shown in Fig. 16b uses a four-stage fractionating pump stack. The fractionating section in the boiler compartment, combined with the ejector stage, ensures continuous purification of pump oil. This self-purifying action results in satisfactory performance despite conditions of poor diffusion pump oil and/or vacuum system contamination. Because the diffusion pump oil will decompose and oxidize if exposed to the atmosphere at operating temperature, an oil diffusion pump can neither pump on nor exhaust to atmospheric pressure. Therefore, the mechanical forepump, in series with the diffusion pump, acts to create, at the base of the pump, a vacuum that causes gas molecules pumped by the diffusion pump to exhaust to atmosphere. In some versions of mass spectrometer leak detectors, a second mechanical pump known as the roughing pump (Figs. 2 and 14) evacuates the chamber to the point at which its volume can be exposed to the diffusion section without raising the pressure in the diffusion section of the systems to critical levels.

Turbomolecular Pump

The system design of a turbomolecular pump (Fig. 17) is similar to that of a diffusion pump system, using a common roughing and foreline pump. It is possible, however, to rough pump a chamber right through the turbo; in this case, the turbomolecular pump will gain speed as system pressure is reduced.

Turbomolecular pumps are very clean mechanical compression pumps. They pump by using a high speed rotating surface to give momentum and direction to gas molecules. They operate smoothly and contribute little vibration to the operating system. They are the only mechanical vacuum pump that can reach pressures of less than 0.7 µPa (5 ntorr) without using traps. (Metal gaskets and mild bakeout of the vacuum system are necessary to reach this pressure.)

When operated correctly, turbomolecular pumps are highly reliable and clean. Because they can operate from steady state inlet pressures as high as 1 Pa (8 mtorr) to below 70 nPa (0.5 ntorr), turbomolecular pumps are used in wide variety of applications. They are ideal for applications requiring a vacuum relatively free of hydrocarbons. Turbomolecular pumps offer the advantages of a fast startup to full pumping speed and a clean, oil free vacuum.

Like the diffusion pump, the turbomolecular pump cannot exhaust directly to atmosphere. Usually a rotary mechanical pump or dry vacuum pump is used as a forepump for the turbo.

Pump Components

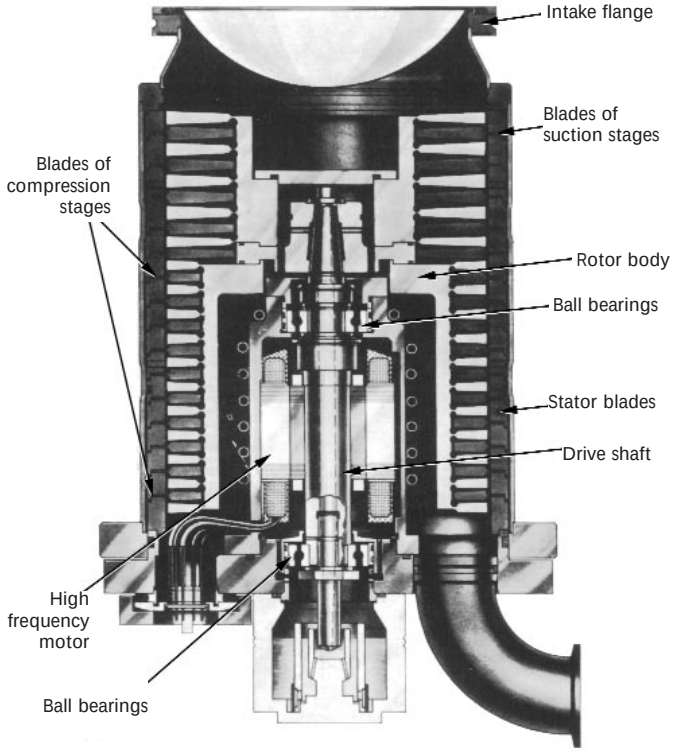
The turbomolecular pump is composed mainly of rotating and fixed disks, called *rotors* and *stators,* respectively. The rotor disks are arranged alternately with the stator disks. On each disk are blades. The number of blades on a disk, the blade length, width, spacing, and rotational speed determine its ability to pump gases.

Each rotor and stator disk can be called a compression stage. A pump may have as many as 10 to 40 stages. The rotor is driven by a motor capable of reaching speeds from 900 to 9000 rad·s^{-1} (9000 to 90 000 revolutions per minute), depending on pump size. The motor is typically powered through a special power supply. Compressed gases are expelled from the pump through a foreline that must be evacuated by some type of forepump.

Pump Operation

On the stages closest to the inlet, the blades have a large angle so as to pump at a faster rate, because more open space

FIGURE 17. Turbomolecular pump of single ended axial flow design.



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allows more access to the chamber. The blades closest to the foreline have a small angle for greatest compression. This works to move the gases from the inlet into the foreline. It also works to keep the gas and oil molecules in the foreline for making their way to the inlet.

Turbo pumps typically operate at speeds from 900 to 9000 rad·s^{-1} (9000 to 90 000 revolutions per minute), For any given turbomolecular pump, variations in the rotational speed will strongly affect the pumping performance. The pumping speeds and compression ratios achieved with a turbomolecular pump are related to rotational speed.

Procedure for Starting and Stopping Leak Detector Vacuum Pumping

The exposure of hot organic pump fluids to atmospheric pressure would result in decomposition of the pump fluid. Therefore, the system must first be exhausted to a pressure less than 10 Pa (0.1 torr) before the diffusion pump is turned on. Similarly, it is important to shut off the diffusion pump first and then to wait to allow the pump oil to cool before turning off the mechanical forepump or venting the diffusion pump to atmosphere. The turbomolecular pump must also be forepumped before starting, but without concern for decomposing pump fluids.

The blower (which cools the walls of the diffusion pump), forepump and diffusion pump should be interlocked for maximum protection of the diffusion pump oil. A thermal, self-restoring circuit breaker should provide protection and intermittent operation to maintain vacuum sometimes. The blower should be fused with the diffusion pump so that blower failure will disrupt heating power to the diffusion pump. The diffusion pump cannot be turned on unless the forepump has been started.

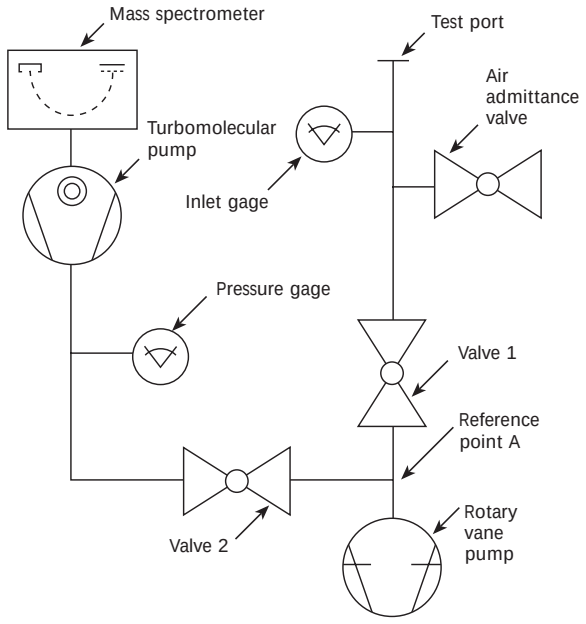
In typical installations, when the forepump is first turned on, a gurgling sound will be heard, caused by the high pressure air being exhausted. When the forepump stops gurgling, the diffusion pump can be turned on. The forepump and diffusion pump switches are interlocked so that the diffusion pump cannot operate unless the forepump is on. Only after the diffusion pump is in operation should the electronic circuits of the mass spectrometer be turned on. The electronics on/off switch also turns on the discharge gage, if used. Therefore, the electronics power should not be turned on too early because unnecessary contamination of the gage will result. If the pressure reading of the discharge gage is off scale, the electronics should be shut off for an additional few minutes of waiting period. After this waiting time, the pressure meter reading should be on scale. When the pressure reading is 10 mPa (0.1 mtorr) or lower, liquid nitrogen can be added to the cold trap in the direct flow leak detector.

Counterflow Configuration with Turbomolecular Pump

Counterflow architecture overcomes the drawbacks of the direct flow system by using one of the characteristics of the common diffusion pumps. Both diffusion and turbomolecular pumps exhibit different maximum compression ratios for gases of different molecular weights. The maximum compression ratio of any compressor is defined by its ability to prevent gases from returning from the exhaust to the inlet. The designs of these two diffusion pumps permit maximum compression ratios of the order of 1 000 000:1 for heavy gases, but only about 100:1 for helium.

The result of this is that the pump can provide excellent vacuum for the mass spectrometer, protecting it from heavy gases, while being relatively transparent to helium presented at the foreline. Figure 18 shows the design of a leak detector designed to use this principle. This design uses a single rotary vane pump to provide the preliminary

FIGURE 18. Compact counterflow architecture.



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evacuation, as well as to back the diffusion pump, by appropriately opening valves 1 and 2.

When a part to be tested is connected to the test port, valve 2 is closed and valve 1 is opened. When the pressure at the inlet is at 10 Pa (0.1 torr), valve 2 is opened again. Now a portion (one percent) of any helium partial pressure at point A will be evident at the mass spectrometer, as it diffuses back through the turbomolecular pump.

The equation $Q = PS$ can now be applied at point A to determine the effects of changing pump speeds. The partial pressure of helium will be reduced by adding a vane pump with higher pumping speed. In this configuration the turbomolecular pump can provide very high pumping speed at the mass spectrometer but its compression ratio for helium must be low and stable. The mechanics and electronics of turbomolecular pumps permit this to be accomplished.

Counterflow architecture permits parts and systems to be tested at 20 Pa (0.2 torr) — a factor of 2000 higher pressure than the typical direct flow leak detector. This pressure is substantially easier to obtain in most applications. In addition, the turbomolecular pump protects the mass spectrometer from water vapor, solvents, oils etc. that come from typical test parts, resulting in significantly lower maintenance efforts.

The weaknesses in the counterflow architecture include low pumping speed at the test port and the fact that throughout the process the part under test is exposed to the rotary vane pump. Because the oil vapors from this pump can migrate under the molecular flow conditions obtainable at these pressures, it is possible that some oil vapors can backstream into the test port. The amount of this backstreaming is miniscule, but for some highly critical applications this is not tolerable.

Enhanced Counterflow

A special purpose turbomolecular pump has been designed to provide a combination of high pumping speed and cleanliness in a counterflow leak detector. Figure 19 shows the architecture in a simplified schematic.

When this leak detector is used, the roughing pump evacuates the test part to 10 Pa (0.1 torr); then valve 1 is closed and valve 2 opened. During the remainder of the test, the test piece is continuously being evacuated, the speed boosting stages of the turbomolecular pump (in fact, the high vacuum and speed boosting stages are on a single shaft, in a single housing, turned by the same motor.) The high vacuum stages of this pump maintain the appropriate vacuum in the mass spectrometer and permit back diffusion of the helium.

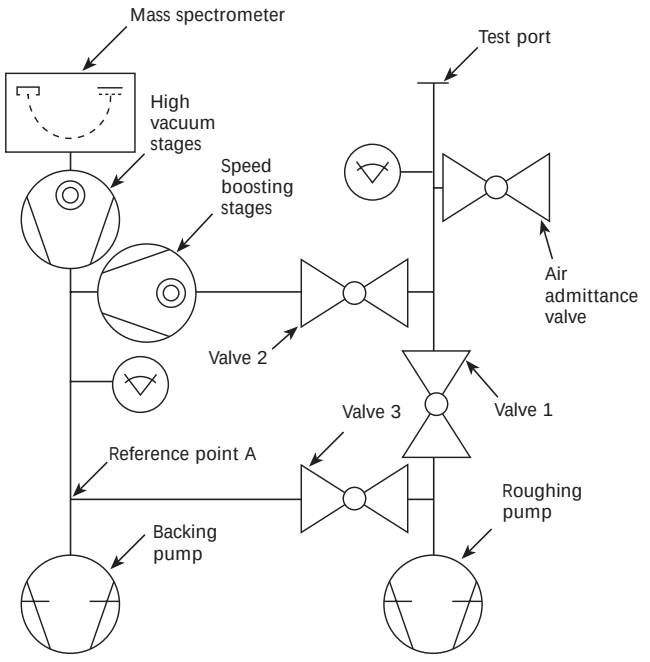
When the pressure at the inlet is 10 Pa (0.1 mbar), the gas flow is high enough that the backing pump alone does not keep the pressure at point A low enough. Valve 3 is opened at this time, to provide additional pumping speed. For highest sensitivity when the gas flow is sufficiently reduced, valve 3 is closed.

The addition of the speed boosting stages precludes backstreaming of vane pump fluid to the test piece and provides a high pumping speed at the test port.

Operation of Cold Trap in Mass Spectrometer Leak Detector

The function of the cold trap in a vacuum system is to freeze out the residual condensable vapors to avoid contamination of the spectrometer tube or discharge gages. The cold trap also serves as the third type of pump used in the spectrometer's vacuum system. The freezing action is accomplished by providing highly refrigerated surfaces on which the vapors are trapped. The most commonly encountered vapors are oil vapor (from the diffusion and mechanical pumps) and water vapor. When refrigerated with liquid nitrogen, the cold trap acts as a highly efficient pump for condensables such as water vapor. Its use therefore reduces pumpdown times by a factor of as much as ten or more.

FIGURE 19. Counterflow architecture enhanced for speed and cleanliness.



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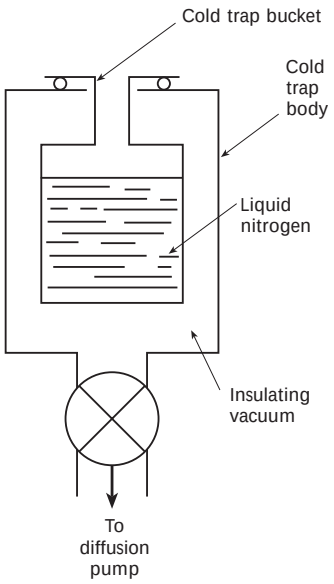
Construction of Mass Spectrometer Cold Trap

The construction of the cold trap is similar to that of a thermos bottle. It consists of an inner and an outer shell with the intervening space insulated by vacuum (Fig. 20a). In operation, the inner low heat loss stainless steel refrigerant bucket is filled with liquid nitrogen. The filling port is in the top work surface of the cold trap illustrated in Fig. 20b. The trap can be completely filled up with 2 L (120 in.^{3}) of liquid nitrogen. To minimize transfer losses, a dewar designed for filling the trap is recommended. Liquid nitrogen should be handled with care and all safety precautions should be observed.

FIGURE 20. Liquid nitrogen cold trap for condensation of vapors that would otherwise contaminate vacuum systems of mass spectrometer leak detectors: (a) schematic diagram; (b) cold trap removed from mass spectrometer.

(a)



(b)

Selection of Refrigerant for Cold Trap

The lower the temperature of the coolant, the more effective is the cold trap in pumping and condensing vapors in the vacuum system. The recommended coolant for most commercial mass spectrometer leak detectors is liquid nitrogen. Liquid air is almost equally effective but more dangerous (because the liquid oxygen in the liquid air may constitute a fire hazard).

Operation of Cold Cathode Discharge Gage for Pressure

The cold cathode discharge gage is one of several types of pressure gage used in leak detector vacuum systems. Vacuum gages are used in mass spectrometer leak detectors to provide indications of pressure and to permit automatic control. These gages supply output electrical signals corresponding to vacuum system pressures. The cold cathode discharge gage (see Fig. 21a) consists of two electrodes mounted inside the vacuum. External to the gage is a permanent magnet that establishes a magnetic field through the loop of the anode (Fig. 21b). High voltage applied across the gage electrodes produces a gaseous electrical discharge. The current passing through the electrical discharge is proportional to the pressure in the discharge gage. This signal current can be read out on a panel instrument calibrated in pressure units.

The useful pressure range of the cold cathode discharge pressure gage is between 100 and 0.01 mPa (0.1 mtorr and 0.1 μtorr). The discharge gage is used to monitor the pressure in the high vacuum section of the mass spectrometer leak detector and to trigger the filament protection circuit.

At vacuum system pressures above 13 kPa (100 torr), the discharge gage behaves as if the system pressure were much lower. It might act to reenergize the filament of the mass spectrometer tube at dangerously high operating pressures. To avoid filament operation at high pressures, a spark gap is placed in parallel with the discharge gage. The gap sparks over at a pressure reading of 13 kPa (100 torr). This provides a protection over the entire pressure range from 40 mPa to

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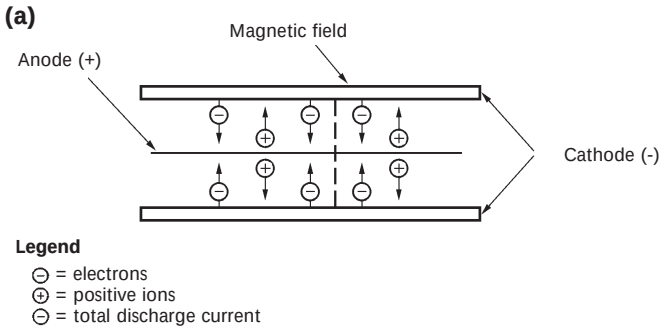
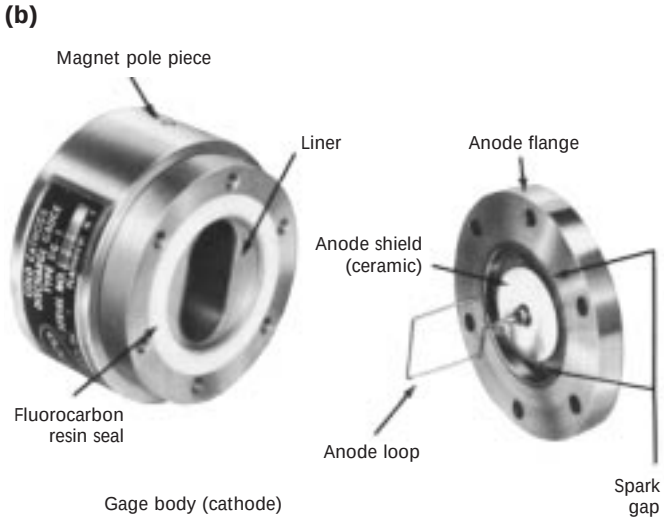
100 kPa (0.3 mtorr to 750 torr). Notwithstanding these protective devices, care must be exercised to avoid exposing the mass spectrometer tube filament to repeated bursts of high pressure. Otherwise, the filament life may be shortened. With reasonably careful operation, the filament will provide several thousand hours of leak testing service.

Operation of Pirani Gage for Measuring Pressure in Vacuum System

The Pirani gage consists of a resistive element *R*, mounted inside the vacuum system (Fig. 22). The number of gas molecules in the gage is directly proportional to the gas pressure. These molecules act as a heat sink for the resistive element of the Pirani gage, which is heated by ohmic losses when it received electrical current from a constant current generator. The varying voltage across the resistive element of the Pirani gage is amplified and provides an indication of vacuum chamber pressure on a panel meter. The Pirani gage is typically located in the test manifold. During an automatic roughing cycle, the Pirani gage determines if pressure has been sufficiently reduced to allow exposure of the test manifold to the high vacuum section.

FIGURE 21. Cold cathode gage used to measure vacuum system pressure: (a) schematic diagram showing operating principle and assembly; (b) basic components.



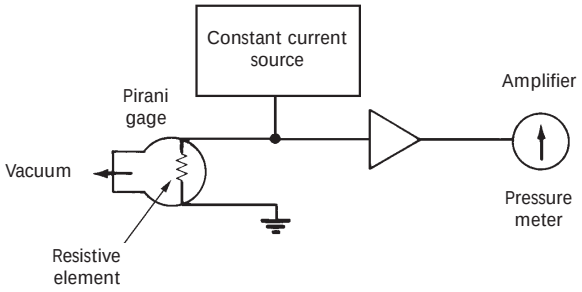


Functions of Control Valves in Mass Spectrometer Leak Detector

High vacuum valves are used in the mass spectrometer leak detectors where, under certain circumstances, it is necessary to isolate, either partially or completely, sections of the leak detector from one another. This function is performed by valve types designed especially for high vacuum service. Figures 14 show typical locations for valves known by their functions as accumulator, diffusion pump isolation, throttle, pump and vent valves.

The diffusion pump isolation valve serves a dual purpose. First, it provides a means of isolating the diffusion pump from the vacuum chamber, cold trap and mass spectrometer tube. It is extremely convenient in servicing and maintenance. For example, by closing the valve and venting the vacuum system, the cold trap and mass spectrometer analyzer can be removed for cleaning, the filament can be changed and other maintenance functions can be performed without shutdown of the diffusion pump. This is less relevant when a turbomolecular pump is used, for it can be stopped and restarted quickly. Thus, the leak detector can be restored to operational status within minutes after reassembly. Under

FIGURE 22. Pirani gage used to measure vacuum system pressures in manifold section of mass spectrometer leak detector.



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ordinary leak test conditions, the diffusion pump isolation valve is left completely open.

The same (diffusion pump isolation) valve is also known as the accumulator valve because the user may employ accumulation leak testing techniques by partially closing the accumulator valve. This decreases pumping speed and makes possible the detection of smaller leaks.

The throttle valve is the most important valve in the direct flow leak detector. It controls the rate of gas flowing into the leak detector from the system being leak tested. In testing for relatively large leaks, it is often necessary to throttle from a test system pressure of several tens of pascal (hundreds of mtorr) down to a leak detector operating pressure of some 30 mPa (0.2 mtorr). The throttle valve is a manual override of the test valve. When leak testing in the automatic mode, the throttle valve is always left in the full open position. This valve is normally used only when leak testing in the manual mode.

Vacuum System Cleanliness and Sources of Spurious Background Signals

During mass spectrometer leak testing, spurious background signals can arise from such sources as (1) helium contamination, (2) scattering of ions due to excessively high pressure in the mass spectrometer tube and (3) hydrogen or hydrocarbon contamination.

Furthermore, elastomeric gaskets, greases, rubber hose, painted surfaces and castings, exposed even once to high concentrations of helium, tend to adsorb and absorb helium and become sources of background helium signals. These spurious helium signals tend to reduce the ability of the helium leak detection instrument to respond to minute true leaks.

Indications of Contaminated Vacuum System in Mass Spectrometer

With the typical helium mass spectrometer, a contaminated vacuum system may be evidenced either by an offscale reading of the vacuum gage or by excessive helium or other background signals. Normally, after about 25 min of vacuum system pumpdown time, the pressure meter should indicate less than 100 mPa (1 mtorr) pressure. If this pressure meter indicates above 100 mPa (1 mtorr), it may be due to a contaminated vacuum system.

High readings may, however, be due to causes other than contamination. Among the possible causes of high readings of vacuum are (1) high pressure due to leaks from the atmosphere into the mass spectrometer system, (2) outgassing of external equipment or systems under test, which are attached to the mass spectrometer vacuum system, (3) high vapor pressure products from pump oils (such as water), (4) contaminants from decomposed diffusion pump oils, (5) diffusion pump not turned on, (6) heater for diffusion pump not operating, (7) leakage past O-ring valve seals due to lint and similar foreign particles, (8) electrical short circuit in the discharge gage cable or circuit and (9) liquid nitrogen trap recently run dry, permitting water to evaporate.

Sources of Contamination of Vacuum System of Mass Spectrometer

Contamination of the vacuum system of mass spectrometer helium leak detectors can occur due to the repeated connection of unclean test objects or test systems to the test port. It can also result from the backstreaming and subsequent deposit of decomposition products from the diffusion pump oil on the interior walls of the vacuum system. Diffusion pump oil will be decomposed if the oil is exposed to air while at operating temperature. If the pump oil has been decomposed or cracked by air exposure, an acrid smell at the exhaust of the mechanical pump may be observed.

Excessive background indications can be caused by testing objects that are contaminated with lubricating oils or other materials or by operation of the mass spectrometer with a contaminated auxiliary pumping system. Quite often the removal of background (degassing) can be effected by warming the contaminated areas with a heat gun while the vacuum system is pumping. This is the simplest decontamination technique for vacuum systems and should be tried before disassembly of vacuum plumbing.

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Procedures for Cleaning of Contaminated Vacuum System of Mass Spectrometer

When it has been established that contamination is the cause of the high pressure reading within the mass spectrometer, the entire vacuum system should be cleaned and a new charge of pump oil should be added to both the diffusion pump (when used) and the mechanical pump or pumps. Similarly, if background exists and tends to obscure the true helium peak, the sensitivity of the mass spectrometer leak detector is impaired. This situation should be corrected immediately by cleaning the contaminated sections of the leak detector systems. The following procedure can be used to locate the contaminated sections of the mass spectrometer leak detector.

1. Isolate the mass spectrometer from objects to be tested and also isolate it from auxiliary pumps or setups. In most cases, isolation is achieved by plugging the ports and then placing the mass spectrometer in its test condition.
2. If background remains after the mass spectrometer is isolated by the procedure of step 1, close the throttle valve of the mass spectrometer. If the background then disappears, the sources of contamination should be suspected to be in the test valve, in the vent valve or in the oil of the roughing pump.
3. If the background still remains after step 2, the next procedure is the following. (a) Turn off the electronic circuits of the spectrometer. (b) Turn off the diffusion pump. (c) Remove the source cable. (d) Allow 10 to 15 min for the diffusion pump to cool. (e) Vent the vacuum system through the throttle valve. (f) Drain and flush the forepump with clean oil (specified by pump manufacturer). (g) Fill the forepump to proper level with clean new pump oil. The vacuum system can then be started up in accordance with the normal operating procedure. Replace the source cable after the mass spectrometer tube has been evacuated to recommended pressure levels.
4. Finally, if the preceding steps prove to be ineffectual, the vacuum system should again be shut down. The diffusion pump, spectrometer tube and cold trap should then be cleaned by cleaning procedures recommended by the leak detector manufacturers.

Rather than prescribing specific cleaning techniques, it is recommended that the manufacturers instructions be followed. Because a variety of oils and vacuum lubricants are used, no single cleaning procedure can be effective in all cases.

Precautions in Handling and Cleaning of O-Ring Seals

Synthetic rubber O-rings are susceptible to absorbing large quantities of the solvents used in cleaning. The subsequent evaporation of these solvents must be avoided within the vacuum system. Therefore, O-rings and gaskets should be removed from the flange grooves and treated as a cleaning problem separate from that of cleaning glass and metal parts. It is recommended that replacement of O-rings and gaskets be done during the reassembly of cleaned vacuum system components. Alternatively, the O-rings and rubber gaskets can be carefully wiped clean with a lint-free material and inspected for surface damage before they are used again. If new O-rings are used, they should be wiped, inspected and lubricated in the same manner as used O-rings. When reassembling, clean the O-rings and regrease with a minimum of high vacuum grease. When reassembling vacuum actuator or valve assemblies, clean and regrease liberally both the piston O-ring and the walls of the cylinder, or cover existing grease.

O-rings should never be removed with a metal tool, as this would inevitably scratch the O-ring groove. This could cause a potential, if not an actual, leak. Instead, it is recommended that wood or plastic be used. The O-ring can be removed by inserting a plastic or wooden tool (such as a toothpick or piece of plastic) between the outside of the O-ring and groove and sliding this tool around the O-ring. This causes the O-ring to pop up. It may be necessary to hold the O-ring down on the side opposite the tool to prevent the O-ring from turning in the groove. This technique gives better extraction than trying to pry the O-ring from the groove.

Final Degassing of Components by Heating before Reassembly of Spectrometer

Each unit of the high vacuum assembly of the mass spectrometer leak detector vacuum system, with the exception of the mechanical pump, may join its adjacent unit either by flanges or by quick couplings. The flanges are sealed by

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synthetic rubber O-rings or fluorocarbon resin gaskets. The vacuum system can be easily disassembled, allowing inspection and cleaning of all components parts of the vacuum system. When the system is to be disassembled for cleaning, remove the cold trap bucket, decant the liquid nitrogen and dry the bucket thoroughly before replacing. Heating of vacuum system components before reassembly should be considered as a necessary part of final cleaning. This degassing of internal surfaces by heating is easily accomplished by a hot air stream from a heat gun. If the entire vacuum system is being cleaned, new synthetic rubber O-rings should be used during its reassembly.

Disassembly and Cleaning of High Vacuum Pumps

Leak detector diffusion pumps are usually designed to be readily disassembled for cleaning, in accordance with manufacturers' instructions. Do not try to clean turbomolecular pumps without contacting the manufacturer first.

After cleaning, the diffusion pump can be reassembled and refilled with the oil specified by the manufacturer. Do not use any substitute oil or degradation of the spectrometer performance can result. Note that fresh diffusion pump fluid gives off large quantities of gas when it is first exposed to low (vacuum) pressures. This gas evolution causes the vacuum system pressure to rise. Pressure rises of this type should not be mistaken for an indication of leakage.

Cleaning and Refilling of Mechanical Pump of Mass Spectrometer

Mechanical pumps used in mass spectrometer leak detectors often become contaminated with large amounts of water and/or decomposition products from the oil diffusion pump. To change the oil in the mechanical pump, disconnect the mechanical pump from the vacuum system. Warm the oil by operating the mechanical pump with the intake closed, for about 15 min. Then stop the pump and remove the oil drain cap.

Use safety precautions. Operators are warned that the pump oil will be hot! Most of the oil will drain out freely. Be careful not to use solvents or light flushing oils in the mechanical pump. Their removal is difficult and their high vapor pressures will prevent the attainment of high vacuum.

After the mechanical pump has been flushed completely clean, refill it by pouring new pump oil into the exhaust port. Fill to proper level indicated in sight glass. Replace dust cap. A gurgling noise is characteristic when high pressure air is drawn through the mechanical pump. This gurgling noise should disappear quickly as the intake pressure is reduced when pumping out the vacuum system. If the mechanical pump continues to gurgle, its oil level may be too low. Insufficient oil in the mechanical pump does not give proper sealing or lubrication. If this occurs, oil should be added through the exhaust port until it reaches the proper level.

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10

CHAPTER

Leak Testing with Halogen Tracer Gases

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PART 1. Introduction to Halogen Tracer Gases and Leak Detectors

Halogen Vapor Tracer Gases and Detectors

Leak testing with halogen vapor tracer gases uses leak detectors that respond to most gaseous compounds containing halogens such as chlorine, fluorine, bromine or iodine. (The elemental halogen gases are not commonly used as tracers. They are toxic and typical halogen vapor detectors do not respond sensitively to these elemental gases.) Preferred halogen tracer gases are nontoxic chemical compounds such as the common refrigerant gases and other leak testing tracers listed in Table 1. Concentration limits prescribed by the United States Occupational Safety and Health Administration should be observed.[1] For example, refrigerant-134a is tetrafluoroethane. In addition to being a refrigerant, this gas is an excellent halogen tracer gas because it is inert, nontoxic, liquid at moderate pressures and readily available in convenient small and large containers.

For example, if a closed component, pipe, vessel or system is pressurized with one of the halogen tracer gases or with a mixture of a halogen gas with air or nitrogen, a halogen vapor leak detector can be used to locate leaks and/or to measure the rate of leakage. Three types of halogen leak sensors or detectors used in halogen leak testing are (1) the halide torch, (2) the heated anode halogen detector and (3) the electron capture (electronegative gas) detector. The uses of each of these halogen tracer gases and detectors are described in detail later in this section, following an introduction to methods.

Selection and Handling of Halogen Tracer Gases for Leak Testing

The most popular halogen tracer gases for leak testing are the gases refrigerant-22 (monochlorodifluoromethane, $CHClF_2$) and refrigerant-134a (1,1,1,2-tetrafluoroethane, $C_2H_2F_4$). These compounds are available from local refrigeration suppliers in containers varying in size from small cans to full size pressurized liquid cylinders. Also available from such refrigeration suppliers are refrigerant fittings and hose. Table 2 lists the properties of refrigerant-22 and refrigerant-134a gases.

Refrigerant-134a and refrigerant-22 gases are stored as liquids under pressure at room temperature. They exert vapor pressures above the liquid refrigerant of 488 kPa (70 $lb_f \cdot in.^{-2}$ gage) for

TABLE 1. Halogen compound tracer gases used in halogen vapor leak testing. Limits of atmospheric concentrations (see Key[1]) must be observed for health and safety reasons.

Generic Name	Chemical Formula	Trade Designation	Concentration Limit ($\mu L \cdot L^{-1}$)
Fluorotrichloromethane	CCl_3F	R-11	1000
Dichlorodifuoromethane	CCl_2F_2	R-12	1000
Chlorotrifluoremethane	$CClF_3$	R-13	No standard
Trifluoromonobromomethane	$CBrF_3$	R-13B1	1000
Dichloromonofluoromethane	$CHCl_2F$	R-21	1000
Monochlorodifluoromethane	$CHClF_2$	R-22	No standard
Trichlorotrifluoroethane	$C_2Cl_3F_3$	R-113	1000
Dichlorotetrafluoroethane	$C_2Cl_2F_4$	R-114	1000
1,1,1,2-tetrafluoroethane	$C_2H_2F_4$	R-134a	1000
Sulfurhexafluoride	SF_6	Electronegative gas tracer	1000
Carbon tetrachloride	CCl_4		10
Methyl chloride	CH_3Cl		100
Perchloroethylene (or tetrachloroethylene)	C_2Cl_4		10
Trichloroethylene	C_2HCl_3		100
Vinyl chloride	C_2H_3Cl		1

TABLE 2. The properties of refrigerant-22 and refrigerant-134a gases.

Refrigerant Gas	Properties R-22	R-134a
Chemical formula	$CH\text{-}ClF_2$	$CF_3\text{-}CH_2F$
Molecular weight	86.4	102.03
Leakage rate relative to air	1.5	1.5
Boiling point at 100 kPa (°C)	–40.8	–26.1
Boiling point at 1 atm (°F)	–41.4	–14.9
Liquid density at boiling point ($kg_m \cdot m^{-3}$)	1413	1374
Liquid density at boiling point ($lb_m \cdot ft^{-3}$)	88.2	85.8
Liquid density ($kg_m \cdot m^{-3}$) at 21 °C (70 °F)	1209	1221
Liquid density ($lb_m \cdot ft^{-3}$) at 21 °C (70 °F)	75.5	71.4
Vapor pressure above refrigerant[a] (kPa gage)	842	488

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refrigerant-134a and 840 kPa (122 $lb_f \cdot in.^{-2}$ gage) for refrigerant-22 at 21 °C (70 °F). If the pressure in the storage cylinder is reduced through a valve and the refrigerant is introduced into a test system or chamber, some or all of the liquid refrigerant will vaporize and diffuse to fill the chamber. Liquid refrigerant will continue to vaporize until the pressure in the closed system or chamber is equal to the vapor pressure above the liquid or until no more liquid is left. The maximum pressure of halogen vapor possibly attained from a storage bottle of liquid refrigerant-134a or refrigerant-22 can be determined from Fig. 1 for a wide range of system temperatures.

During vaporization, these refrigerant gases are cooled considerably. If the gases are being vented into a rather large chamber, the refrigerant storage cylinder may cool to the point where vaporization of the refrigerant is extremely slow. When this occurs, it may be advisable to accelerate evaporation of liquid refrigerant by placing the cylinder in a tank of warm water.

Dilution of Halogen Tracer Gases Used as Leak Test Tracers

It is sometimes desirable to dilute the halogen tracer gases during leak tests for the following reasons.

Liquefaction at High Pressure. Because refrigerant-134a gas liquefies at 488 kPa (70 $lb_f \cdot in.^{-2}$ gage) and refrigerant-22 gas liquefies at 840 kPa (122 $lb_f \cdot in.^{-2}$ gage) at 21 °C (70 °F), a pressurized system being leak tested at room temperature cannot have a 100 percent tracer gas pressure greater than these pressures. If leak testing is to be done at higher pressures, additional pressurized air must be added to the refrigerant pressure to obtain the desired test pressure in the system.

Dilution with air without increasing pressure would reduce the leak test sensitivity. However, use of a higher pressure increases the leakage rate in more than a compensatory fashion.

The increase in leakage sensitivity is proportional to the difference of the squares of the absolute pressures on the respective sides of the leak, whereas the decrease in sensitivity is only directly proportional to the decreases in halogen vapor concentration.

Quantitative Measurement. It may be desired to measure the leakage quantitatively. In that case, the halogen concentration reaching the detector must be relative low, less than 1 $\mu L \cdot L^{-1}$.

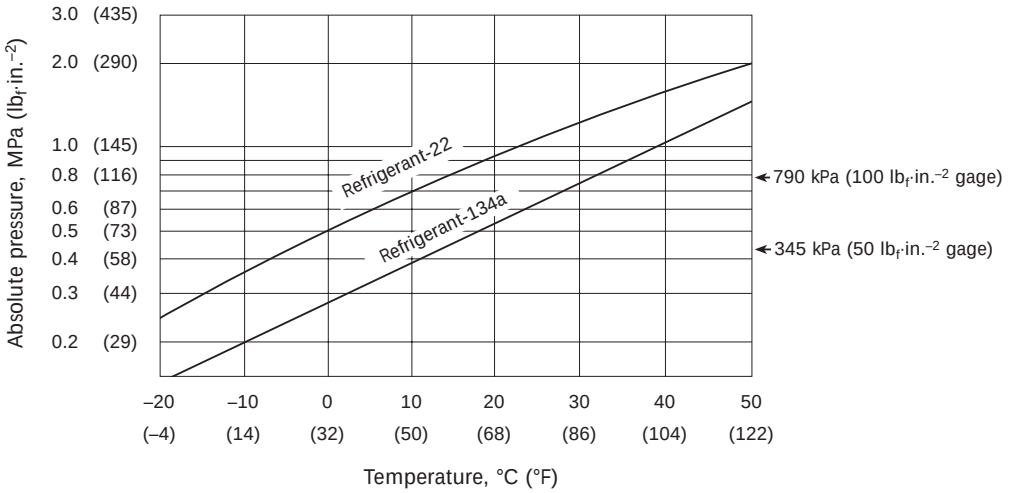
Cost for Large Volumes. In testing large systems, the cost of the tracer gas may be considerable. If only large leaks are of interest, the dilution of the tracer gas will reduce the overall cost and, as an added feature, decrease the amount of background contamination by leakage.

Figures 16 and 17 (below) relate refrigerant tracer gas concentrations to total gas pressures when pressuring up with diluted tracer gases.

Effect of Vapor Pressure of Refrigerant Tracer Gases

If refrigerant-22 gas is used, a slight penalty in leakage sensitivity is incurred. This could be offset by the difference in vapor pressures between refrigerant-134a and refrigerant-22. Pure refrigerant-134a cannot be introduced into a system above

FIGURE 1. The maximum pressure possible from a bottle of halogen tracer gas (refrigerant-22 or refrigerant-134a) at various ambient temperatures in SI units (pascal).



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its vapor pressure of 488 kPa, whereas refrigerant-22 can be added up to 840 kPa (122 $lb_f \cdot in.^{-2}$ gage). If the bulk of testing is to be done below 488 kPa (70 $lb_f \cdot in.^{-2}$ gage), refrigerant-134a is recommended. Above 488 kPa (70 $lb_f \cdot in.^{-2}$ gage), refrigerant-22 would function better because it can provide pressures up to 840 kPa (122 $lb_f \cdot in.^{-2}$ gage) at room temperature.

Effects of Low Diffusion Rates of Halogen Tracer Gases

The low diffusion coefficient of the heavier halogen tracer gases presents another problem in the efficient use of the halogen detector. To produce a dependable signal at a leak, the tracer gas mixture in air with which the system is charged must have uniform composition. Any blind passages in the system must be flushed with well mixed tracer gas, or leaks from blind passages will simply leak air and escape detection. Those leaks not flushed will remain at low halogen concentration for long periods — the halogen concentration is low for long periods because of the low diffusion rate of halogen tracer gas in air (above 8.5 $mol \cdot m^{-3} \cdot h \cdot$unit molar concentration gradient). The time required for a blind duct 1 m (3 ft) long to reach 40 percent of full halogen concentration is on the order of 3 h if diffusion alone is acting. Figure 2 shows the time required for various lengths of blind duct to reach 10 and 50 percent of open end halogen concentration. On the other hand, settling of heavier halogen from an initially well mixed tracer gas is not significant.

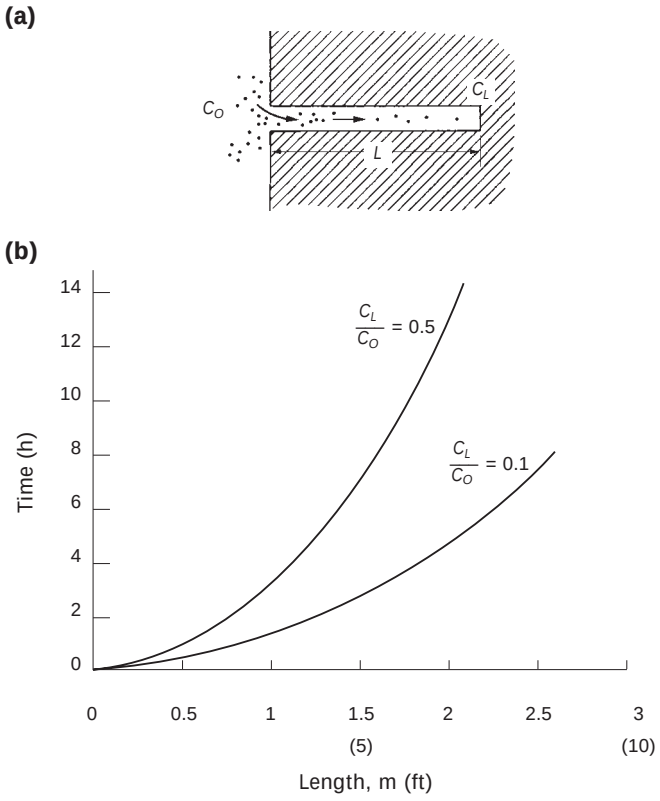
Effects of Halogen Vapor Accumulation on Surfaces

Experience has shown that many of the halogen compound vapors cling to surfaces for several minutes or longer and therefore may cause a sluggish recovery response in the detector. This effect should be kept in mind in connection with the construction of the detector probe and materials of the system being tested. This effect of halogen tracer hangup is similar to the effect of helium hangup.

Effects of Relatively High Density of Halogen Tracer Gases

Halogen tracer gases have about three times the density of air. If tracer gas emerges from a relatively large leak, it will flow into all nearby nooks and crannies and remain there for long periods of time. Its presence in confined spaces may give ghost leakage readings up to 24 h after the original leak has been repaired. The nature and persistence of these ghost signals are highly dependent on the geometry of the stagnant pocket and the ventilation around this space. Pure halogenated gas in an open beaker will be undetectable about 15 min after the beaker is filled. An open mouth Erlenmeyer flask, on the other hand, will still contain detectable amounts after being open to still air for 24 h or more. If this same flask were placed in a light breeze near an open window, the halogenated gas would vanish in a few minutes.

FIGURE 2. Diffusion of halogenated refrigerant-134 a gas in a blind duct: (a) cross section; (b) diffusion curves. Lower curve shows time required for C_L to reach 10 percent of C_O. Upper curve shows time required for inner end halogen concentration C_L to reach 50 percent of concentration at open end C_O.



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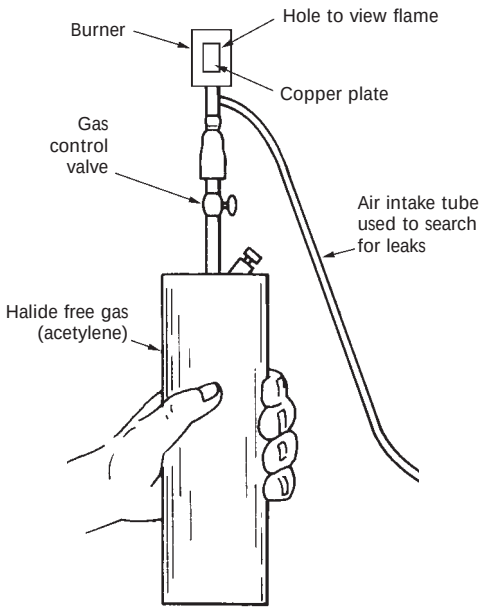
Halide Torch Detection of Halogen Tracer Gas Leaks[2]

The halide torch is used to locate leaks in systems filled with air containing halogen tracer gas. The color of its flame changes on introduction of halogen gas. The halide torch consists of a burner connected to a tank of halide free fuel such as acetylene gas or alcohol (Fig. 3). Some of the air for combustion is drawn into the flame (chimney fashion) through a tube near the bottom of the burner. A flexible extension of this air intake is a detector probe tube used as a probe to locate leaks. When the open end of this tube passes near a halogen tracer gas leak, some of the gas is drawn into the flame.

The halide flame detector is a small burner arranged to pull primary combustion air through a tube and into the burner, which heats a copper plate.

FIGURE 3. Halide torch for leak location.



The flame is a pale blue if only air is pulled into the burner through the suction hose. If small amounts of vapor containing halogen compounds enter the suction tube, the flame turns green, characteristic of copper. The halide torch procedure is used to locate leaks in pressurized systems. It is a desirable technique because of its low cost and portability.

The torch permits locating leaks as low as 250 or 300 g (8 or 10 oz) of refrigerant gas per year. The sensitivity to refrigerant gas is about 100 $\mu L \cdot L^{-1}$. This makes the general sensitivity about 10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-3} std $cm^3 \cdot s^{-1}$), with an air flow of 100 $Pa \cdot m^3 \cdot s^{-1}$ (2 std $ft^3 \cdot min^{-1}$).

The torch is available both as an individual unit and as an attachment to the portable gas cylinders. The flame color change detector, a more complex instrument using the same principle, is also available. Some properties of these instruments are listed in Table 3.

Advantages and Limitations of Halide Torch Leak Testing

In general, the halide torch leak test is about as sensitive and rapid as bubble emission leak testing. In addition, the torch technique permits location of leaks in places where bubble indications could not be seen. Moreover, no residue of test solution must be removed before the test apparatus can be used. Most of the refrigerant gases are nonflammable. Once a leak is detected, it may be soldered without fear of explosion. Other advantages of the halide torch are low cost, portability, simplicity and ease of operation.

The halide torch has no means of accurate calibration. A single large leak, may mask other adjacent smaller leaks, necessitating prior location (and correction) of such larger leaks by separate leak tests. The halide torch procedure uses halogen tracer gases and therefore has the same diffusion and stratification problems as described above.

TABLE 3. Comparison of halide torch detector and flame color change detector.

Characteristics	Halide Torch Detector	Flame Color Change Detector
Sensitivity	10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-3} std $cm^3 \cdot s^{-1}$)	5×10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (50 $\mu L \cdot L^{-1}$ or 5×10^{-5} std $cm^3 \cdot s^{-1}$)
Tracer gas	Halogen compounds	Compounds containing halogen
Output	Visual observation	Meter
Power requirement	Compressed fuel gas source	120 V, 60 Hz alternating current
Unit size	100 mm diameter × 400 mm (4 in. diameter × 16 in.)	400 × 400 × 250 mm (16 × 16 × 9.8 in.)
Unit weight	0.5 kg (1 lb)	16 kg (35 lb)

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Hazards of Toxic Gas Generation by Halide Torch Leak Testing

When the atmosphere is contaminated and a leak is located, toxic gas is generated from halides by the torch flame. Therefore, the torch should not be used in confined areas. The open flame may be a serious hazard in certain atmospheres and should never be used with flammable or explosive gas environments. The large, hot flame chamber can cause severe burns if contacted.

Techniques of Leak Testing with Halide Torch

In the detector probe technique, the halide torch is lighted and checked for proper operation by sucking in a trace of halogen gas from the supply tank. Then the air intake tube is used to search the surface of the system being tested at a scanning rate of about 10 $mm \cdot s^{-1}$ to locate leaks as small as 250 cm^3 (8 oz) of refrigerant gas per year. Because the tracer gas density is up to four times the density of air, it is advisable to start scanning on the upper side of a possible leak. A small trace of halogen gas will show up as a green flame, a large quantity as a violet flame.

This detector probe technique involves filling the system, or some part of the system that can be isolated, with halogen tracer gas. Then the surface of the system is scanned to detect traces of gas that issue from the leaks. Depending on the size of the vessel and the sensitivity desired, the air may or may not be evacuated before the tracer gas is introduced. Evacuation before pressurizing with halogen tracer gas takes longer and is not practical for very small pipes, but if accomplished, it makes possible a pure tracer gas atmosphere that can be pumped back into the storage tank after completion of leak testing.

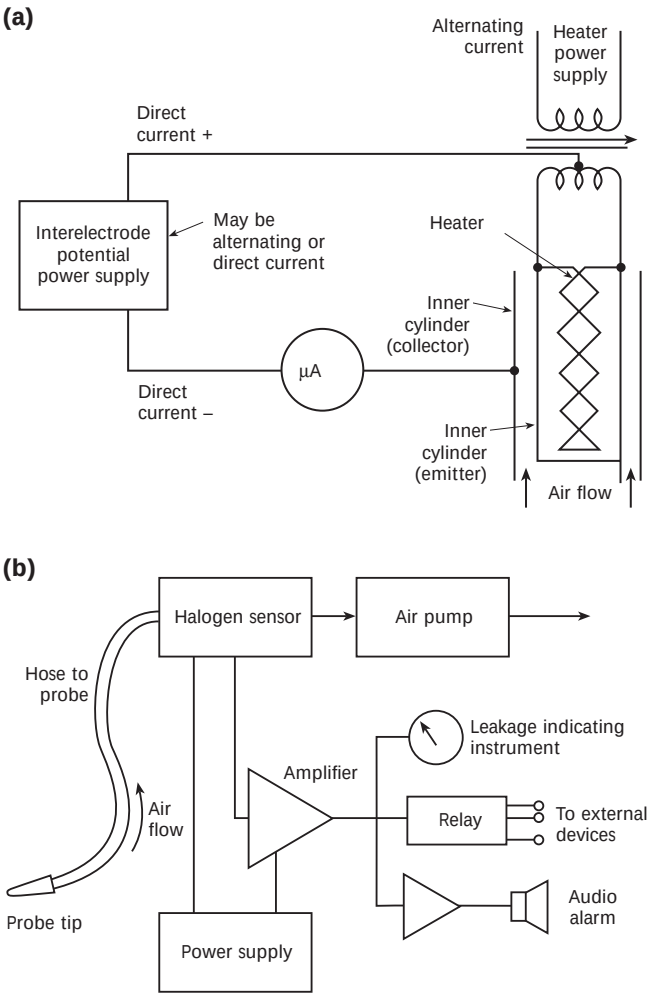
Principles of Operation of Heated Anode Halogen Detector

The heated anode halogen detector shown in Fig. 4a makes use of a red hot platinum and ceramic heater element that emits positive ions. These ions are collected on a negatively charged cylindrical cathode to provide a leak signal current. The presence of small traces of halogen vapors increases the emission of positive ions markedly. It is this increase in positive ion emission that is measured to indicate the presence of a leak.

Characteristics of Positive Ion Emission

In general, emission of ions means loss of material from the surface emitting them. One unique feature of ion emission, however, is that it can be made to occur readily in air at atmospheric pressure. Because platinum and some ceramic materials can be operated at red heat with little oxidation and loss from evaporation, such material is very useful as an ion emitting source. The rate of ion emission from such materials varies greatly, depending on temperature, area, nature of the surface and purity. The emission current drops slowly with operating time, eventually reaching a small but finite equilibrium value for any fixed temperature.

FIGURE 4. Halogen leak detector: (a) basic circuit of heated anode halogen leak detector, showing two-element heated anode sensing structure; (b) block diagram of halogen leak detector.



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The steady emission of ions in air is greatly increased when halide vapors strike the emitter surfaces. In the presence of even a small amount of a halogen compound vapor, there is a marked increase in ion current in the halogen leak detector. Common halogen tracer gases widely used in industry are refrigerant-134a and refrigerant-22 refrigerants. Table 1 lists other halogen compounds to which the heated anode alkali ion diode responds.[1] Relative responses to various halogen compounds are design dependent. For best results the detector should be calibrated with the tracer gas.

Functional Components of Heated Anode Leak Detector

The essential elements of a heated anode halogen leak detector are shown in Fig. 4. The basic instrument is provided with a portable detector probe and with a halogen vapor detector. The two-element sensing structure is in the form of concentric cylinders. The air contaminated with halogen vapor to be detected is passed between these two closed spaced cylinders (Fig. 4a). The inner cylinder is kept red hot by an internal wire heater. The outer cylinder is operated at a negative potential. The detector includes means of forcing air containing the tracer gas between the cylinders at a constant low velocity. The air pump provides a flow rate of about 1 $cm^3 \cdot s^{-1}$ (0.1 $ft^3 \cdot h^{-1}$) through the sensing element and then out to an exhaust port (Fig. 4b). An increased concentration of halogen gas passing through the sensor produces an increased electric signal current from the detector. Because of its heated element, the halogen leak detector should not be used in the presence of flammable atmospheres or explosive gas mixtures.

The electrical circuit of the heated anode halogen detector (Fig. 4a) contains a low voltage power supply for the heater. Another power supply delivers a few hundred mA at potentials between 50 and 500 V alternating current or direct current for use as the interelectrode field for the sensing element. Sufficient current amplification is contained in the detector circuit to make a small increase in direct current signal due to ion emission variation readily detectable.

Indication of Signal Current in Halogen Leak Detector

There are several ways by which the increases of current due to exposure to halogen gas may be indicated on the halogen leak detector. The simplest is by means of a microammeter or a galvanometer. Another method is to use the change in voltage across a high resistance to control an amplifier. This in turn operates a relay that activates visible or audible alarms or external recorders. A third method is to use a relaxation oscillator incorporating a capacitor and glow discharge tube with a loudspeaker as an audible indicator. The current through the sensing element builds up a charge in the capacitor. When the voltage is sufficiently high, the glow discharge tube operates and the pulse of current resulting from the discharge of the capacitor produces a click in the loudspeaker. The repetition rate of the click is an indication of the amount of current and the rate of voltage buildup caused by introduction of halogen tracer gas.

With any circuit used, it is desirable to include a protective resistor of about 100 kΩ to prevent overloading or damage to the sensing element or indicating device. Such damage could result from an overdose of tracer gas or a short circuit between sensor electrodes.

Applicability of Heated Anode Halogen Leak Detector

Equipment for heated anode halogen detector leak testing is primarily built for detector probe leak location with a detector probe operating in atmospheric air. However, this equipment may be used without modification in static accumulation leakage measurements. The major advantage of halogen detectors is that they are designed to operate in air at ambient pressure.

Sensitivity of Heated Anode Halogen Leak Detector

The halogen leak sensitivity of the heated anode detector probe instruments operating in air at atmospheric pressure is in the range of 1 $nL \cdot L^{-1}$ (part per billion) of halogen in air. This corresponds to a leakage rate of 1×10^{-10} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-9} std $cm^3 \cdot s^{-1}$) using the standard air pumps (about 1 $cm^3 \cdot s^{-1}$ or 0.1 $ft^3 \cdot h^{-1}$)

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on the detector. The sensitivity of the heated anode detector varies with different halogen compounds.

Operational Controls of Halogen Vapor Detectors

The control features of typical commercially available halogen leak detectors may include the following.

1. A volume control permits the operator to adjust the loudspeaker to give an audible signal at any predetermined leakage rate within the leak detector's range.
2. A sensitivity selector permits the operator to adjust the full scale meter range to leakage rates from 3×10^{-10} to 3×10^{-6} Pa·m^3·s^{-1} (3×10^{-9} to 3×10^{-5} std cm^3·s^{-1}) in ten equal steps.
3. An automatic manual balance selector allows the operator to read total concentration of halogen in the area when the selector switch is in the manual position. In the automatic balance position, the leak detector responds only to sudden changes in halogen concentration. Some portable halogen leak detectors are small and simple and contain no adjustment switches other than a continuously variable sensitivity adjustment switch. The automatic balance feature is an integral part of the detector and remains in the circuit at all times.

Function of Automatic Balance in Halogen Vapor Leak Detectors

The automatic balance feature in heated anode halogen vapor detectors is particularly useful in regions where a high halogen concentration is present in the air. A detected leak is signaled by a sudden pointer deflection on the leak indicating meter. If the detector probe is held on the leak, the pointer will maintain the signal level until the leak detector rebalances to the increased concentration of halogen surrounding the leak. The pointer will then return to zero and automatically maintain this position despite varying background concentrations of halogen vapors.

In the manual balance position, the leak detector responds to sudden changes in halogen concentration and also responds to any halogen in the surrounding atmosphere. For example, if the detector probe is held on the leak, the pointer will maintain the signal reading; it will not return to zero setting. To compensate for constant level background halogen concentrations, the balance control is reset to zero.

Types of Detector Probes Used in Halogen Vapor Leak Test Units

Some halogen leak detectors for use in air have a gun shaped detector probe held in the hand to probe areas where leakage is suspected in pressurized systems. The leak signal can be seen both on a meter in the hand held gun and on a meter in the control unit. An adjustable audible alarm is also provided. Other systems provide a detector probe gun designed to be mounted permanently in a fixed location as in assembly line leak testing.

Another type of halogen vapor detector uses a pencil shaped probe that can be inserted into otherwise inaccessible regions. The sensitive element is located in the control unit, rather than in the detector probe itself.

Halogen leak detectors for use in vacuum may have a detector unit designed to be connected to a vacuum system in which a leak is to be detected. The optimum pressure operating range of vacuum detectors is between 0.5 and 2 Pa (4 and 15 mtorr) but they can be used satisfactorily at absolute pressures between 0.1 and 50 Pa (1 and 400 mtorr).

Function of Air Proportioning Halogen Detector Probes

An air proportioning nozzle is used in some halogen leak detector probes. Halogen free air is provided by passing ambient air through a charcoal filter in the control unit. It removes all traces of halogen. The purified air is then pumped through a second hose to the proportioning nozzle. At the nozzle, this clean air can be mixed in any proportion with the tracer gas and air mixture that is being sampled with a detector probe during leak testing. This mixture is then carried by hose and drawn past the sensitive element in the control unit. In this manner, it is possible to obtain high halogen test sensitivity and stability in a contaminated atmosphere. No leakage sensitivity is lost as long as the intake flow is adjusted at a level great enough to pick up all the leaking gas. However, prolonged exposure to excessive amounts of halogen can cause loss of sensor sensitivity.

Operating Lifetimes of Heated Anode Halogen Vapor Detector Sensing Elements

The sensitive elements used in different models of heated anode halogen leak detectors vary in sensitivity and useful life. The highly sensitive elements used in industrial leak detectors have expected lifetimes of 500 to 1000 h with proper maintenance and operation. The less

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sensitive elements used in the leak detectors of service personnel are much smaller and with proper usage should last 50 to 100 h. The life of the element is greatly shortened when large amounts of halogen gas pass through the sensing element. Thus, it is very important to keep the atmosphere in the test area clean and free from halogen gas contamination. Atmospheric contamination usually results from large leaks or from dumping gas in the area after tests are completed. If the atmospheric contamination reaching the sensor continually contains more than a few parts of halogen gas for each million parts of air, the life of the sensitive element will be shortened considerably.

Some battery operated halogen leak detectors have limited duty cycles. The detector cannot be continuously operated for more than 1 h at a time and *off* time must be at least equal to *on* time.

Advantages of Heated Anode Halogen Vapor Detector Leak Testing

Advantages of the heated anode halogen leak detector are that it (1) operates in air at atmospheric pressure, (2) responds specifically to halogen compound tracer gases, (3) may detect oil clogged leaks and (4) is portable, efficient, safe and simple to use.

The greatest advantage of the heated anode halogen detector is that the detector operates in air at ambient pressures on the earth's surface. It can therefore be operated efficiently as a detector probe and can be used without vacuum pumping equipment. The detector is relatively inexpensive and portable. It can be used by leak testing personnel without extensive instruction. Another major advantage is that the detectors are specific for halogen compounds. Although halogen contamination in the atmosphere will sometimes present a problem, the specificity leaves no doubt when the tracer gas is being measured.

Another advantage of the halogen leak detector stems from the fact that halogen containing gases are soluble in oil. Oil effectively plugs small leaks against internal pressure. Even high pressure differences are incapable of clearing oil because of the small area of the hole. However, oil has a high solubility for halogen gases. Consequently, the halogen gases diffuse through the oil clogging the leak and can be picked up by the sensitive halogen detector. The detector can be operated in a quantitative mode determining the relative size of individual leaks.

Disadvantages of Heated Anode Halogen Vapor Detectors

Disadvantages of heated anode halogen leak detectors include the following.

1. Many of them are hazardous to use near flammable materials or in explosive atmospheres.
2. They respond to residual contaminants that contain halogen compounds.
3. Except for models in which the sensor is in the hand piece, a time lag in their signal response results from detector probe hose gas transit time.
4. Their leak signals tend to disappear with prolonged exposure to halogen tracer gases.
5. Their sensing elements deteriorate with time or by excessive exposure to halogen gases or vapors.

A primary disadvantage of some heated anode halogen vapor detectors is that they can be dangerous in any atmosphere that contains combustible or explosive gases. A second disadvantage of heated anode detectors is that they respond to any gases that contain halogen compounds. For example, detectors will respond to solder fluxes, cleaning compounds and aerosol container propellants. Care must therefore be taken that these halogen containing compounds are not present in the test area.

Linearity and Speed of Response of Heated Anode Halogen Vapor Detector

The linearity of halogen leak detector response depends on halogen tracer gas concentration. A positive ion current flows at all times from the emitter to the collector of the diode. When trace amounts of halogen gas are present, the ion current increases linearly with concentration of halogen in the range from 0 to 1 $\mu L \cdot L^{-1}$. For concentrations between 1 and 1 000 $\mu L \cdot L^{-1}$, the signal increase is an exponential function of halogen concentration. Above 1000 $\mu L \cdot L^{-1}$, no further increases of ion current occur and the sensing element desensitizes rapidly. The readings for concentrations above 1 $\mu L \cdot L^{-1}$ are strictly instantaneous and cannot be maintained.

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Thus, halogen leak signals tend to disappear as detector exposure to tracer gas is prolonged.

Although some models have the sensor located in the hand piece, in other models there is the necessity of transferring the gas sample from the detector probe through a hose connection to the heated anode, which may be some distance away. A time lag therefore ensues between the positioning of the probe and the response of the instrument. In continuous probe scanning, the leak signal may perhaps be detected only after the probe has passed by the leak site.

Leakage Signal Indicators Used with Heated Anode Detectors

An amplifier increases the signal from the heated sensor element enough to trigger a leak signal. The leak signal can be either a signal light or a panel meter indication or an audible signal, usually from a built-in speaker or in headphones worn by the test operator when leak testing in noisy environments.

Materials Used in Heated Anode Halogen Sensors

No matter which model of heated anode halogen leak detector is used, the principle of operation is always the same. In the halogen sensitive element or sensor shown in Fig. 4a, the emitter consists of a cylindrical platinum container that houses a specially treated ceramic material. The collector is a platinum wire coil wound helically round the emitter and electrically insulated from it. The collector is heated to about 900 °C (1650 °F). Alkali atoms from the treated ceramic material migrate to the surface of the heated platinum emitter cylinder. The presence of a halogen bearing gas causes the alkali to leave the emitter and become ionized on the heated platinum surfaces.

A direct current voltage impressed between collector and emitter results in a current flow through the air between the elements when the ionized alkali is present. When there is no halogen gas in the air passing through the element, the ion current is very small. As the halogen gas concentration increases, the ion current increases up to some useful limit of halogen vapor concentration (about 10 $\mu L \cdot L^{-1}$).

Temporary Desensitization of Leak Detector by High Halogen Concentrations

When a high halogen gas concentration goes through the detector, all available ions are released from the emitter. A significant time is required to again build up the needed alkali ions on the emitter so it can be used again for leak testing. The technique of measuring atmospheric contamination and the means of keeping contamination low are described below.

Detecting Refrigerant Leaks with Portable Halogen Detector

The portable leak detector (Fig. 5) is used to find leaks in installations where a benchtop instrument would be awkward. Because halogen gas is heavier than air, leaks may be detected just below the actual source. The probe is moved along suspected seams, joints or fittings at about 20 $mm \cdot s^{-1}$ (1 $in. \cdot s^{-1}$). When a leak is found, its presence will be indicated by the signal light emitting diodes and audio signal.

The leak detector incorporates an electrochemical sensor comprising a ceramic substrate doped with a reactive element and maintained at high temperature by a built-in heating element. When a halogen bearing gas contacts the hot surface, the chlorine, fluorine or bromine atoms are separated from the molecule and ionized, causing an electrical current to flow within the ceramic to a collection electrode at the center.

FIGURE 5. Portable battery operated halogen leak detector for refrigerators and air conditioners.

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Serviceman's Leak Detector for Alternating Current Operation

The halogen leak detector of Fig. 6 contains a printed circuit board amplifier, plug-in sensitive element, air pump and power transformer housed beneath a chassis cover. A reference leak, sensitivity switch and a balance control are located on the top of the control chassis. The sensing probe, comprising a nozzle with a transparent probe tip, air flow indicator ball and neon leak signal lamp, is connected to the control chassis by a length of flexible tubing. A speaker provides a variable pitch audio signal when a leak is detected. The reference leak assembly is a built-in bottle containing refrigerant gas refrigerant-134a and is calibrated to provide a leakage rate equal to about 15 g (0.5 oz) of refrigerant gas per year. The accessories provided with the leak detector consist of a maintenance kit, filters and airflow indicator balls.

The sensor uses a positive ion emission technology, commonly known as the heated diode. It is very sensitive to only halon substances (refrigerants), making the instrument resistive to false alarms while retaining sensitivity for pinpointing refrigerant leaks difficult to find otherwise.

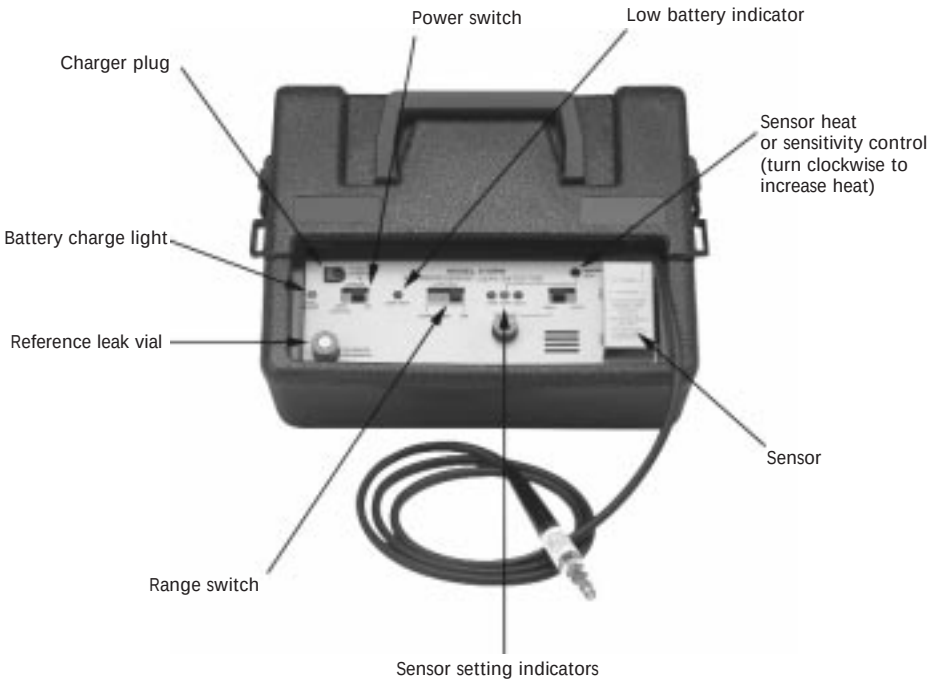
A pump inside the unit draws air through the sensor. Any presence of halogen gases causes an ionized current to flow that sounds a speaker and illuminates a neon light in the probe. Sensitivity to pinpoint both large and small leaks can be controlled by adjusting the three-position switch.

At the most sensitive of the three settings, the instrument will indicate 3 $g \cdot yr^{-1}$ (0.1 $oz \cdot yr^{-1}$) or greater leakage rates and is used for fluorine based gases like refrigerant-134a and sulfur hexafluoride. The instrument's medium setting is used for chlorine based gases such as refrigerant-22.

The least sensitive setting is used to locate gross leaks of any refrigerant. If a large leak is suspected, switch the unit to manual balance mode, adjust the sound to two to three ticks per second and slowly approach the test area. Continue to turn the balance knob counterclockwise as necessary to maintain two to three ticks per second. As the vehicle or equipment is approached, the gas concentration will increase. Each time an alarm occurs, readjust the balance and continue the process until the leak is located. Blowing out the test site with shop air may help the leak to be located more quickly.

The leak detector of Fig. 6 was designed primarily for use as a tool by service personnel. Although it can be used for production line leak testing, it was not designed for this type of service. Normally, the service technician will use it for leak testing automobile air conditioning, home and/or commercial air conditioning or refrigeration equipment.

FIGURE 6. Components and controls of refrigerant leak detector.



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Industrial Halogen Leak Detector

The industrial halogen leak detector of Fig. 7 can detect extremely small leaks when the unit to be tested is pressurized with refrigerant (refrigerant-12) gas. Nominal full scale sensitivity is 9×10^{-7} to 9×10^{-5} Pa·m^3·s^{-1} (9×10^{-6} to 9×10^{-4} std cm^3·s^{-1}) under clean air conditions. Leaks are indicated visually by a panel meter mounted on the detector and by a light on the probe and also by a speaker that produces an audible tone proportional to leakage indication. These leak detectors can be calibrated to read directly in Pa·m^3·s^{-1} or std cm^3·s^{-1} by using a suitable calibrated standard reference leak.

When current is applied to the coiled heater wire, the temperature of the sensor assembly is raised to about 900 °C (1650 °F) causing a small current to flow in the core rod. This small current flow, because of ionization of the core material, increases linearly to a useful limit in proportion to the amount of halogen in the air or gas passed through the assembly. Beyond this limit, however, the increase in current is extremely nonlinear and excessive increases in halogen in the gas only serve to shorten the life of the sensor.

The actual current between the halogen sensor's emitter and collector (with and without halogen present in the assembly) is very low. A high gain amplifier circuit generates a signal proportional to the halogen leakage rate displayed on a leakage rate meter to provide a visual indication of leak size or leakage rate. The amplified signal is also used to trigger an audible alarm through a speaker and visible alarm through a solid state lamp on the hand held searching probe.

FIGURE 7. Industrial halogen vapor leak detector.

Relation of Alkali Diode Leak Signal to Halogen Vapor Concentration

The output signal from the alkali diode halogen sensor element is an electric current whose magnitude varies with the halogen vapor concentration within the cylindrical sensing element. This is a positive ion current emitted by the heated anode and is aided by the action of the halogen.

Operational Characteristics of Industrial Halogen Leak Detector

The industrial leak tester of Fig. 7 operates in high background contamination that may preclude other methods of leak testing. The unit provides a pencil shaped probe to make it easier to detect leaks in confined areas. If dropped, no costly damage to the detector will result. The response time is 1 s with a 0.3 m (3 ft) probe. Any extension of the normal cable length will be at the expense of response time.

Air Flow System of Industrial Halogen Leak Detector

The schematic diagram for a detector probe air flow system of an industrial halogen leak detector is shown in Fig. 8. The hand held probe is designed for detecting leaks in industrial pressurized or vacuum systems where halogen compound gases are used. A pump draws a gas sample in through the detector probe tip and through a filter to remove particulate matter. The heated anode halogen sensor provides positive halogen ions from the emitter that pass to the ion collector, which operates at a negative potential. This collector current is amplified to provide the leak signal.

The detector unit's leakage rate meter indicates the presence and size of a leak in conjunction with the sensitivity selector switch. The unit contains a speaker, which provides a scale adjustable audible alarm in the event that a predetermined leak or leakage rate is detected and the leakage rate meter is not visible. The hand held probe also has a scale adjustable alarm lamp that lights when a predetermined leak size or rate of leakage is encountered.

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Leak Detector Air Flow System to Reduce Problems from Background Contamination

An industrial leak detector incorporates a background reducing air flow system as sketched in Fig. 9. A bellows pump pushes air through an activated charcoal filter and circulates it past the tip of the probe, where the outside halogen laden sample air is drawn in by aspiration. The filtered air, mixed with the sample air, flows through the probe back through the manifold to the sensor and thence to the pump. The pump drives the air back to the filter where a portion equal to the quantity taken in at the probe tip is exhausted, the remainder is recirculated through the filter and the cycle is repeated. The design of the filter assembly for the probe tip not only protects the air circuit from dirt but also limits the intake of the sample air to roughly a tenth of the total flow.

Although the intake flow of sample air is low, it remains many times greater than the largest leakage rate measured by the leak detector. As a result, whenever the probe encounters an actual leak, the sample is completely absorbed and measured. At all other times, the air taken in by the probe amounts to only about one tenth of the total flow in the air circuit. Therefore, regardless of the background level of the environment, the leak detector is forced to handle only a tenth of the steady state contamination.

The air system manifold is positioned in the circuit adjacent to the entrance to the sensor. Its construction is such that a portion of the airflow bypasses the sensor and the remainder is further mixed with filtered air before encountering the sensor. As a result, the manifold accounts for about another ten-to-one reduction in the

FIGURE 8. Air flow connections for halogen leak detector: (a) detector connections; (b) manifold detail.

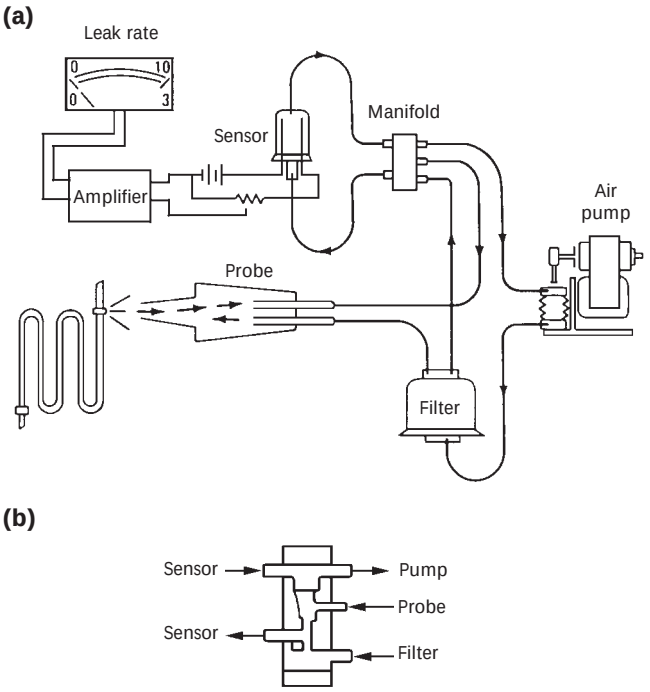
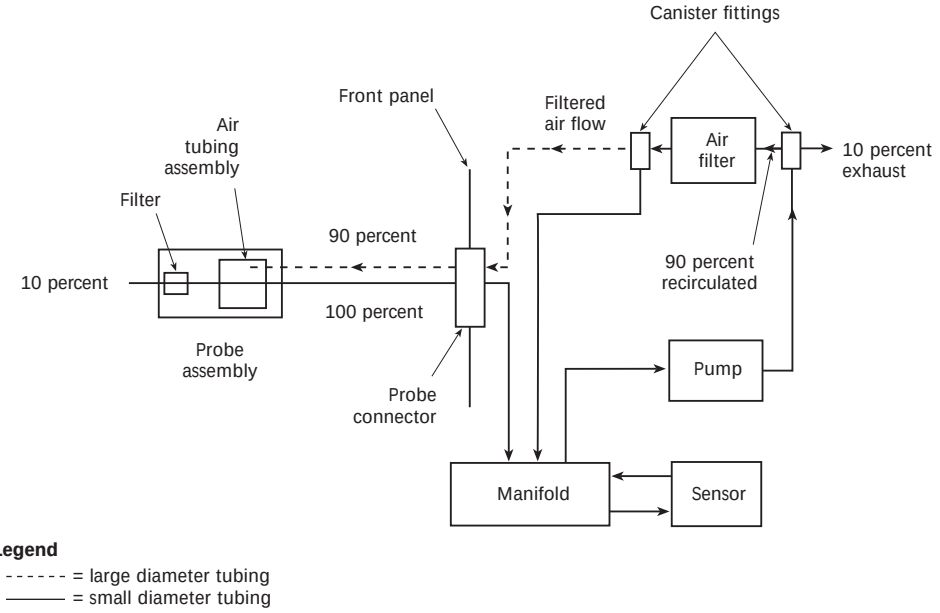


FIGURE 9. Air flow system for representative industrial halogen vapor leak detector.



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halogen exposure of the sensor. These combined reductions of halogen levels provide twin benefits of prolonged halogen sensor life and of improved sensitivity.

Factors Influencing Sensitivity of Halogen Leak Detector

One of the characteristics of the positive ion probe under operating conditions is the small amount of current that flows even in the presence of pure dry air. This leakage current is usually of the order of 1 to 10 mA. In the presence of an air diluted vapor of a chemical element or compound to which the device is sensitive, this current will increase several fold.

The probe's sensitivity to a halogen compound vapor varies with the velocity of air flow between its electrodes. With an air speed of the order of 20 $mm \cdot s^{-1}$ (50 $in. \cdot min^{-1}$) or less, there is an extreme sensitivity to some vapors of about 1 $\mu L \cdot L^{-1}$. With an air speed of more than 0.30 $m \cdot s^{-1}$ (60 $ft \cdot min^{-1}$), the sensitivity and response are reduced to a point where there is little response. If the air speed is too rapid, then the vapor apparently has only limited opportunity to strike the hot cathode and dissociate. The sensitivity of the device, therefore, decreases as the air flow is increased.

Part of the decreased sensitivity is due also to the additional cooling of the anode with the increased air flow. On the other hand, if the air flows at too low a rate, the device will be extremely responsive to vapors to which it is sensitive. However, considerable time will elapse before the ion current returns to its normal no-vapor condition even after the inlet is again given a supply of pure air.

The heated anode probe is very sensitive to carbon tetrachloride, chloroform and dichlorodifluoromethane. At room temperature it does not respond to chlorinated phenol. However, if the chlorinated phenol is heated to 60 °C (140 °F) or more, the vapor pressure becomes sufficiently high to give a response. It also responds to solid particles of the iodides, chlorides, bromides and fluorides. Therefore, it detects smoke from burning materials containing such compounds.

Maintaining Sensitivity of Heated Anode Halogen Leak Detector Sensor

The heated anode halogen vapor detector is not affected significantly by exposure to elemental halogen vapors, provided this exposure is limited to a relatively short time. If the time the sensing element is exposed to a halogen vapor is too long or if it is exposed to too highly concentrated halogen vapor, it may lose its sensitivity. Long operation in pure air with voltage between the electrodes will restore its sensitivity if the contamination has not been too great. If the emitter contamination has been too great, it may be necessary to clean or replace the electrodes. The ease of contamination varies greatly from one compound to another; for instance, carbon tetrachloride contaminates the electrodes more easily than refrigerant-12.

If the sensing element has been hot for some time in the absence of an interelectrode voltage, a heavy transient current flows when voltage is applied. The time taken to return to normal depends on the time the element has been hot. A similar transient rush of ion current occurs if the interelectrode voltage is interrupted momentarily and then reapplied.

The response of the sensing element increases markedly with the ion source temperature over a narrow range. Below about 850 °C (1560 °F), the emission current is too small to be easily used. Over about 950 °C (1740 °F), it becomes unstable and random fluctuations will hide any signal.

It is necessary to keep the space between the electrodes free from dust, lint or other particles that may be sucked in by the air flow. Such particles would short circuit the electrodes and give false indications. It is usually desirable to filter the incoming air supplied to the heated anode halogen vapor detector.

Operating Lifetimes of Heated Anode Halogen Leak Sensors

The heated anode elements used in industrial leak detectors may be expected to last 500 to 1000 h with proper maintenance and operation. However, those elements used in more portable leak detectors are much smaller and may not last as long. In general, little can be done to extend the life of these units. However, the smaller elements are much lower in cost.

Electron Capture Technique of Halogen Vapor Leak Detection

The electron capture technique of halogen leak detection uses the affinity of the halide gases for electrons. It eliminates or minimizes most of the difficulties encountered with the heated anode detectors. In this technique a small amount of a gas that does not capture electrons, generally nitrogen or argon, is

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used as the background gas. (Air cannot be used as the background gas, because oxygen is an electron capturing gas.) The background gas flows through the sensor element where it is ionized to produce free electrons by a weakly radioactive tritium source. Tritium ($_1H^3$) decays by electron emission to helium three ($_2He^3$) with a half life of over 12 yr. The electron is of comparatively low energy (19 kV), so that radiation shielding is no problem so long as the source (typically a titanium foil impregnated with tritium) remains sealed within the sensor element.

In use, air from the leak detecting probe is also drawn through the sensor. When this air contains halide gases, electron capture occurs, thereby reducing the electron current between the electrodes. This reduction in current is the measure of the concentration of the halogen ions present.

Advantages of Electron Capture Halogen Detector

The chief advantage of the electron capture detector over the heated anode halogen detector is calibration stability. Although it can be desensitized temporarily, the electron capture sensor cannot be damaged nor can its calibration be changed by any amount of use or overexposure. Another advantage is that there is no heated element that could constitute a hazard.

Leak testing instruments based on the electron capture method appear to be considerably less sensitive than heated anode halogen detectors to detectable contaminants (such as smoke particles) in the ambient atmosphere. This method also seems to be particularly effective with sulfur hexafluoride (SF_6) as a tracer gas. Further, its sensitivity to some common halogen tracer gases is comparable to that of the heated anode instruments. It can be used over a wider range of leakage rates. Maximum sensitivity is claimed to permit detection of leakage rates of 10^{-12} Pa·m^3·s^{-1} (10^{-11} std cm^3·s^{-1}).

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PART 2. Introduction to Techniques of Halogen Leak Testing

Halogen Leak Detector Components

The typical halogen leak detector system consists of a halogen sensing element (Fig. 10), a small air pump to draw the leakage sample through the sensing element, power supplies and amplifiers to provide signal outputs. Leakage signals are usually evidenced by an instrument indication, a variable frequency audio signal and a relay operating a leak signal lamp or external alarm. The air sample is drawn through the sensing element or probe at about 0.5 $L \cdot min^{-1}$ (30 $in.^3 \cdot min^{-1}$).

Halogen Detector Probe Leak Search Procedure

When searching for leaks from a vessel pressurized with a halogen tracer gas, the probe tip is moved over joints and seams suspected of leaking (Fig. 11). Certain precautions are necessary in this probe exploration. Searching too rapidly may miss the very small leak. If this risk is to be avoided, the speed at which the probe is moved must be in proportion to the minimum leakage tolerance. In testing welded seams for an allowable leakage of the order of 10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} std $cm^3 \cdot s^{-1}$), probe travel speed can be about 20 to 50 $mm \cdot s^{-1}$ (50 to 120 $in. \cdot min^{-1}$). However, the probe speed should be reduced to 10 $mm \cdot s^{-1}$ (25 $in. \cdot min^{-1}$) for smaller leaks.

The probe tip should lightly touch the surface as it is moved over the test object (Fig. 12). External forced ventilation must be stopped during the actual leak testing or care must be exercised to make certain that drafts do not blow the leaking gas away from the test probe. When the probe passes over or close to a leak, the tracer gas is drawn into the probe with the air and through a sensitive element where it is detected. The leak signal is either audible or visual.

FIGURE 10. One-piece halogen sensing element used in halogen leak detector.

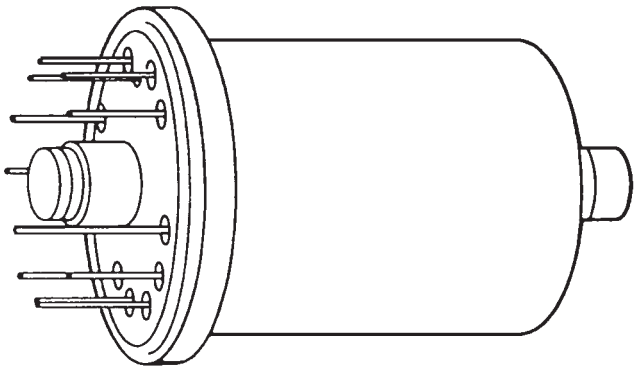
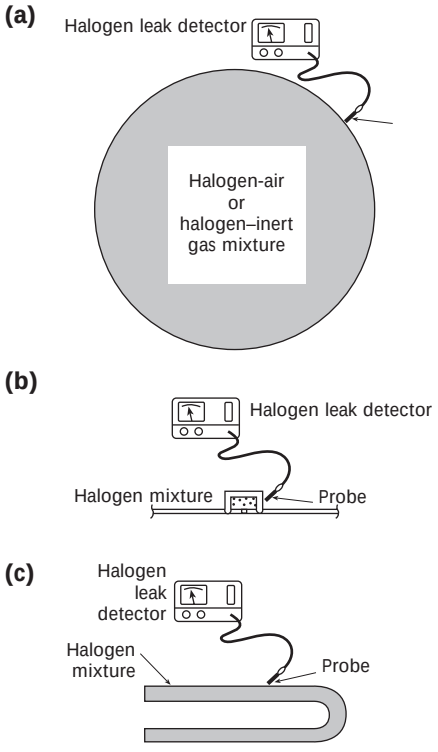


FIGURE 11. Examples of halogen detector probe technique for detecting halogen tracer leaks from pressurized systems, weld seams and components: (a) pressurized vessel or air lock; (b) leak chase channel; (c) pipe coil.



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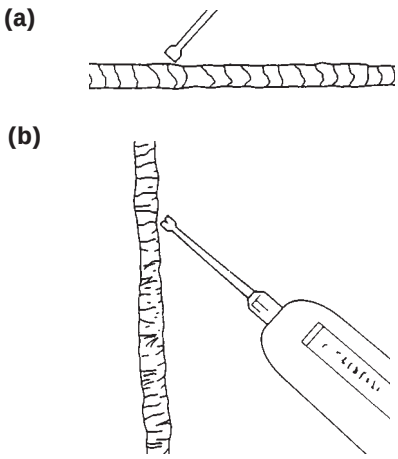
Technique for Halogen Leak Testing of Vacuum Systems

For locating leaks in a vacuum system, a special leak detector is used. The control unit is the same as for the pressure application. However, the sensor of the halogen leak detector is located separately in a 16 mm (0.625 in.) diameter pipe section about 100 mm (4.0 in.) long, with one end closed. The open end of the detector assembly is sealed into the vacuum system. A jet of halogen tracer gas is used to probe the outside of the evacuated system with tracer gas, in the same way that helium is used in tracer probe leak testing with the mass spectrometer leak detector. When the jet of tracer gas is on a leak, the tracer gas leaks into the vacuum system, reaches the sensitive element and is detected. When testing small evacuated volumes of about 50 L (1 to 2 ft^3), leakage of about 10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-6} std $cm^3 \cdot s^{-1}$) can be detected. The response time is usually 1 to 2 s. During testing of large volume vacuum systems, when the system being tested has restrictions to flow of gases between the leak and the detector, the sensitivity of the leak detector is reduced by a factor of ten or more and the response time will be increased, as described below.

FIGURE 12. Manipulation of detector probe when locating leaks by halogen tracer gases. Suction pump causes probe to inhale air and tracer gas escaping from leaks in pressurized systems: (a) halogen detector probe lightly touching weld during manual scanning of test surface for leakage; (b) notched plastic tubing tip on halogen detector to maintain proper distance above leak surface.



Limitations of Halogen Leak Detector in Operating Vacuum Systems

When the halogen sensor is operated in a pumped vacuum system for vacuum leak detection, tracer gas movement within the evacuated volume takes place by diffusion rather than by positive displacement pumping action at atmospheric pressure. This makes response to tracer gas and recovery from tracer gas application much slower, particularly when testing long, large or restricted devices or systems. In addition, the quantitative accuracy of halogen leak testing is adversely affected by operation in vacuum. The equipment cost is greatly increased due to the addition of vacuum pumps. For these reasons, the use of the halogen sensor in vacuum should be avoided when possible, except where vacuum systems with pumps are in operation.

Halogen Tracer Leak Testing When Vacuum System is Subsequently Filled with Air

If a total leakage test under vacuum with halogen tracer gases is a necessity, the halogen detector can still be operated at atmospheric pressure by a very simple expedient. First, the halogen tracer gas is applied to the exterior of the evacuated device under test. If leaks exist, some halogen gas enters the vacuum enclosure. Then the vacuum is broken by filling the enclosure with halogen free air at atmospheric pressure. Then a test is made for halogen content due to leakage in the air tracer gas mixture within the enclosure.

Calibration of Halogen Leak Detectors with Reference Leaks

The heated anode halogen leak detectors do not have inherent fixed sensitivity. Detector sensitivity drifts in some relation to (1) the number of hours the sensitive element has been used, (2) the amount of halogen compound gas to which the sensitive element has been exposed and (3) the temperature of the detector. Response is also affected by other variables, as described above. For these reasons, the halogen leak detector must be calibrated with a reference standard halogen leak.

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Setting Halogen Leak Detector to Reject a Specified Leakage Rate

For the pressure mode type of leak testing, a calibrated reference halogen leak is used (Fig. 13). When it is desired to reject all leaks equal to or greater than a specified rate, the halogen reference standard leak is adjusted to leak at that reject level. Then the leak detector is allowed to probe for the standard leak and the leak detector is adjusted to give a half scale signal on the panel instrument of the leak detector. The leakage signal is then observed. When searching for leaks, a signal equal to or greater than that indicated when using the reference standard leak indicates that a leak has been detected that should be repaired or rejected. Fig. 14 shows examples of typical standard leak settings on the flow meter dial gage of the reference halogen leak shown in Fig. 13.

Calibration of Vacuum Halogen Leak Detector

For calibrating the vacuum leak detector, calibrated leak capillaries (glass tubes) are available. This type of leak contains no tracer gas. One end is covered by a cap and the other end must be sealed into a vacuum system. When the calibrated leak is used, the cap is removed and the exposed open end is blanketed with halogen tracer gas. Refrigerant-134a leaks into the system at a known rate, so the leak detector can be calibrated. More than one calibrated leak is sometimes used to determine the detector response to leaks of the same size in different parts of the vacuum system. This is particularly important for leak testing of large volumes or complex volumes where there are restrictions between the leak and the detector. Also, should one calibrated leak become plugged, the second calibrated leak can be used to check the performance of the leak detector. These leak capillaries are available for almost any leakage rate from 10^{-10} to 10^{-4} Pa·m^3·s^{-1} (10^{-9} to 10^{-3} std cm^3·s^{-1}).

FIGURE 13. Halogen tracer gas reference standard leak with adjustable leakage rate is used to calibrate halogen leak detectors for leakage rate. Internal reservoir of refrigerant-22 or refrigerant-134a halogen tracer gas and flow regulators permit leakage rate from probe outlet to be controlled and indicated by dial gage.

FIGURE 14. Examples of dial gage indications showing typical standard leakage rate settings: (a) 1.8×10^{-6} Pa·m^3·s^{-1} (1.8×10^{-5} std·cm^3·s^{-1}); (b) 6.1×10^{-7} Pa·m^3·s^{-1} (6.1×10^{-6} std·cm^3·s^{-1}); (c) 7×10^{-8} Pa·m^3·s^{-1} (0.7×10^{-7} std·cm^3·s^{-1}).

(a)

(b)

(c)

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Preparation and Tests Preceding Halogen Detector Probe Leak Tests

The following preliminary operations are used before halogen testing in one large industrial fabricating facility.

1. As with all nondestructive examinations or tests, remove slag from weld areas and clean all test surfaces. Inspect visually to locate and repair any areas suspected of bad welding or obvious bad workmanship before conducting halogen detector probe leak tests.
2. Any test object to be leak tested by a halogen detector probe must be free of standing water. If a test object is to be both halogen detector probe tested and hydrostatically tested, the halogen test should be performed first in order to detect leaks that could later be temporarily plugged with water during hydrotesting.
3. Except when pressurizing by the leech box technique, perform a solution film bubble emission air pressure test before conducting a halogen detector probe test. A properly performed solution film test and repair of detected leaks eliminates large leakage that can cause background contamination and reduces or eliminates most of the other leakage. This ultimately reduces total test time by decreasing the necessity for multiple repetitions of the halogen detector probe test.
4. Before performing a halogen detector probe test, leak test all test equipment connections to detect and eliminate leakage in these areas as a source of background contamination. The test connections should be retested each time the equipment is connected for a new test.

Injecting Halogen Tracer Gas and Pressurizing Systems for Leak Testing

A leak test manifold (Fig. 15) is used for pressurizing with both refrigerant and air or nitrogen. Never use oxygen or combustible gases such as propane or acetylene as the pressurizing gas for halogen leak testing. The manifold should be assembled with solder joints and refrigerant fittings and hose. It is essential to pressurize with premixed gases unless the device under test is of a compact shape without small blind extensions. When these exist, air should be evacuated before pressurizing. This ensures the dispersion and mixing of the refrigerant throughout the test system. The maximum refrigerant gas pressure possible from a bottle of refrigerant-22 or refrigerant-134a can be determined from Fig. 1 for a wide range of ambient temperatures.

The type of tracer gas, the required gas pressure and the required leak testing sensitivity or leak test system sensitivity are usually listed in the test procedure. However, the resulting halogen mixture in percent by volume, which must be known to calibrate a leak detector for a specific test, is not always given in the instructions. This halogen tracer concentration figure can be obtained from Figs. 16 and 17 and Tables 4 to 6.

Precautions in Use of Halogen Tracer Gases and Heated Anode Halogen Detectors

The halogen detector probe should never be placed in a stream of pure refrigerant from a cylinder. Such exposure to concentrated tracer will temporarily or permanently contaminate and shorten

FIGURE 15. Leak test manifold for pressurizing a system with both refrigerant and air or inert gas: (a) manifold for separate injection of tracer gas and later injection of air or inert gas; (b) manifold for simultaneous mixing and pressurizing with both refrigerant and air or inert gas. It is preferred that it be assembled with solder joints and refrigerant fittings and hose on the refrigerant portion of the manifold.

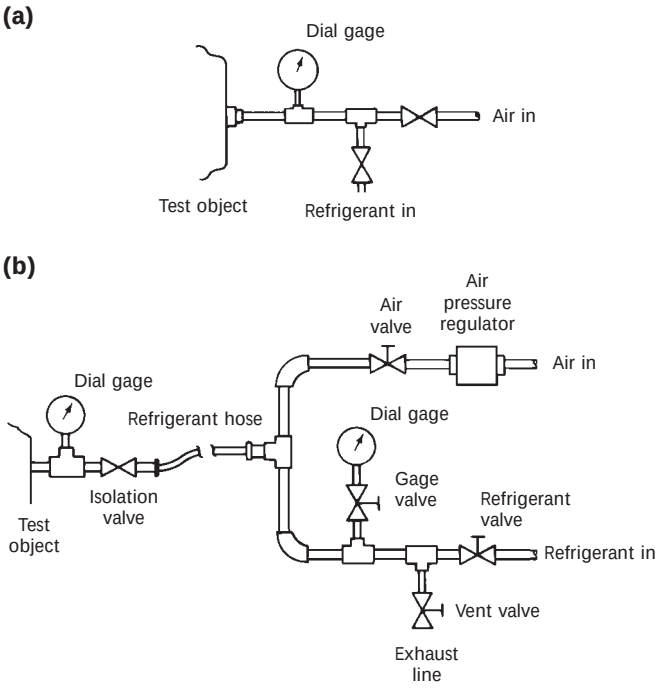

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FIGURE 16. Halogen concentration (percentage by volume) for a system evacuated to 7 kPa (1 $lb_f \cdot in^{-2}$ absolute) and pressurized with halogen tracer gas and air.

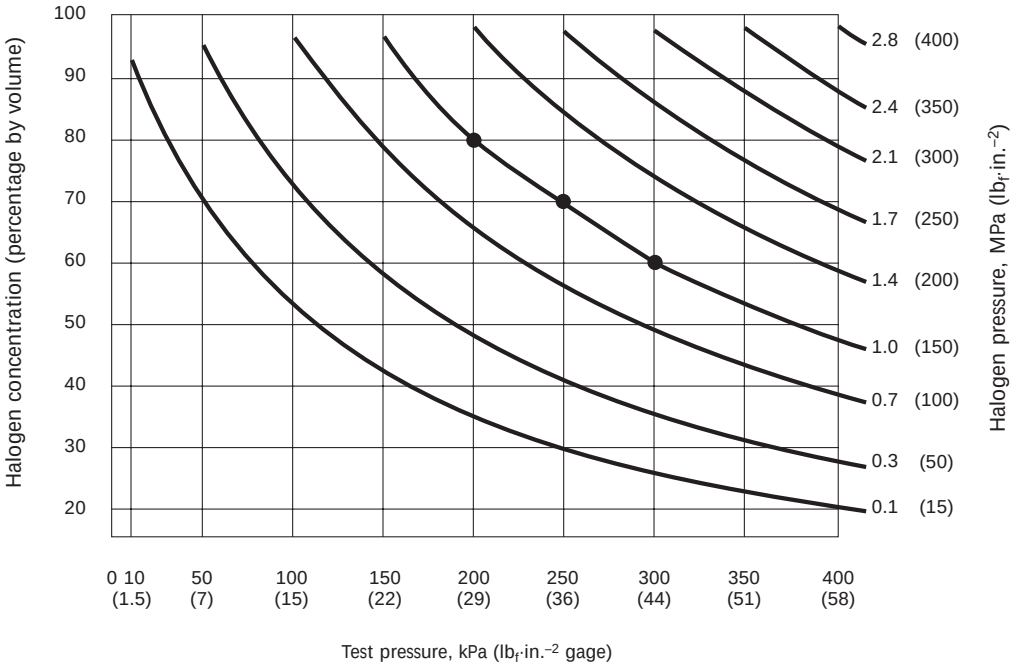
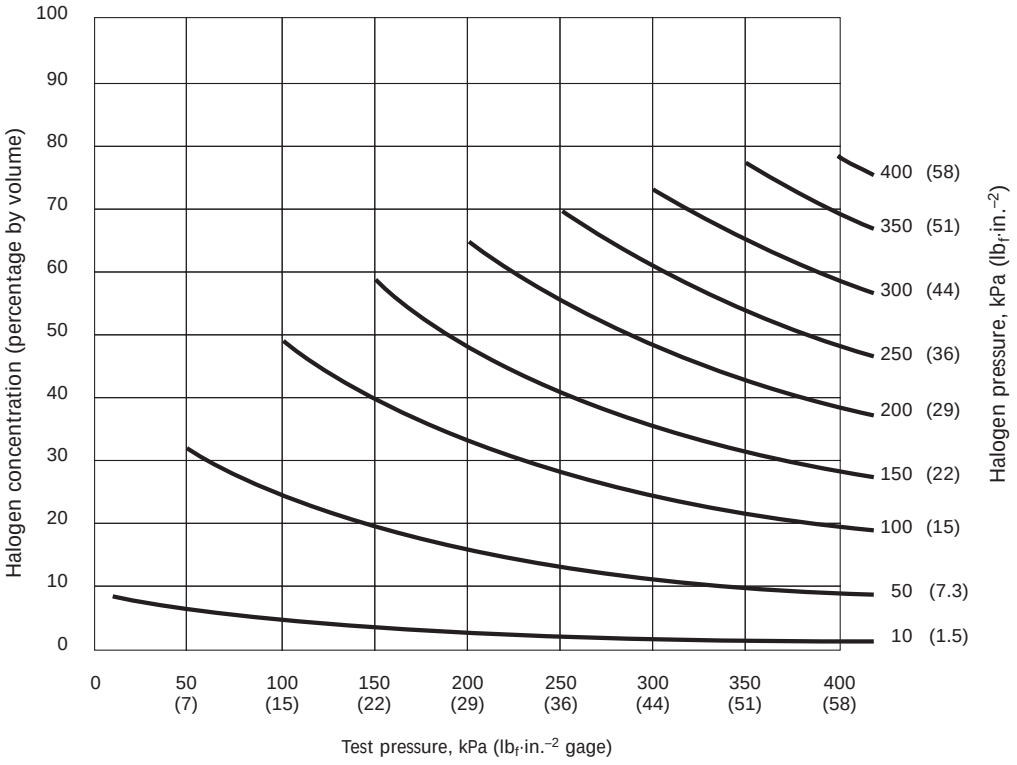


FIGURE 17. Halogen concentration (percentage by volume) for a system containing air at atmospheric pressure (100 kPa or 14.7 $lb_f \cdot in^{-2}$ absolute) and pressurized with halogen tracer gas and air.



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the life of the instrument's heated anode sensing element. It will either cause delay due to the long time it takes for the element to clear or necessitate the replacement of the sensing element.

Do not use positive ion or halide torch types of halogen leak detectors in a combustible or explosive atmosphere. The heated anode detector operates at a temperature of about 900 °C (1650 °F) and could ignite flammable gas mixtures.

An operator performing a halogen detector probe test in a confined area should refrain from smoking. Tobacco smoke is rich in alkali ash that can contaminate the instrument sensing element and cause repeated erratic signals on the leakage rate meter. If a halogen detector probe test is conducted in an area such as a shop where the air is heavy with alkali rich welding and burning fumes, the surrounding air should be cleared by forced ventilation with outside air.

When venting a halogen mixture from a pressurized system, the exhaust line should terminate outdoors to prevent the vented gas from entering the test area. This precaution will keep the test area clear of background contamination that could delay further testing. Applicable regulations should be observed in releasing halogens to the environment.

Do not perform welding repairs in an atmosphere rich with refrigerants. In addition, the shop air should be monitored to be sure safety limits for the halogen being used are not exceeded. Refrigerants, in the presence of high temperatures may break down into hydrogen chloride, hydrogen fluoride, chlorine and phosgene gas (mustard gas), which are highly toxic compounds. It is necessary to purge background halogen vapors from the space around equipment before making repairs by welding.

Procedure for Using Halogen Detector Probe to Locate Leaks

During leak testing with the halogen detector probe, the operator should search for leaks by systematically probing with the gun or probe held at the distance from the surface specified in the test instructions. To maintain this distance more easily, the end of the gun or probe can be fitted with a piece of plastic tubing that is notched on the end and projects this distance beyond the tip of the gun.

About every 2 h while performing leak testing, the operator should check the sensitivity of the instrument with the standard leak with the manual button on the probe held down and the probe tip inserted firmly in the leak fitting.

When leakage is indicated, the operator should retest the same leak area to verify that the signal was caused by leakage and not by background. The operator should move the detector probe tube to approach the suspected leak area from two or more directions. The leakage will usually be located midway between the two or more points at which the signal is first indicated. Another useful technique consists of temporarily blocking the leakage by covering the suspected area with plastic and sealing it with pressure sensitive adhesive tape. Then slowly remove the tape while probing the new exposed area until a signal is received.

Procedure for Setting Standard Halogen Leak and Instrument Calibration for Testing

When testing with the halogen detector probe, the operator should establish by calibration that the leak detector is capable of detecting leakage of a certain size or larger. The operator is not usually concerned with actually measuring the leak size. The following guide is used for determining the sensitivity of the detector probe leak test in typical industrial leak testing.

1. The sensitivity (size of the smallest leaks that are to be detected) will be specified in the test instructions.
2. Finding the percent by volume halogen mixture for the test from Tables 4 to 6 or Fig. 16 or 17 and knowing the required test sensitivity, the operator can determine the leakage rate value at which to set the standard leak by using Table 7, which simply states that the test sensitivity is the same percentage of the leak standard as the halogen

TABLE 4. Halogen concentration, percent by volume (corresponding to Fig. 16 and Table 5).

Halogen Pressure (kPa gage)	Test Pressure (kPa gage)								
	10	50	100	150	200	250	300	350	400
	Halogen concentration (percent by volume)								
10	94.0	71.0	53.0	42.5	35.0	30.5	26.5	23.5	21.0
50	—	95.5	72.5	57.5	48.0	41.5	36.0	32.0	29.0
100	—	—	96.0	78.0	64.5	55.5	49.0	43.5	39.0
250	—	—	—	97.0	81.0	69.5	61.0	54.5	49.0
200	—	—	—	—	97.5	84.0	73.5	65.5	59.0
250	—	—	—	—	—	98.0	86.0	76.5	69.0
300	—	—	—	—	—	—	98.0	88.0	79.0
350	—	—	—	—	—	—	—	98.5	89.0
400	—	—	—	—	—	—	—	—	98.7

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TABLE 5. Halogen concentrations expressed as a percentage of volume (corresponding to Fig. 16 and Table 4) in English units for systems evacuated to 1 $lb_f \cdot in.^{-2}$ absolute before pressurizing with refrigerant. (1 $lb_f \cdot in.^{-2}$ = 6.9 kPa.)

Halogen Pressure ($lb_f \cdot in.^{-2}$ gage)	Test Pressure ($lb_f \cdot in.^{-2}$)														
	2	5	10	15	20	25	30	35	40	45	50	55	60	65	70
	Halogen concentration (percent by volume)														
2	94.0	80.0	64.0	53.3	45.7	40.0	35.5	32.0	29.0	26.6	24.6	22.8	21.3	20.0	18.8
5	—	95.0	76.0	63.3	54.3	47.5	42.2	38.0	34.5	31.6	29.2	27.1	25.3	23.7	22.3
10	—	—	96.0	80.0	68.5	60.0	53.3	48.0	43.6	40.0	36.9	34.2	32.0	30.0	28.2
15	—	—	—	96.6	82.8	72.5	64.4	58.0	52.7	48.3	44.6	41.4	38.6	36.2	34.1
20	—	—	—	—	97.1	85.0	75.5	68.0	61.8	56.6	52.3	48.6	45.3	42.5	40.0
25	—	—	—	—	—	97.5	86.6	78.0	70.9	65.0	60.0	55.7	52.0	48.7	45.8
30	—	—	—	—	—	—	97.8	88.0	80.0	73.3	67.6	62.8	58.6	55.0	51.7
35	—	—	—	—	—	—	—	98.0	89.0	81.6	75.3	70.0	65.3	61.2	57.6
40	—	—	—	—	—	—	—	—	98.2	90.0	83.0	77.0	72.0	67.5	63.5
45	—	—	—	—	—	—	—	—	—	98.3[a]	90.7	84.2	78.6	73.7	69.4
50	—	—	—	—	—	—	—	—	—	—	98.5	91.4	85.3	80.0	75.3
55	—	—	—	—	—	—	—	—	—	—	—	98.6	92.0	86.2	81.2
60	—	—	—	—	—	—	—	—	—	—	—	—	98.7	92.5	87.0
65	—	—	—	—	—	—	—	—	—	—	—	—	—	98.7	92.9
70	—	—	—	—	—	—	—	—	—	—	—	—	—	—	98.8

a. Example of how halogen concentration values were determined. A specification or procedure requires that a test system be evacuated to 1 $lb_f \cdot in.^{-2}$ absolute and then pressurized with refrigerant to test pressure of 45 $lb_f \cdot in.^{-2}$ gage. Assuming barometric pressure of 100 kPa, the percent by volume halogen concentration would be 100[(45+14)/(45+15)] = 98.3 percent.

TABLE 6. Halogen concentration expressed as a percentage of volume (corresponding to Fig. 17) in English units,[a] for systems pressurized from atmospheric pressure with refrigerant.

Halogen Pressure ($lb_f \cdot in.^{-2}$ gage)	Test Pressure ($lb_f \cdot in.^{-2}$ gage)														
	2	5	10	15	20	25	30	35	40	45	50	55	60	65	70
	Halogen concentration (percent by volume)														
2	12.0	12.0	8.0	6.7	5.7	5.0	4.5	4.0	3.9	3.3	3.1	2.9	2.7	2.5	2.3
5	—	25.0	20.0	16.7	14.3	12.5	11.1	10.0	9.1	8.3	7.7	7.1	6.7	6.3	5.9
10	—	—	40.0	33.3	28.6	25.0	22.2	20.0	18.2	16.7	15.4	14.3	13.3[a]	12.5	11.8
25	—	—	—	50.0	42.9	37.5	33.3	30.0	27.3	25.0	23.1	21.4	20.0	18.8	17.6
20	—	—	—	—	57.1	50.0	44.4	40.0	36.4	33.3	30.8	28.6	26.7	25.0	23.5
25	—	—	—	—	—	62.5	55.5	50.0	45.5	41.7	38.5	35.7	33.3	31.3	29.4
30	—	—	—	—	—	—	66.7	60.0	54.5	50.0	46.2	42.9	40.0	37.5	35.3
35	—	—	—	—	—	—	—	70.0	63.6	58.3	53.8	50.0	46.6	43.8	41.2
40	—	—	—	—	—	—	—	—	72.7	66.6	61.5	57.1	53.3	50.0	47.1
45	—	—	—	—	—	—	—	—	—	75.0	69.2	64.3	60.0	56.3	53.0
50	—	—	—	—	—	—	—	—	—	—	77.0	71.4	66.7	62.5	58.8
55	—	—	—	—	—	—	—	—	—	—	—	78.6	73.3	68.8	64.7
60	—	—	—	—	—	—	—	—	—	—	—	—	80.0	75.0	70.6
65	—	—	—	—	—	—	—	—	—	—	—	—	—	81.3	76.5
70	—	—	—	—	—	—	—	—	—	—	—	—	—	—	82.4

a. 1.0 $lb_f \cdot in.^{-2}$ = 6.9 kPa.
b. Example of how halogen concentration values were determined. A specification or procedure requires that a test system be pressurized with refrigerant from atmospheric pressure to a test pressure of 70 kPa (10 $lb_f \cdot in.^{-2}$) gage and then further pressurized with refrigerant to test pressure of 410 kPa (60 $lb_f \cdot in.^{-2}$ gage). Assuming barometric pressure of 14.7 $lb_f \cdot in.^{-2}$ absolute, the percent halogen concentration by volume would be 100[10/(60+15)] = 13.3 percent.

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concentration.[3] For example, if the required test sensitivity were 1×10^{-6} Pa·m^3·s^{-1} (1×10^{-5} std cm^3·s^{-1}) and the halogen mixture for the test were 20 percent by volume, from Table 7 the standard leak setting would be 2×10^{-7} Pa·m^3·s^{-1} (2×10^{-6} std cm^3·s^{-1}).

3. Some typical standard leakage rate settings with the respective values for these leakage rate sizes are shown in Fig. 14 for the standard halogen leak. When the leak test is to conform with the allowable leakage rate and technique for a halogen detector probe test specified by Article 10 of the *ASME Boiler and Pressure Vessel Code*.[3] The 1×10^{-5} Pa·m^3·s^{-1} (1×10^{-4} std cm^3·s^{-1}) required test sensitivity column identified in Table 7 should be used.

Sensitivity Loss of Halogen Vapor Detector with Background Contamination

The inherent sensitivity of the halogen detector cannot be reached in practice if there is background contamination. A large leak near a small leak may completely obscure the signal from the small leak. In many factory test areas, a background level of halogen gas will build up because of leaks in the units being tested, leaks in the refrigerant supply tank, dumping of gas and other sources that may allow halogens to enter the area.

In a testing setup, the chief precaution is that of making certain that the testing is carried on in an environment sufficiently free of halogen vapors. In many instances, an exploration of the available floor space will indicate a location free of these vapors. If such a location is not available, it may be necessary to partition off a testing area and provide proper ventilation to bring in outside air that is halogen free. When speaking of halogen free air, it should be understood that the presence of halogen vapors in proportions of 10 μL·L^{-1} may be sufficient to contaminate the air so as to cause a loss of sensitivity in the testing equipment.

When venting a halogen mixture from a pressurized system, the exhaust line must terminate outdoors to prevent the vented gas from reentering the test area. This precaution will keep the test area clear of background contamination that could delay further leak testing. Venting must also conform to applicable environmental control regulations.

TABLE 7. Selection of proper halogen leakage rate (size) for required leak test sensitivity, with various concentrations of refrigerant in pressurizing tracer gas.

Halogen Mixture Percent by Volume	Required Test Sensitivity (Pa·m^3·s^{-1})[a]				
	1×10^{-3}	1×10^{-4}	1×10^{-5} [b]	1×10^{-6}	1×10^{-7}
	Halogen Standard Leak Size (Pa·m^3·s^{-1})[a]				
1	1.0×10^{-5}	1.0×10^{-6}	1.0×10^{-7}	1.0×10^{-8}	—
2	2.0×10^{-5}	2.0×10^{-6}	2.0×10^{-7}	2.0×10^{-8}	—
5	5.0×10^{-5}	5.0×10^{-6}	5.0×10^{-7}	5.0×10^{-8}	—
10	1.0×10^{-4}	1.0×10^{-5}	1.0×10^{-6}	1.0×10^{-7}	1.0×10^{-8}
15	1.5×10^{-4}	1.5×10^{-5}	1.5×10^{-6}	1.5×10^{-7}	1.5×10^{-8}
20	2.0×10^{-4}	2.0×10^{-5}	2.0×10^{-6}	2.0×10^{-7}	2.0×10^{-8}
25	2.5×10^{-4}	2.5×10^{-5}	2.5×10^{-6}	2.5×10^{-7}	2.5×10^{-8}
30	3.0×10^{-4}	3.0×10^{-5}	3.0×10^{-6}	3.0×10^{-7}	3.0×10^{-8}
35	3.5×10^{-4}	3.5×10^{-5}	3.5×10^{-6}	3.5×10^{-7}	3.5×10^{-8}
40	4.0×10^{-4}	4.0×10^{-5}	4.0×10^{-6}	4.0×10^{-7}	4.0×10^{-8}
45	4.5×10^{-4}	4.5×10^{-5}	4.5×10^{-6}	4.5×10^{-7}	4.5×10^{-8}
50	5.0×10^{-4}	5.0×10^{-5}	5.0×10^{-6}	5.0×10^{-7}	5.0×10^{-8}
55	5.5×10^{-4}	5.5×10^{-5}	5.5×10^{-6}	5.5×10^{-7}	5.5×10^{-8}
60	6.0×10^{-4}	6.0×10^{-5}	6.0×10^{-6}	6.0×10^{-7}	6.0×10^{-8}
65	6.5×10^{-4}	6.5×10^{-5}	6.5×10^{-6}	6.5×10^{-7}	6.5×10^{-8}
70	7.0×10^{-4}	7.0×10^{-5}	7.0×10^{-6}	7.0×10^{-7}	7.0×10^{-8}
75	7.5×10^{-4}	7.5×10^{-5}	7.5×10^{-6}	7.5×10^{-7}	7.5×10^{-8}
80	8.0×10^{-4}	8.0×10^{-5}	8.0×10^{-6}	8.0×10^{-7}	8.0×10^{-8}
85	8.5×10^{-4}	8.5×10^{-5}	8.5×10^{-6}	8.5×10^{-7}	8.5×10^{-8}
90	9.0×10^{-4}	9.0×10^{-5}	9.0×10^{-6}	9.0×10^{-7}	9.0×10^{-8}
95	9.5×10^{-4}	9.5×10^{-5}	9.5×10^{-6}	9.5×10^{-7}	9.5×10^{-8}
100	1.0×10^{-3}	1.0×10^{-4}	1.0×10^{-5}	1.0×10^{-6}	1.0×10^{-7}

a. 10 Pa·m^3·s^{-1} = 1 std cm^3·s^{-1}.
b. Use this column when *ASME Boiler and Pressure Vessel Code*[3] allowable leakage requirements are specified.

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The automatic balance feature of the control unit of heated anode halogen detectors will balance out a certain amount of halogen background, provided the concentration is constant or changing slowly. However, if this background level builds up to a point where normal air currents present in the room cause sudden changes in background concentration, sudden leak signals may result even when the detector has not encountered a leak.

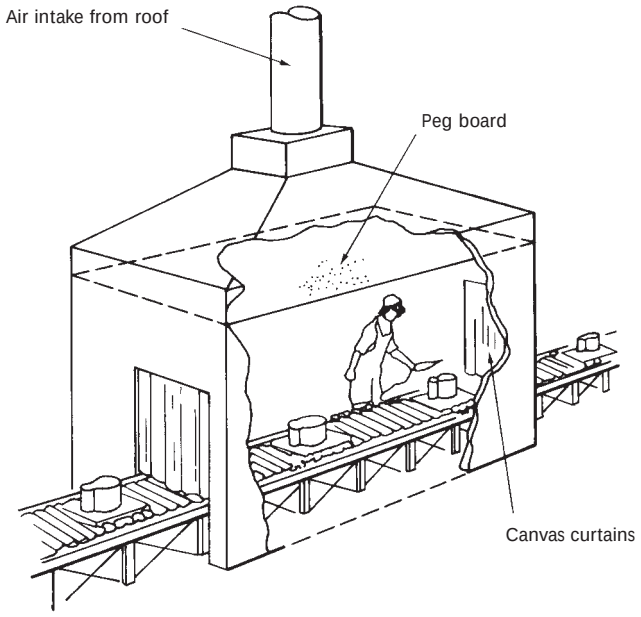
Methods for Combating Background Halogen Vapor Contamination

There are three methods of combating background contamination with halogen vapors.

1. Eliminate sources of background.
2. Provide a controlled environment of fresh air in the testing area.
3. Use a proportioning detector probe.

In some instances, elimination of the halogen background may be an inexpensive and simple step to control the testing atmosphere. For example, it should be possible to control indiscriminate dumping of refrigerant charges, leaky lines, degreasers using halogen solvents, paint fumes, etc. The second approach of providing a controlled fresh air environment can be a very elaborate one or a very simple and inexpensive one, depending on the level of background contamination.

FIGURE 18. Controlled atmosphere leak testing booth.



In other areas where the background level is quite low, it may not be necessary to use any special ventilating techniques, other than the normal ventilation required for good health. A simple ventilating device that can be used, if it is impractical to build a special room or booth, is a portable 0.4 to 0.5 m (16 to 20 in.) diameter fan placed in a window or doorway. Arranged in this manner, the fan will usually clear out the halogen background to the point where leak testing can be accomplished satisfactorily.

Design of Simple Leak Testing Booth

Manufacturers of refrigerators and air conditioners have found it desirable to construct a small room or booth for leak testing because the background level of tracer gas is usually very high in their testing areas because of the many sources of halogen gas. This room or booth is fed fresh air from outdoors at a very low velocity to prevent excessive drafts and eddy currents of air within the room. The booth, in effect, isolates the leak testing area and helps to keep background contamination from interfering with the leak testing.

Basically, the booth is a four-sided structure with a roof. Fresh air is introduced through the roof to provide an air change once or twice a minute. A test booth is illustrated in Fig. 18.[2] Some important things to keep in mind when constructing the leak testing booth are the following.

1. Fresh air should be supplied to the booth from the outside. In some cases, this air should go through an activated charcoal filter bed. This will remove any halogen gas present in the air.
2. The fresh air from the blower should be diffused before entering the booth to prevent drafts or eddy currents of air within the booth. A false ceiling made of perforated board is an ideal way to provide an even flow of air in the booth. When the booth is kept under this even positive air pressure, contaminated air will not come in from the shop or factory.
3. If a conveyor goes through the booth, canvas or rubber curtains help prevent contaminated air from coming into the booth.
4. An air conditioner in the booth will be helpful in providing comfort for the booth personnel and to help clear the air. It will, of course, also help remove humidity. Care should be taken to baffle the air conditioner so that the cool air is distributed as evenly as possible in the booth.

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5. Construction techniques will vary from factory to factory. Some fabricate the booth pit of wooden studs. Sheathing can be a type of hardboard or sheet rock. Others use the movable metal partitions commonly found in offices.

The roof of the booth quite often is fabricated of metal. The air intake pipe is standard air conditioning air handling pipe (containing an air heater if used in cold weather) and runs through the roof or through the side of the building. A filter chamber should be large enough to allow a liberal amount of activated charcoal to be used. The larger this chamber, the less often the charcoal will have to be changed. Thought should be given to the ease of changing charcoal. One way to contain the charcoal is to use a drawer; the bottom of the drawer can then be a mesh screen to allow air flow through the charcoal.

Proportioning Probe Halogen Vapor Detector

The third method used to overcome leak test problems commonly associated with area contamination is to use the proportioning probe. During tests, air is drawn from the atmosphere into the probe. Incoming air is mixed with pure air to effectively reduce contamination.

In a heavily contaminated leak test area, the proportioning valve is partially closed to restrict the sample intake. At the same time, pure air from the probe's fresh air filter is pumped to the probe and is mixed with the incoming sample. The mixture travels into the control unit and passes over the sensitive element. If the probe has passed near a leak, a signal proportional to the size of the leak is indicated.

With a built-in pure air filter, the probe requires no costly construction of specially ventilated test facilities to combat area contamination. However, to avoid halogen saturation and subsequent replacement of the probe's expendable air filter, a supply of fresh air can be ducted directly to the filter. Figure 19 illustrates a simple, inexpensive method.[2] A small pipe can be led from the test area to the outdoors or any other area that is relatively free of halogen contaminants. A small, low volume centrifugal blower is then installed to force a supply of fresh air to the test end of the pipe. One end of an extension tube is connected to the filter intake of the heated anode leak detector and the other end is allowed to rest in the pipe exhaust. Pipe size, blower capacity, exhaust hole size and exhaust hole quantity can vary greatly. The only criterion is that a positive exhaust pressure be maintained in the pipe to prevent halogen contamination from entering the extension tube.

Alternative Sources of Uncontaminated Air for Leak Testing

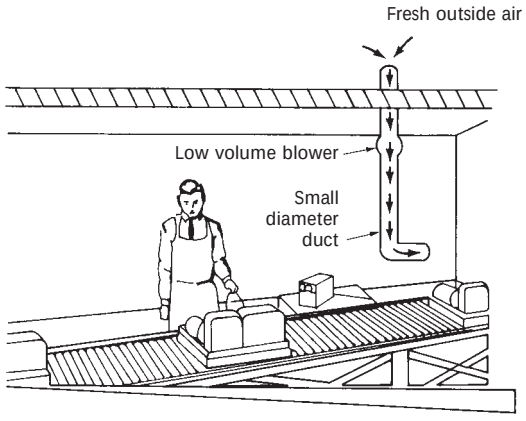
If no satisfactory supply of uncontaminated air is available from the outdoors or the plant area, one of the following alternatives should be used.

1. Shop air can be metered to bleed off into a small container at a rate that will maintain an uncontaminated atmosphere around the filter tube extension resting in the container. This method is useful only when the intake to the shop compressor is in an area free of halogen contamination.
2. A high pressure tank of air or nitrogen can be metered to bleed off into a small container at a rate that will maintain an uncontaminated atmosphere around the container. This method has the advantage of portability when the probe has to be used for leak testing in more than one area.
3. A large, separately mounted air filter can be used to replace the small air filter supplied with the halogen vapor detector. This could consist of a container, airtight except for intake and exhaust, filled with commercially available activated charcoal.

Measuring Halogen Contamination in Testing Area

It is possible to measure the extent of atmospheric contamination if a leak standard, a halogen detector and a pure air supply are available. In a contaminated

FIGURE 19. Fresh air ducting for a proportional halogen vapor probe.



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area, with the leak detector operating at a known air flow, the leak detector should be allow to intake pure air for about 60 s; then the detector should sample the contaminated area. If the leak detector gives a signal, the area is contaminated. The magnitude of the leak detector setting is noted but the sensitivity of the leak detector should not be adjusted.

The leak detector and leak standard should then be moved to an area where there is no atmospheric contamination. The leak standard must be adjusted so that when the leak detector samples the leak standard port, the leak signal indication is the same as when the leak detector sampled air in the contaminated area. The indicated leakage rate in $Pa \cdot m^3 \cdot s^{-1}$ (on the leak standard) multiplied by 10.0 and divided by the air flow of the leak detector in $m^3 \cdot s^{-1}$ equals the contamination in $\mu L \cdot L^{-1}$ (parts of halogen per million parts of air). For older units use std $cm^3 \cdot s^{-1}$ (on the leak standard) multiplied by 10^6 and divided by the air flow of the leak detector in $cm^3 \cdot s^{-1}$.

Safety Considerations in Leak Testing with Heated Anode Halogen Detector

In the heated anode halogen detector instruments, the leak detection element operating temperature is about 900 °C (1650 °F) and voltages of 300 V are present in the amplifier circuit of some models. The following safety precautions must be observed:

1. Never enter an area where there is an explosive vapor with the halogen leak detector energized. If there is any question, first test the area with an explosion meter.
2. Never test in enclosed spaces such as bearing housings, oil tanks or piping without first testing with an explosion meter.
3. Because voltages as high as 300 V are present in some models, the instrument case should be kept at ground potential by using a three-pronged grounded alternating current power plug and receptacle.

Techniques of Leak Testing with Heated Anode Halogen Detector

The procedure for leak testing with the halogen detector is applicable for two types of operation: testing in atmosphere and testing in vacuum. In atmosphere, the equipment is used for static leakage measurement and detector probe leak location. In vacuum, the equipment is used for tracer probe leak location and for dynamic leakage measurement.

The use of the leak detector is not limited to apparatus into which a detector gas can be injected. In liquid filled sealed units, the same leak testing techniques can be used if a halogen tracer compound can be added to the liquid at the time of filling without detriment to the liquid. The halogen tracer compound used must be one that will quickly vaporize. In some types of electrical and thermal apparatus such as liquid filled indicating thermometers and liquid cooled equipment, the liquid used may itself contain a halogen compound and therefore will not require any additives.

The one requirement is that the halogen compounds used have an appreciable vapor pressure. For example, nonflammable insulating oil cannot be detected with a halogen leak detector at room temperature. However, if the compound is heated, the vapor pressure is increased to a point where detection is possible.

Procedure for Pressurizing with Halogen Tracer Gases

To best use the capabilities of the halogen leak detector, the following procedure is recommended.

1. Detect and repair larger leaks first. This should be done before the system is fully pressurized to save time and prevent excessive contamination of the ambient atmosphere.
2. Charge the system with tracer gas after larger leaks have been corrected. When a closed system is to be pressurized with a mixture of a refrigerant tracer gas and air, always pressurize with the refrigerant first and then pressure up with air to attain the required halogen pressure and test pressure. In some cases, an inert gas such as dry nitrogen may be required in place of air as the diluent. Never use oxygen or combustible gases as the pressurizing gas for leak testing with halogen leak detectors. When use is made of premixed tracer gas, its concentration should be checked by appropriate metering to avoid overrich and underrich mixtures.
3. During charging with tracer, dead end ducts should either be opened to allow trapped air to escape or time should be allowed for diffusion to take place. (This time may be days for small tubes of substantial length.) Alternatively, the system should be evacuated before charging with tracer gas.

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Leak Location with Heated Anode Halogen Detector Probe

For leak location using a detector probe, the system under test is pressurized with tracer gas and probed with the detector. Certain precautions are necessary in this probe exploration. Too rapid a search may miss the very small leak. If this risk is to be avoided, the speed at which the probe is moved must be in proportion to the minimum leak tolerance. In testing equipment for a leak specification of the order of 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}), the rate of probe travel can be 20 to 50 mm·s^{-1} (1 to 2 in.·s^{-1}). The probe speed should be reduced to the range of 10 mm·s^{-1} (0.5 in.·s^{-1}) for smaller leaks. The degree of mechanization used in a testing setup depends on the production rate, the uniformity of test pieces and the total time available for hand work by the operator.

Control of Halogen Background Level in Test Areas

If the tracer gas escapes into the room after the test has been completed and the test object is opened, sufficient time must be allowed to permit the ambient environment to again become free of halogen vapors. If the waiting period cannot be tolerated, there are two alternatives that may be used: the test object can be removed to an outside area before permitting the gas to escape, or a vacuum system can be used to remove the gas before opening the test object.

The use of the vacuum system has been found to be more satisfactory. Although some gas usually remains in the test object after evacuation and therefore tends to contaminate the air, the time lost in regaining clear air is relatively small. If large volumes are to be tested, it is possible to recompress the tracer gas for reuse. This also avoids possible violation of air pollution laws.

It is not possible to depend on natural dissipation of escaping gas in high speed production leak testing. It therefore becomes necessary to remove gas in the shortest possible time. The design must include adequate vacuum lines to remove the gas from the test piece after testing; proper forced ventilation must be used to clear the surrounding air. A hood has been found to be most suitable for this purpose. When sufficient incoming air flow is used to clear away leaking gas, steps must be taken to properly direct the air so that it does not remove the gas at the point of the leak before the detector has had time to pick it up and indicate the leak.

Leakage Measurement with Halogen Refrigerant Filled Products in Cartons

Leakage measurement using the detector operated in air is a static accumulation procedure. Because the system will be leaking to atmosphere, the collecting container does not have to be completely tight. For example, some manufacturers package their refrigerators and air conditioners in cartons and make a final leak check on their product in the warehouse before shipment. This is accomplished by making a hole near the bottom of the carton with a punch the size of a pencil and then inserting the probe of the detector into this hole. The leak detector will sample air from the carton. A leak signal will occur if the unit in the carton is leaking.

Even though the overall leakage rate of the unit in the carton may be very low, a substantial leak signal will be obtained when performing this test if the unit has been in the carton any length of time. The approximate total leakage rate of products in cartons can be determined as follows.

1. Note the signal from the leak detector when detector probing the carton.
2. Adjust the leak standard so that when probing the standard the signal is the same as for the carton.
3. Compute the total volume leakage rate Q_{total} in std cm^3·s^{-1} for halogen filled systems in cartons by Eq. 1:

$$\textbf{(1)} \quad Q_{total} = \frac{RV}{4t}$$

where V is volume of the carton minus volume of the unit in the carton (cubic centimeter); R is indicated leakage rate (std cm^3·s^{-1}) with the detector probe air pump set for 4 cm^3·s^{-1}; and t is time (second) the unit has been sealed in the carton.

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PART 3. Recommended Techniques for Pressure Leak Testing with Halogen Detector Probe[4]

Application of Halogen Leak Tests

This discussion covers procedures for testing and locating the sources of halogen tracer gas leaking at the rate of 5×10^{-11} Pa·m^3·s^{-1} (5×10^{-10} std cm^3·s^{-1}) or greater. The test may be conducted on any device or component across which a pressure differential of halogen tracer gas may be created and on which the effluent side of the area to be leak tested is accessible for detector probing with the halogen leak detector. These techniques require halogen leak equipment with a full scale readout of at least 3×10^{-10} Pa·m^3·s^{-1} (3×10^{-9} std cm^3·s^{-1}) on the most sensitive range, with a zero drift and sensitivity drift not exceeding ±15 percent of full scale during 60 s in this range and of ±5 percent or less in other ranges.

Summary of Halogen Leak Testing Methods

Five methods of halogen leak testing are described in Standard E 427 of the American Society for Testing and Materials (ASTM): Method A, direct probing with no significant halogen contamination in the atmosphere; Method B, direct probing with significant halogen contamination in the atmosphere; Method C, shroud test; Method D, air curtain shroud test; and Method E, accumulation test.[4]

Methods C, D and E are well adapted for automation of valving and material handling.

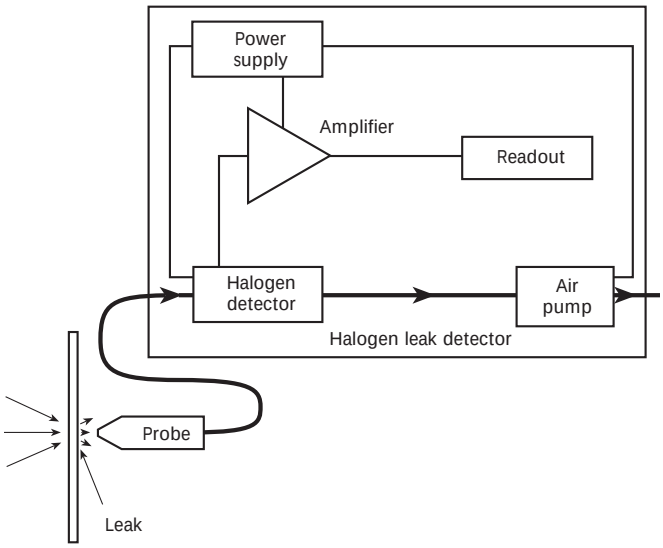
Method A — Direct Detector Probing with Standard Halogen Detector

The direct detector probe technique sketched in Fig. 20 is the simplest test. It requires only that (1) a halogen tracer gas pressure differential be created across the pressure boundary area to be tested and (2) the atmospheric side of the area be searched with the detector probe leak detector. This technique enables detection of leakage and the location of its source or sources when used in a test area free from significant halogen contamination in the atmosphere. Experience has shown that leak detection down to 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}) in factory environments will usually be satisfactory if reasonable precautions are taken against releasing halogens in the building. If a test booth is constructed so as to be purged with clean outdoor air, this level may be reduced to 10^{-8} Pa·m^3·s^{-1} (10^{-7} std cm^3·s^{-1}). Testing for leakage rates as low as 10^{-10} Pa·m^3·s^{-1} (10^{-9} std cm^3·s^{-1}) will require additional halogen removal, which can be accomplished by passing the test booth purge air through a bed of activated charcoal.

Method B — Direct Detector Probing with Proportioning Probe Halogen Detector

The test arrangement sketched in Fig. 21 is essentially the same as Method A, except that the amount of air drawn by the detector probe from the test area is reduced and the required sample flow is made up with pure (that is, zero halogen) air. This reduced sample intake has the disadvantage of reducing the vacuum cleaner effect of the larger flow, requiring closer and more careful detector probing. However, the tolerance to background

FIGURE 20. Standard detector probe and halogen leak detector used with Method A halogen leak testing.[4]



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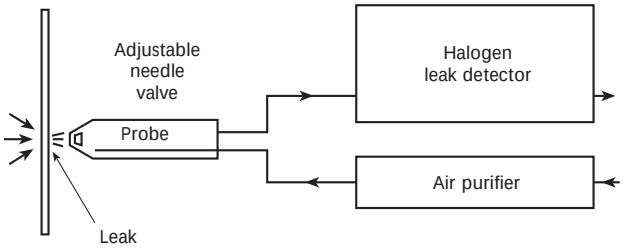
atmospheric halogen can be increased up to 100 times. Also, large leaks beyond the range of Method A can be accurately located (but not measured) by Method B.

Method C — Shroud Test

The test arrangement sketched in Figs. 22 and 23 is suited for leak testing items that have a maximum cross section dimension of 50 mm (2.0 in.) but may be as long as 10 m (30 ft). In this method, air, either atmospheric or purified, is passed over the halogen pressurized part inside a close fitting container. The discharge air from the container is sampled by the halogen detector and any additional halogen content indicated. The shroud principle may be applied in a manner as simple as Fig. 23, wherein a piece of tape is applied around a flanged joint to be tested or as complete as in Fig. 22. The test arrangement of Fig. 22 provides isolation of the detector from atmospheric halogens, pure air reference supply and a convenient calibration means. This enables detection of leaks as small as 10^{-10} Pa·m^3·s^{-1} (10^{-9} std cm^3·s^{-1}).

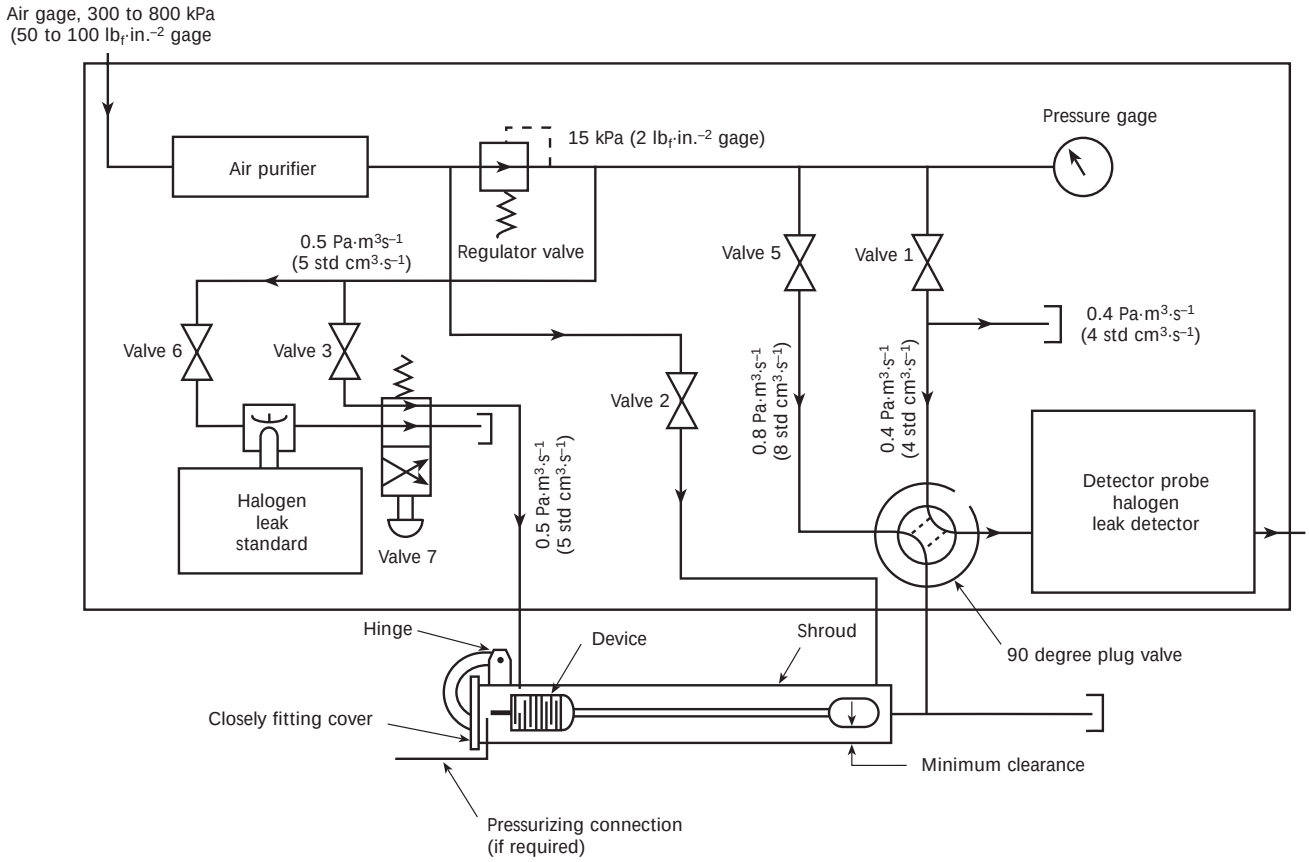
FIGURE 21. Proportioning detector probe arrangement used with Method B halogen leak detector.[4]



Method D — Air Curtain Shroud Test

The test arrangement sketched in Fig. 24 is useful for high production testing of small items such as electronic components that have been previously subjected to bombing (pressurizing of a halogen gas above atmospheric pressure) or for testing the sealed end of a fill tube, and the like. In this technique, the upper end of the shroud is always open and the halogen detector probe leak detector always draws a sample from the lower end. Atmospheric halogens are prevented from entering by a laminar flow pure air curtain. When any leaking object is

FIGURE 22. Purge sample, detect, and calibrate (PSDC) unit used in Method C shroud leak test with halogen tracer gas.[4]



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inserted below the flow division level, the leakage is detected by the detector probe. This technique is useful for detecting leaks down to 10^{-10} Pa·m^3·s^{-1} (10^{-9} std cm^3·s^{-1}).

Method E — Accumulation Leak Testing with Halogen Detector Probe

The test arrangement sketched in Fig. 25 is similar to Method C, except it provides for testing parts up to several cubic meter in volume. This is accomplished by letting leakage accumulate in the chamber for a fixed period while keeping it well mixed with a fan. The internal atmosphere is then tested for an increase in halogen content. The practical sensitivity attainable with this method depends primarily on (1) the volume between the shroud and the object and (2) the amount of halogen outgassing by the object. Thus, a part containing rubber, plastics, blind cavities or threads (which trap halogen vapors and outgas later) cannot be tested with the sensitivity obtainable with a smooth metallic part.

FIGURE 23. Simple shroud halogen leak testing arrangement.[4]

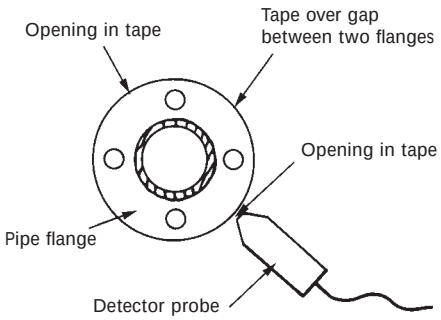
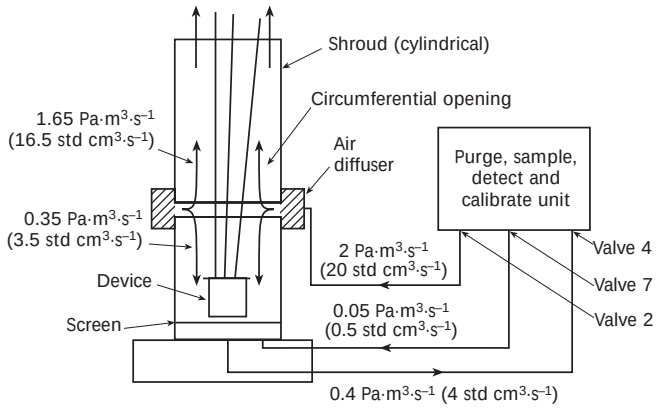


FIGURE 24. Arrangement for Method D, air curtain shroud halogen leak testing with a purge sample, detect and calibrate (PSDC) unit.[4]



Rate of Halogen Tracer Gas Partial Pressure Increase during Accumulation Test

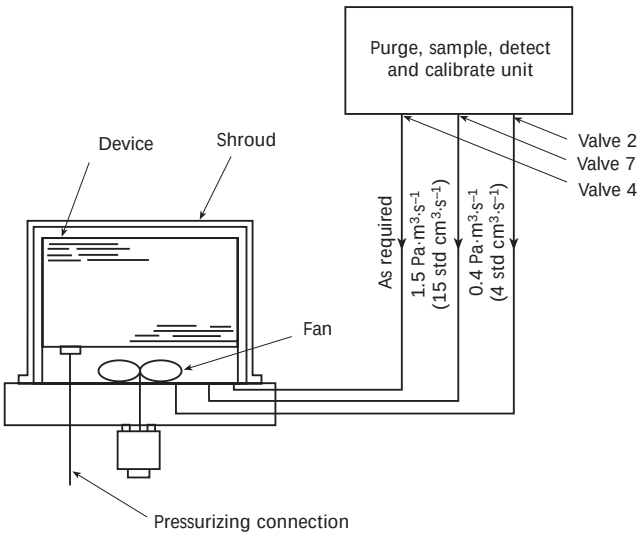
The halogen leak detector measures the partial pressure of halogen tracer gas in the accumulation volume. When the halogen leak detector is zeroed at the start of an accumulation test period and the change in halogen concentration is indicated at the end of this period, the sensitivity of the accumulation test and net volume of the system are related by Eq. 2:

$$\textbf{(2)} \qquad A_s = \frac{Q - F}{V}$$

where A_s is the rate of halogen tracer partial pressure increase in the accumulation volume (Pa·s^{-1}); Q is the rate of leakage into the volume (m^3·s^{-1}); F is the flow rate in the detector probe (Pa·m^3·s^{-1}); and V is the net volume of the accumulation system (cubic meter).

For practical operating considerations, the minimum value of the rate of halogen pressure accumulation A_s that should be used is about 2×10^{-12} Pa·s^{-1} (2×10^{-11} std cm^3·s^{-1}). This will give a leak detector readout of $50 \times 2 \times 10^{-12}$ or 10^{-10} Pa·m^3·s^{-1} ($50 \times 2 \times 10^{-11}$ or 10^{-9} std cm^3·s^{-1}) after 50 s accumulation period. Thus, based on a probe flow rate F = 0.4 Pa·m^3·s^{-1} (4 std cm^3·s^{-1}), a 5×10^{-11} Pa·m^3·s^{-1} (5×10^{-10} std cm^3·s^{-1}) leak may be detected in a system of 100 cm^3 (6 in.^{3}) net volume, or a 5×10^{-6} Pa·m^3·s^{-1}

FIGURE 25. Method E halogen accumulation leak testing arrangement with purge sample, detect and calibrate (PSDC) unit.[4]



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(5×10^{-5} std $cm^3 \cdot s^{-1}$) leak in a 10 m^3 (350 ft^3) system. Where variables of time, volume and leakage rate permit, values of readout should be set in the 10^{-8} to 10^{-9} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-7} to 10^{-8} std $cm^3 \cdot s^{-1}$) range for less critical operations.

Interference by Atmospheric Halogen Contamination

When Method A or B direct probing is used to locate leaks, the halogen leak detector probe is drawing in air from the atmosphere. If the atmosphere is contaminated with halogen to a degree that produces a noticeable indication on the detector, the detection of halogen from leaks becomes much more difficult. Significant atmospheric contamination with halogen is defined as the level where the detector response, when the probe is moved from zero halogen air to test area atmosphere, exceeds that expected from the smallest leak to be detected. For reliable testing, atmospheric halogen concentration must be kept well below this level.

Halogens Outgassed from Absorbent Materials

When leak testing is done in enclosures that prevent atmospheric contamination from interfering with the test (Methods A, B and C), halogen absorbed in various nonmetallic materials (such as rubber or plastics) may be released in the enclosure. If the amount of halogen compounds released by outgassing starts to approach the amount of input from the leak in the same period of time, it becomes more difficult to conduct a reliable leak test. The amount of such halogen absorbing materials in the enclosure, or their exposure to halogen, must then be reduced to obtain a meaningful leak test.

Pressurizing with Test Gas

To evaluate leakage accurately, the test gas in all parts of the device must contain substantially the same amount of tracer gas. When the device contains air before the introduction of test gas, or when an inert gas and a tracer gas are added separately, this may not be true. Devices in which the effective diameter and length are not greatly different (such as tanks) may be tested satisfactorily by simply adding tracer gas. However, when long or restricted systems are to be tested, more uniform tracer distribution will be obtained by first evacuating to less than 1 kPa (10 torr), and then filling with the test gas. If it is not 100 percent tracer, the test gas must be premixed before it is added to a system.

Requirements for Halogen Leak Detector Apparatus

To perform leak tests as specified in ASTM E 427,[4] the leak detector should meet the following requirements.

1. An alkali ion diode sensor should be used.
2. The readout may be digital or analog.
3. The linear range is 3×10^{-7} to 3×10^{-10} $Pa \cdot m^3 \cdot s^{-1}$ (3×10^{-6} to 3×10^{-9} std $cm^3 \cdot s^{-1}$) full scale or arbitrary equitable scales.
4. The response time should be ≤ 3 s.
5. The stability of zero and sensitivity values should meet applicable leak testing specifications. Normally for refrigeration, a maximum variation of ±15 percent of full scale is allowable on the most sensitive range, while the detector probe is in pure air. The maximum allowable variation is ±5 percent of full scale on other ranges for a period of 60 s.
6. The range control should be adjustable.
7. An automatic zeroing option is desirable.

Requirements for Halogen Reference Leakage Standards

To perform leak tests as specified in ASTM E 427,[4] the reference leak standard should meet the following requirements.

1. Ranges are 10^{-6} to 10^{-10} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} to 10^{-9} std $cm^3 \cdot s^{-1}$) full scale.
2. Adjustable leak standards are convenient but not mandatory,
3. Accuracy should be ±25 percent of full scale value or better,
4. Temperature coefficient shall be stated by the manufacturer.
5. The halogen content of the specification leak should remain compatible with the expected level of atmospheric halogen and the test method as outlined earlier. Fixtures or other equipment specific to one test method are listed under that method later in this discussion.

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Requirements for Halogen Tracer Gases

To be satisfactory, the test gas should be nontoxic, nonflammable, inexpensive, not detrimental to common materials and have a response factor of one. Refrigerant-12 (dichlorodifluoromethane, CCl_2F_2) has this characteristic. Refrigerant-22 provides a pressure of 900 kPa gage (130 $lb_f \cdot in.^{-2}$ gage) at 21 °C (70 °F). If the test specification allows leakage of 1×10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-5} std $cm^3 \cdot s^{-1}$) or more or if large vessels are to be tested, consideration should be given to diluting the tracer gas with nonhalogen gas such as dry air or nitrogen. This will avoid operating in the nonlinear portion of the sensor output, or, in the case of large vessels, save tracer gas expense. When a vessel is not evacuated before adding test gas, the test gas is automatically diluted by 100 kPa (750 torr) of air at atmospheric pressure already contained in the vessel under test.

Producing Premixed Halogen Test Gas

If the volume of the device or the quantity to be tested is small, premixed gases can be conveniently obtained in cylinders. The user can also mix gases by batch in the same way. Continuous mixing using calibrated orifices is another simple and convenient method when the test pressure does not exceed 50 percent of the tracer gas pressure available. (Caution: The liquid tracer gas supply should not be heated above ambient temperature.) Another method is to pass the nonhalogen gas through the liquid tracer, which produces test gas containing the maximum amount of tracer gas.

Requirements for Halogen Free Gas Used in Pressurizing Test Volumes

Pure air, air from which halogens have been removed to a concentration of 1 $nL \cdot L^{-1}$ (or other suitable nonhalogen gas, such as nitrogen) should meet the following requirements: (1) less than 1 $nL \cdot L^{-1}$ of halogen; (2) less than 10 $\mu L \cdot L^{-1}$ of gases reactive with oxygen, such as petroleum base solvent vapors; (3) dew point 10 °C (50 °F) or more below ambient temperature; and (4) reasonable freedom from rust, dirt, oil etc. Air or gas of suitable purity may be produced by first passing it through a conventional filter drier (if necessary) and then through activated charcoal.

Calibration of Halogen Leak Detector

The leak detectors used in making halogen vapor leak tests are not calibrated in the sense that they are taken to the standards laboratory, calibrated and then returned to the job. Rather, the leak detector is used as a comparator between a leak standard (part of the instrumentation set to the specified leak size) and the unknown leak. However, the sensitivity of the leak detector is checked and adjusted on the job so that a leak of specified size will give a readily observable reading not off the meter scale. More specific details are given later under procedures for each method. To verify detection, reference to the leak standard should be made before and after a prolonged test. When rapid repetitive testing of many items is required, the leak standard should be referred to often enough to ensure that desired test sensitivity is maintained.

Test Specifications for Using Halogen Leak Detector

User should have a halogen leak testing specification that includes the following test information: (1) gas pressure on the high side of the device to be tested and on the low side if different from atmospheric; (2) test gas composition, if there is need to specify it; (3) maximum allowable leakage rate in $Pa \cdot m^3 \cdot s^{-1}$ (or std $cm^3 \cdot s^{-1}$); (4) whether the leakage rate is for each leak or for total leakage of the device; and (5) if a per leak specification, whether or not areas other than seams, joints and fittings need to be tested.

Leakage Rate Safety Factor for Halogen Leak Testing

Where feasible, the test operator should ascertain that a reasonable safety factor has been allowed between the actual operational requirements of the device and the maximum leakage rate specified for testing. Experience indicates that a safety factor of at least ten in leakage rate should be used when possible. For example, if a maximum total leakage rate for satisfactory operation of a device is 5×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-6} std $cm^3 \cdot s^{-1}$), then the test requirement should be 5×10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-7} std $cm^3 \cdot s^{-1}$) or less.

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Selecting Test Pressure for Halogen Leak Testing

It is desirable to pressurize a device to be leak tested at or above its operating pressure and with the pressure drop in the normal direction across the pressure boundary, where practical. Precautions should be taken so that the device will not fail during pressurization and so that the operator is protected from the consequences of a failure.

Disposition or Recovery of Halogen Tracer Gas

Test gas should not be dumped into the test area if further testing is planned. Halogen tracer gas should be recovered for reuse to avoid atmospheric contamination, both indoors and out.

Detrimental Effects of Refrigerant-12 and Refrigerant-22 Tracer Gases

The refrigerant-22 and refrigerant-134a tracer gases are inert and seldom cause any problem with most materials, particularly when used in gaseous form for leak testing and then removed. Refrigerant-12 may no longer be legally made in or imported to the United States. Test gas should not be left in the device unless it is dry and sealed, as most halogen in the presence of moisture accelerate corrosion over a period of time.

When there is a question as to the compatibility of the tracer with a particular material, authorities on corrosion and the specific materials should be consulted. This is particularly true when the material may be subject to chloride stress corrosion under conditions of use. Halogen contamination must not be permitted when the enclosure contains hot or sparking components, or when arc welding or similar high temperature operations may occur. The use of chlorides is generally banned from austenitic materials in nuclear applications.

Correlation of Test Gas Leakage Rates with Leakage Rates of Other Gases or Liquids at Different Pressures

Given the normal variation in leak geometry, accurate correlation of leakage rates with halogen vapors and with other fluids is impossible. However, if a safety factor of ten or more is allowed, adequate correlation for gas leakage within these limits can usually be obtained by assuming viscous flow and using the relation of **EQ**. 3:

$$\textbf{(3)} \quad Q_t = Q_0 \frac{n_1}{n_2} \frac{P_2^2 - P_1^2}{P_4^2 - P_3^2}$$

where Q_t is test leakage rate, $Pa \cdot m^3 \cdot s^{-1}$ (or std $cm^3 \cdot s^{-1}$); Q_o is operational leakage rate, $Pa \cdot m^3 \cdot s^{-1}$ (or std $cm^3 \cdot s^{-1}$); n_2 is viscosity of test gas ($m \cdot s^{-1}$); n_1 is viscosity of operational gas ($m \cdot s^{-1}$); P_2,P_1 are absolute pressures on high and low sides during leak testing (pascal); and P_4,P_3 are absolute pressures on high and low sides in operation (pascal).

Experience has shown that, at the same pressures, gas leaks smaller than 10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} std $cm^3 \cdot s^{-1}$) will not show visible leakage of a liquid such as water that evaporates fairly rapidly. For slowly evaporating liquids such as lubricating oil, the gas leak should be another order of magnitude smaller, namely 10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-6} std $cm^3 \cdot s^{-1}$). (Note that viscosity differences between gases is a relatively minor effect and can be ignored if desired.)

Method A — Direct Halogen Leak Testing in Atmosphere

Apparatus

Equipment and facilities required for Method A, direct halogen leak testing include the following: (1) test specification; (2) halogen leak detector of standard detector probe type; (3) halogen leak standard, upper 90 percent of scale to include halogen content of maximum leak allowable in accordance with the specification, with response factor correction; (4) test gas, at or above specification pressure; (5) pressure gages, valves and piping for introducing test gas and, if required, vacuum pump for evacuating device; (6) pure air supply, if not part of halogen leak detector; and (7) test booth or other atmospheric contamination control, if shown to be necessary.

Procedure

Procedural steps in direct leak detector probe tests by Method A include the following.

1. Set the halogen reference standard at the maximum halogen content of the specification leak.

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2. Start the pure air supply and adjust its flow in excess of that of the leak detector probe. Couple the detector probe loosely to the supply so that air is not forced into the detector.
3. Start the detector, warm it up and adjust it in accordance with the manufacturer's instructions for detection of leaks of size cited in Step 1 above, using the manual zero mode.
4. Remove the detector probe from the pure air supply to the test area. Note the new reading and also minimum and maximum readings for a period of 1 min.
5. Rezero the instrument, place the detector probe at the port of the leak standard and note the reading. (If necessary to obtain a reasonable instrument deflection in the last two steps, return the detector probe to the pure air supply, adjust the range control and rezero if necessary.)
6. If the instrument reading in the test area atmosphere is larger than that attained on the leak standard, or if the 1 min variation is more than 30 percent of the leakage rate of the standard leak, take steps to reduce the atmospheric halogen content of the test area before proceeding with the leak test.
7. If the automatic zero mode is to be used, increase the sensitivity by a factor of three.
8. Evacuate (if required) and apply test gas to the device at the specified pressure.
9. For probe areas suspected of leaking, hold the probe on or not more than 5 mm (0.2 in.) from the surface of the device and move it not faster than 30 mm·s^{-1} (1.2 in.·s^{-1}). If leaks are located that cause a reject indication when the detector probe is held 5 mm (0.2 in.) from the apparent leak source, repair all such leaks before performing final acceptance test. If a marginal indication is observed while detecting automatic zero mode, reduce the sensitivity by a factor of three, switch to the manual zero mode and compare the leakage reading on the leak standard with that on the device.
10. Maintain an orderly procedure in detector probing the required areas, preferably identifying them as tested and plainly indicating points of leakage. Start the probing operation at the top of the test object, because halogen tracer gas is denser than air and tends to flow downward from leak exits.
11. At the completion of the test, evacuate or purge, or both, the test gas from the device.
12. Write the test report, or otherwise indicate test results as required (see Fig. 26).

Method B — Direct Halogen Leak Testing with Proportional Detector

Equipment used for Method B is the same as for Method A except that the halogen leak detector is of the proportioning detector probe type. The testing procedure is the same as for Method A except that use is made of a self-contained pure air supply activated by closing the detector probe tip valve tightly, which sends 100 percent pure air to the sensor. Some halogen detector models have a fixed proportioning detector probe instead of a valve.

In Procedure Step 2 of Method A, the detector probe valve is open wide (above two turns), which sends 100 percent atmospheric sample to the sensor. If the conditions of Step 6 are met, proceed with the test. If not, partially close the probe valve until they are met. However, do not reduce the valve opening below the point at which the response to the leak standard is reduced by 30 percent.

Method C — Shroud Test with Halogen Leak Detector

Apparatus

Equipment required for Method C shroud testing with halogen tracer gas includes (1) test specification; (2) test gas, at or above specification pressure, if the device is not already pressurized; and (3) purge sample, detect and calibrate (PSDC) unit (Fig. 22) plus shroud as in Fig. 24 to fit device.

FIGURE 26. Sample halogen leak test report form.[4]

HALOGEN LEAK TEST REPORT

Tester _____ Date of Test _____
Test witnessed by _____
Tested per ASTM std. _____ Method _____
Device tested _____ No. pieces _____
No. accepted _____ No. rejected _____
Max leakage accepted pieces ___ x 10 ___ std cm^3·s^{-1} or Pa·m^3·s^{-1}
Total _____ or per point _____ leakage
Device evacuated before charging _____
If evacuated, pressure _____ Pa or torr
Test pressure _____ lb$_f$·in.$^{-1}$ or kPa gage
Test gas: ____ % ___ Tracer: ___ % _____ gas
Atmospheric halogen equivalent ____ x 10 ____
Leak detector serial No. _____
Leak standard serial No. _____

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The upper 90 percent of the halogen leak standard scale shall include halogen leakage rate of maximum leak in accordance with the specification, with response factor correction.

Procedure

Steps involved in halogen leak testing by the shroud Method C, using the purge sample, detect and calibrate (PSDC) unit shown in Fig. 22, include the following.

1. Set the halogen leak standard at the maximum halogen content of the specification leak.
2. Adjust the air pressure, air flows (except purge valve 2) and valves 4 and 7 as indicated in the diagram for this method (Fig. 22). (The addition of flow meters and pressure gages at appropriate places in the circuit to facilitate these adjustments is recommended.)
3. Start the detector, warm it up and adjust it in accordance with the manufacturer's instructions for detection of leaks of specified size, using the manual zero mode.
4. Place a dummy device not containing halogen in the shroud and open valve 2 for as long as is required to purge the shroud of atmospheric halogens.
5. Turn valve 7 to calibrate and valve 4 to the sample position. Note detector indication and adjust the sensitivity if required. Return the valves to the original (standby) positions. Remove the dummy device from the shroud.
6. Insert the device to be tested inside the shroud and connect the evacuating line, pressurizing line or both, if device is not already pressurized with tracer gas.
7. Open valve 2 for as long as is required to purge the shroud of atmospheric halogens.
8. Turn valve 4 to the sample position.
9. If the device is already pressurized, read the leakage, if any, on the detector.
10. If the device is not pressurized, check the leak detector for indication of incomplete purging, then pressurize and read the leakage, if any. A leak detector indication greater than that obtained during calibration shows leakage greater than allowed by the specification.
11. If the device has been pressurized with halogen tracer for the leak test only, exhaust the test gas outside the test area, or recover for reuse.
12. Remove the device from the shroud and write the test report, or otherwise indicate the results of test as required.

Method D — Air Curtain Shroud Halogen Leak Testing

Steps involved in the air curtain shroud Method D of halogen leak testing with the purge sample, detect and calibrate (PSDC) unit shown in Fig. 22 include the following.

1. Set the halogen leak standard at the maximum halogen content of the specification leak.
2. Adjust the air pressure and flows as indicated in Fig. 24 for this method. Valve 2 is open and valve 4 is set at the sample position continuously.
3. Start, warm up and adjust the detector in accordance with the manufacturer's instructions for detection of leaks of size 1, using the manual zero mode.
4. Place a (dummy) device not containing halogen in the shroud. Turn valve 7 to the calibrate position, note detector indication, adjust the sensitivity if required and return the valve to the original (standby) position. Remove the dummy device.
5. Insert the device to be leak tested (and which has previously been bombed or which is pressurized with halogen tracer) in the shroud. (Any part of the device that is to be leak tested must be below the purge air opening.)
6. Read the leakage, if any. An indication on the leak detector greater than that obtained during calibration shows leakage greater than that allowed by the specification.
7. Remove the device and record the test results as desired.
8. If a large leak is detected, the clean up of the shroud and sensor can be expedited by turning valve 7 to standby for a few seconds. This will purge shroud, lines and sensors with pure air.

Method E — Halogen Accumulation Leak Testing

Apparatus

Equipment required for Method E halogen accumulation leak testing includes the following: (1) test specification; (2) test gas, at or above specification pressure, if the device is not already pressurized; and (3) purge sample, detect and calibrate unit (Fig. 22) plus shroud as in Fig. 25.

The upper 90 percent of halogen leak standard scale shall include halogen content of maximum leak per specification, with response factor correction.

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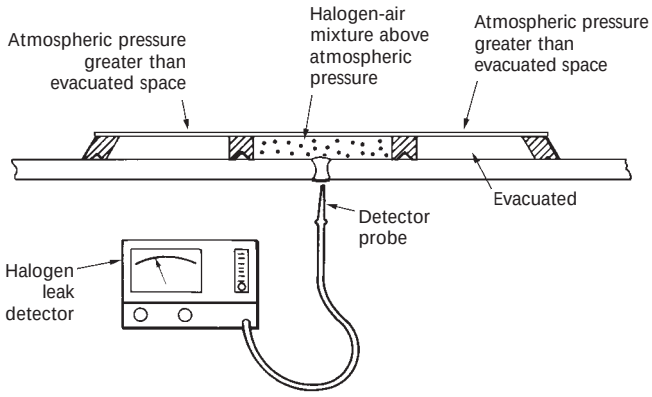
Procedure

Steps required in accumulation leak testing by Method E using the purge sample, detect and calibrate unit of Fig. 22 include the following.

1. Set the halogen leak standard at maximum halogen content of the specification leak.
2. Adjust the air pressure and flows (except purge valve 2) as indicated on the diagram of Fig. 25 for this method.
3. Start, warm up and adjust the detector in accordance with the manufacturer's instructions for detecting leaks of specified size using the manual zero mode.
4. Place a (dummy) device not containing halogen under the shroud.
5. Open valve 2 for as long as is required to purge the shroud of atmospheric halogen.
6. Turn valve 7 to the calibrate position, allow an appropriate accumulation period (with fan running), turn valve 4 to the sample position and note detector indication. If necessary, adjust the sensitivity and repeat Steps 5 and 6. Remove the dummy device.
7. Insert the device to be tested inside the shroud and connect the evacuate or pressure line, or both, if device is not already pressurized with tracer gas.
8. Open valve 2 for as long as is required to purge the shroud of atmospheric halogens.
9. Turn valve 4 to the sample position.
10. If the device is already pressurized, note whether the detector reading increases (in the allotted accumulation period) beyond that obtained during calibration. If so, reject the device.
11. If the device is not pressurized, check the leak detector for indication of incomplete purging, then pressurize and proceed as in Step 10.
12. Alternatively, sampling for leakage (valve 4) may be delayed until the end of the accumulation period. However, if this is done, time is lost and the sensor will be subjected to a more concentrated halogen sample if the device has a large leak.
13. If the device has been pressurized with halogen tracer for leak test only, exhaust the test gas outside the test area or recover for reuse.
14. Remove the device from the shroud and write the test report (Fig. 26) or otherwise indicate the results of the test as required.

FIGURE 27. Cross section of combination pressure vacuum leech box used for halogen leak testing by the detector probe technique.



Combination Pressure Vacuum Box Leak Testing of Halogen Pressurized Systems

By means of a flexible combination pressure vacuum box, a temporary closed system capable of being pressurized is locally produced over a section of weld in the test boundary to be tested with the halogen detector probe. Figure 27 shows a cross section sketch of a typical combination pressure vacuum leech box for halogen leak testing. Figure 28 shows fabrication details, including valves and dial gages.

Both compartments of the combination pressure vacuum leech box shown in Fig. 27 are first evacuated using an air ejector or vacuum pump. The resulting greater external atmospheric pressure physically seals the box against that weld test section. The center compartment over the weld section can then be internally pressurized with refrigerant for halogen detector probe test. With the detector probe the operator then scans the opposite side of the boundary, such as a weld, pressurized by the halogen tracer filled internal chamber of the leech box.

An example of use of this type of pressure vacuum box is the leak testing of bottom head welds of a nuclear containment vessel, which are to be embedded in concrete before completion of the vessel. These welds must be halogen detector probe tested before embedding because of their inaccessibility during final test of the vessel.

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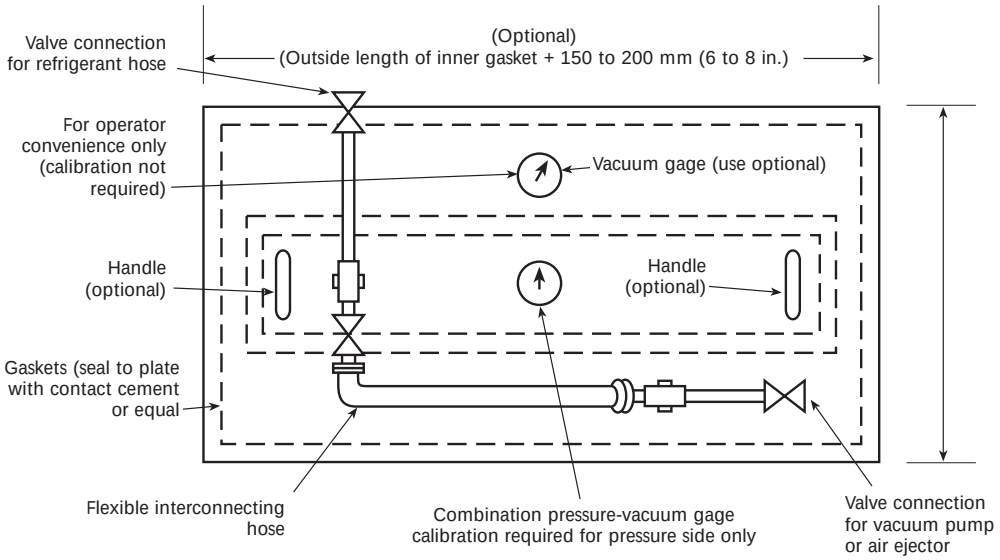
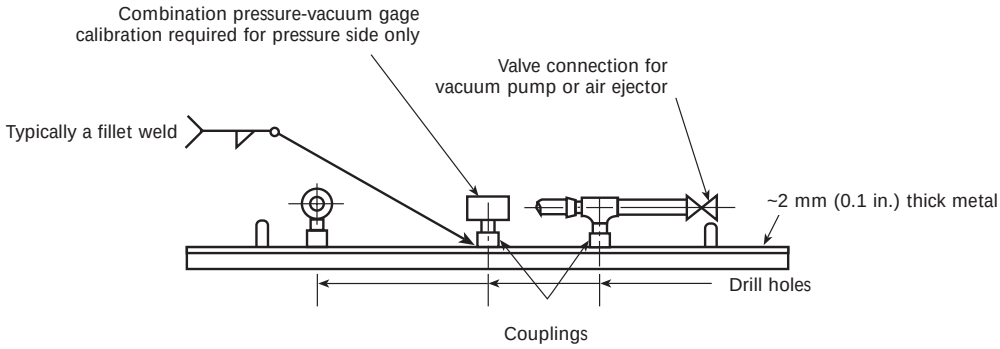
FIGURE 28. Fabrication details for design and construction of combination pressure vacuum leech box shown in Fig. 27: (a) plan view; (b) side view.

(a)



(b)



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PART 4. Industrial Applications of Halogen Leak Detection

Application of Halogen Leak Tests to Pressurized Enclosures

Pressurized enclosures and piping can be easily tested for leaks by using heated anode halogen leak detectors. Leaks are located by pressurizing these enclosures with a halogen tracer gas and manually probing seams, joins, welds etc. with the detector probe of the detector. If a leak exists in the enclosure, some of the halogen gas will pass through the leak and be detected by the leak detector.

Some examples of equipment that have been leak tested with halogen leak detectors are automobile air suspension systems, automobile radiators, air conditioning and refrigeration equipment, pressurized radar systems, nuclear power system structures and components, heat exchangers, dairy equipment, air compressors, steam boilers and piping, valves and pipe fittings, lengths of pipes and tubes, missile fuel tanks and fuel lines, aircraft fuel tanks, aircraft hydraulic systems, chemical and petroleum systems, underground pipe lines, transformers and hermetically sealed instruments and components. These heated anode halogen vapor detectors are suitable for use in any atmosphere that does not contain combustible or explosive gases.

Comparison of Halogen Vapor Tests with Other Leak Tests

In most cases halogen leak detectors are much more sensitive, faster, more reliable and in general a cleaner and easier means of leak testing than the ordinary methods such as bubble, hydrostatic, pressure drop or halide torch testing. However, there are some applications where one of these alternative methods may be more suitable or more sensitive. For example, bubble testing is better for locating leaks in a tank filled with natural gas than is heated anode leak detection, which presents the hazard of an explosion. Safer alternative methods would be to use an electron capture type of halogen leak detector or ultrasound detector. With leak testing equipment such as boilers that are given a high pressure hydrostatic test as required by law, it may be that the hydrostatic test is actually more sensitive than the halogen leak detector. This follows because it is possible to use much higher pressures with the hydrostatic test than with tracer gas and compressed air, for safety reasons. The extremely high pressures possible during hydrostatic testing sometimes cause leaks to open up that do not exist at lower pressures.

There are a few cases such as those cited above where the halogen vapor leak detector may not be as suitable or as sensitive as another method of leak testing. However, on most applications, the halogen test is among the most sensitive, most positive, easiest and cleanest to use of all methods of leak detection for leakage to the atmosphere.

Determining Percent Tracer Gas and Test Pressure Required for Leak Tests

The percent tracer gas and the pressure required within the enclosures depend on the size leak to be detected and the normal operating pressure of the equipment being tested. A relatively low positive pressure within the enclosure is sufficient to permit leak testing. However, if the piece of equipment being tested normally operates at some positive pressure or if a leakage rate is specified at some pressure, it is recommended that the halogen leak test of the enclosure be performed at its normal operating pressure or at the pressure at which the leakage rate was specified. (For leak testing of some refrigeration or air conditioning systems, for example, a maximum allowable leakage rate of 0.3 $g \cdot yr^{-1}$ (0.01 $oz \cdot yr^{-1}$) of refrigerant-134a refrigerant gas may be specified.)

A safety factor of four or five is used in determining the test leakage rate to compensate for normal factory conditions, operator carelessness, loss in sensitivity of the sensitive element etc. This safety factor of 4.5 has been factored into Table 8, based on a nominal sensitivity setting of near 10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} std $cm^3 \cdot s^{-1}$).

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Determining Partial Pressure of Tracer Gas Required

To determine the partial pressure of refrigerant-12 gas needed in the enclosure to get a certain percent mixture, use may be made of Eq. 4:

$$p = \frac{\%T \times P}{100} \quad (4)$$

where p is partial pressure of refrigerant gas; $\%T$ is percent of tracer gas by volume; and P is total absolute pressure in the tank, in kilopascal ($lb_f \cdot in.^{-2}$ absolute).

Figure 1 indicates the maximum pressures possible with halogen tracer gases refrigerant-22 and refrigerant-134a in equilibrium with their liquid forms at various ambient temperatures. To attain 100 percent halogen tracer gas within test systems at these pressures, the system must be evacuated before filling it with the halogen tracer gas. Dilution of the halogen tracer with a neutral pressurizing gas such as air or nitrogen is necessary to attain test pressures in excess of limits.

Determining Partial Pressure of Tracer Gas from Pressurizing Conditions

The partial pressure of refrigerant is that portion of the total absolute pressure in an enclosure due to the refrigerant gas content (Fig. 16). In other words, if a tank that contains air at atmospheric pressure (about 100 kPa or 15 $lb_f \cdot in.^{-2}$ absolute) and refrigerant gas is added to raise the pressure to about 140 kPa (20 $lb_f \cdot in.^{-2}$ absolute), then the partial pressure of refrigerant is about 40 kPa (5 $lb_f \cdot in.^{-2}$) and the percentage tracer gas is 28 percent. The pressure of refrigerant gas can be added at any air pressure below the vapor pressure above the liquid refrigerant. In this case it was added when the tank was at atmospheric pressure of 100 kPa (15 $lb_f \cdot in.^{-2}$ absolute), raising the pressure to 140 kPa (20 $lb_f \cdot in.^{-2}$ absolute). If the final test pressure were to have been 280 kPa (40 $lb_f \cdot in.^{-2}$ absolute), more air could have been added after the refrigerant gas, thus raising the pressure to 280 kPa (40 $lb_f \cdot in.^{-2}$ absolute). Alternatively, the air could have been added before the refrigerant gas by raising the air pressure to 240 kPa (35 $lb_f \cdot in.^{-2}$ absolute) and then adding the 40 kPa (5 $lb_f \cdot in.^{-2}$) of refrigerant gas. Either sequence can be used unless the final test pressure is to be greater than the vapor pressure of the refrigerant, in which case the refrigerant should be added first.

However, it is advisable to initially pressurize with the refrigerant in order to reduce the described effects of cylinder cooling and cold weather, if applicable. This also aids in the dispersion and mixing of the refrigerant throughout the test system.

TABLE 8. Example of relation of percent refrigerant-22 halogen tracer gas at 200 kPa (15 $lb_f \cdot in.^{-2}$ gage) to detectable leakage rate.[a]

Tracer Gas	Leakage Rates			
Percent	$Pa \cdot m^3 \cdot s^{-1}$	(std $cm^3 \cdot s^{-1}$)	$g \cdot yr^{-1}$	($oz \cdot yr^{-1}$)
100	9.0×10^{-7}	(9.0×10^{-6})	1.5	(0.05)
50	1.8×10^{-6}	(1.8×10^{-5})	3	(0.1)
25	3.6×10^{-6}	(3.6×10^{-5})	6	(0.2)
10	9.0×10^{-6}	(9.0×10^{-5})	15	(0.5)
5	1.8×10^{-5}	(1.8×10^{-5})	30	(1.0)
1	9.0×10^{-5}	(9.0×10^{-4})	150	(5.0)
0.5	1.8×10^{-4}	(1.8×10^{-3})	300	(10.0)

a. Safety factor of 4.5 is included in these tracer gas concentrations. The assumed halogen leak detector sensitivity setting is 2×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (2×10^{-6} std $\cdot cm^3 \cdot s^{-1}$).

Preparing the Pressurized Systems for Halogen Leak Testing

If the enclosure already contains a halogen tracer gas, as do refrigerators or air conditioners that contain one of the refrigerant gases, it is ready for leak testing. Refrigerant gases are relatively inert to metals, rubber, plastic or other types of materials when in the dry vapor state. Other enclosures must have a halogen tracer gas introduced into them under pressure. Before charging an enclosure with tracer gas, it should first be emptied of all liquids. Refrigerant-134a is recommended as a tracer gas because it is odorless, relatively nontoxic, chemically inert and available at any refrigerant supply company. It may be purchased in small cans or in large tanks depending on the most economical arrangement for a particular application. The actual mixing of the desired percentage of tracer gas and the filling can be accomplished in several ways, as described next. Usually one way will be simplest and most satisfactory for a particular setup.

Programmed Fill Method of Charging Test Enclosure with Refrigerant-22 Tracer Gas

In the programmed fill method of providing air mixed with tracer gas, the enclosure being tested is filled with tracer gas (refrigerant-22) to a predetermined pressure (see Table 8), then completely filled with air to the final test pressure. This method is simple and is suitable for

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compact shapes such as tanks where turbulence produced by filling ensures good mixing of the gases. If tubular coils, complete refrigeration systems, or long restricted devices are filled by this technique, large variations in mixture percentage may result and premixing of gases is recommended (see below).

Simultaneous Fill Method of Pressurizing Test Enclosure

In the simultaneous fill method of providing air mixed with tracer gas, air and halogen tracer gas are admitted simultaneously in the proper proportions by means of metering restrictions of valves. This method will produce good mixing in any pressurized device. The test pressure will be limited to about one half that of the halogen gas tank pressure in order to have adequate pressure drop across the metering orifices.

Premixed Fill Method of Pressurizing Test Enclosure

In the premixed fill method, the enclosure under test is filled directly from a line containing the proper mixture of halogen tracer gas and air. This is the best method for high pressure leak testing of devices having an internal shape not suitable for internal mixing, as outlined in the program fill method. It is also convenient where a large number of leak testing stations are to be serviced with the mixture. The mix can be produced in two ways.

1. In the program fill method, the test gas storage tank is filled with a certain ratio of tracer gas and air by using the ratio of partial pressures.
2. When mixing before compression, the proper amounts of halogen tracer gas and air are metered into the compressor intake. Care should be taken that the partial pressure of tracer gas in the tank does not exceed its vapor pressure.

For either method it is suggested that the unit under test be evacuated and backfilled to eliminate trapped air and moisture in capillaries and blind ducts.

Leak Searching Procedures

After the enclosure has been prepared for testing by charging it with a halogen tracer gas, it is ready for leak testing. Leaks are located by manually probing seams, joints, welds and other areas suspected of leaks with the gun or pencil shaped detector probe, depending on which leak detector is being used. When probing along seams and welds, the operator should place the tip of the probe on the test object surface and move it along the seam at a rate of about 2 $cm \cdot s^{-1}$ (0.8 $in. \cdot s^{-1}$). If a leak is encountered, the operator will be warned in two ways: by an audible alarm from the control unit and by a deflection of the pointer of a leak indicating instrument on the control unit. In some cases, another leak indicating instrument is built into the detector probe gun itself.

Accumulation Technique of Halogen Leak Testing

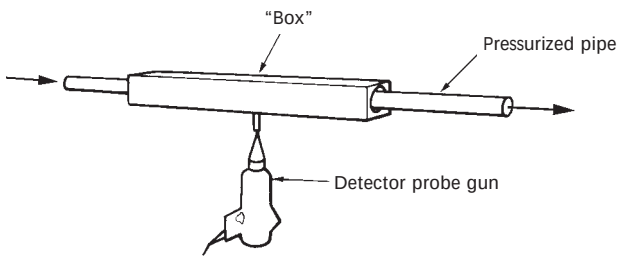
The accumulation technique of leak testing allows greater sensitivity than is possible by direct probing. The unit to be tested is placed in a relatively tight enclosure and leakage, if any, is allowed to accumulate for minutes or hours, depending on the sensitivity required. After the accumulation period the interior of the enclosure is probed through a small hole, or a sample is drawn from within the enclosure through a sampling line or pipe.

Individual welded seams in a tank or cylinder are easily enclosed by taping a section of plastic over them. After allowing time for leaks to accumulate, the edge of the plastic would be lifted just enough to insert the probe of the detector under the plastic. Pipe joints or other fittings can be enclosed in a small plastic bag or ball similar to a split tennis ball.

Special Adapters for Leak Testing Tubing or Pipe

Normally most enclosures can be leak tested with the standard detector probe on the detector. However, some applications may require building a special adapter for use with the detector. Figure 29 shows a special adapter for testing lengths of tubing or pipe for leaks. The tube is first pressurized with a halogen tracer gas and then slowly passed through the box. As it leaks, some of the gas will escape into the box and be drawn into the leak detector. The size of these

FIGURE 29. Special adapter for testing lengths of tubing or pipe.



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enclosures must be limited to a volume that can be swept in a reasonable length of time by the air flow created by the probe.

Halogen Background Control and Correction

In many factories where leak testing is being conducted, a background level of halogen gas will build up from leakage in the units being tested, leaks in the supply tank and lines used to store the tracer gas being used and from other sources that may allow halogens to enter the area. The automatic balance feature of the control unit of halogen vapor detectors will balance out a certain amount of halogen background provided the concentration is constant or changing slowly. However, if this background level builds up to a point where normal air currents present in the room cause sudden changes in background concentration, transient leak signals may result even when the detector has not encountered a leak.

To combat this condition, a two pronged attack is most effective.

1. Eliminate sources of background: indiscriminate dumping of refrigerant charges, leaky lines, degreasers using halogen solvents, paint fumes etc.
2. Provide a controlled environment of fresh air in the testing area. This second approach can be a very elaborate one or a very simple and inexpensive one, depending on the severity of the background problem.

Manufacturers of refrigerators and air conditioners have found it desirable to construct a small room or booth for testing because the background level of tracer gas is usually very high in their testing areas because of the many sources of halogen gas. This room or booth is fed fresh air from the outdoors at a very low velocity to prevent excessive drafts and eddy currents of air within the room.

In other areas where the background level is low, it may not be necessary to use any special ventilating techniques other than the normal ventilation required for good health. A simple ventilating device that can be used, if it is impractical to build a special room or booth, is a large portable electric fan placed in a window or doorway. Arranged in this manner, the fan will usually clear out the halogen background to the point where leak testing can be accomplished satisfactorily.

Calibration with Standard Halogen Leaks

Halogen leak detectors are not quantitative devices in themselves but indicate only the relative size of a leak. For those applications where it is necessary only to determine if a leak exists and the location of the leak, it is not necessary to calibrate the detector. However, it is good practice to have some means of determining when the sensitivity of the heated anode element has dropped off to a point where the element must be cleaned or replaced.

An adjustable reference standard halogen leak is recommended (1) if it is desired to measure the sizes of leaks detected because of leakage rate specifications that must be met by the equipment or (2) if it is necessary to correlate the sensitivity of a leak test made in one area with that made in another area. The leak standard consists of a reference leak that can be adjusted at specified rates within its range. By selecting a range that includes the size of leak to be detected, the operator can calibrate the leak detector by sampling this leak.

Halogen Tracer Gases Other than Refrigerant Gases

A halogen leak detector is sensitive to any gas that contains a halogen and can be used to leak test enclosures that may contain these gases. For example, some power transformers are filled with sulfur hexafluoride, which contains a halogen. These transformers can be leak tested at the factory before shipment or at any time during the life of the unit without having to introduce another tracer gas. (For gases to which the halogen leak detector is sensitive, other than the family of refrigerant gases, see Table 1.)

Reclaiming Tracer Gas from Pressurized Enclosures

It may be desirable to reclaim the tracer gas used to pressurize a test enclosure or test object following leak testing, especially if it is large in volume and a high concentration of tracer gas has been used. This can be accomplished by using a gas compressor that will pump the gas out of the object or system being tested into a storage tank. A succeeding test object is prepared for testing by connecting it to

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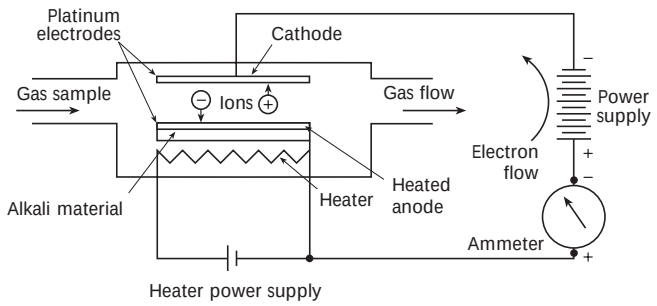
the storage tank and allowing the tracer gas to flow into it. The halogen tracer charging and reclaiming systems should be designed by experienced personnel familiar with the special problems involved.

Halogen Leak Detector Sensor Signal and Operating Range

An important part of any leak detector is the device that gives an output signaling the presence of the tracer to which it is sensitive. The better sensors will have an output of useful magnitude proportional to the amount of tracer input over a wide range. The halogen leak detector consists of a halogen sensing element, a small air pump to draw the leak sample through the element, power supplies and amplifiers to give the required outputs. The outputs are usually an instrument indication, a variable frequency audible signal and a control relay.

The halogen sensing element shown in Fig. 30 is a diode constructed to operate in air instead of a vacuum. It has two platinum electrodes, the positive one of which is heated and a source of alkali metal (lithium, sodium, potassium, rubidium, or cesium), usually contained in one of the electrodes. These alkali atoms then diffuse through the thin platinum of the electrode and at operating temperature collect on the surface as positive ions. If a negative potential is applied to the opposite electrode, some, but not very many, of these positive ions will be attracted to it. However, when halogen is present, many ions are released, the quantity depending on the amount of halogen present. This flow of ions generates the leak signal current, proportional to the tracer gas leakage rate over the range from 10^{-11} to 10^{-6} Pa·m^3·s^{-1} (10^{-10} to 10^{-5} std cm^3·s^{-1}).

FIGURE 30. Schematic diagram of a heated anode halogen vapor sensor.



When the electrical output of the element is amplified by a simple two-stage amplifier, the signal from a 10^{-10} Pa·m^3·s^{-1} (10^{-9} std cm^3·s^{-1}) pure halogen gas leak will typically give a one-third full scale reading on the output instrument. The signal is of exceptionally large magnitude, as many alkali ions are transferred for each molecule of halogen gas passing through the sensor. This makes possible the construction of a leak detector of high sensitivity using very simple electrical circuitry.

Preferred Applications for Halogen Leak Detection

From the foregoing discussions, it is seen that lack of adequate sensitivity rarely rules out use of a halogen leak detector. The halogen detector is eminently suited for the testing of refrigeration systems already charged with a halogen refrigerant. At the other extreme, the halogen detector is practically useless for leak testing aircraft instruments filled with 10 percent helium in nitrogen and sealed. The factors favorable to halogen leak testing include the following.

Low Cost and High Sensitivity. Because of its simplicity, the cost of an operational halogen leak detection system is unusually low. It is, in fact, the lowest priced high sensitivity leak detector available.

Low Operation and Maintenance Expense. The halogen detector is simple to operate and maintain.

Portability. The halogen detector weighs less than 2 kg (4.4 lb_m). This is an asset if the detector must be brought to the product to be tested, or if the system to be tested covers a large area.

Long and/or Restricted Systems to Be Tested. For effective location of leaks, the leak signal from the detector must occur soon after the point suspected of leaking is probed. This signal must also decrease quickly after the probe is removed, as sketched in Fig. 31. A fast response as shown is good; the size and location of the leak can be quickly determined. However, a vacuum leak test response through a long and/or restricted system may have a slow response, as shown in Fig. 31.

A long delayed and smeared response such as this is difficult to interpret. Conditions that cause poor leak test response will be analyzed later as some actual leak testing problems are discussed. The response time of the halogen detector in pressurized leak testing is uniformly fast, regardless of system size and configuration, because the tracer gas from the leak does not have to pass through the system first to get to the leak detector.

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Consumer Products Leak Tested in a Pressurized Condition

Products such as inner tubes, football and basketball bladders, flexible airtight product packaging, etc. are not readily tested under vacuum because of their nonrigid nature. Likewise, containers for pressure dispensed materials such as sprays, foams, dry powder and carbon dioxide fire extinguishers require that the leak test be done after the package is filled and pressurized.

Leak Testing of Fluid Filled Devices with Addition of Halogen Tracer

Many articles already filled with a circulating nonhalogen fluid may be easily tested by adding a soluble halogen tracer. For example, water cooled internal combustion engines may be checked for leaks into the combustion chambers by a teaspoon of trichloroethylene added to the cooling water while sampling the exhaust. Oil filled hydraulic or fuel systems may be similarly tested.

Allowable Levels of Halogen Contamination of the Air in Leak Testing Areas

It is the basic nature of any tracer gas leak detector that it will respond to its own type of tracer gas from any source. Whether tracer gas comes from a leak, from materials in which it has been absorbed, or from the atmosphere, the detector will respond to it. Thus, freedom from spurious sources of tracer gas is a basic requirement of all gas leak detectors. The basic requirement of the halogen sensor is that the background halogen content of the air not exceed about 10 $\mu L \cdot L^{-1}$ or a concentration equal to that existing in the probe because of the mixture of the minimum size leak to be detected with the probe air flow, whichever is smaller.

For most applications, refraining from deliberate release of halogens into the air is sufficient to meet this requirement. Thus, in more than 90 percent of leak testing applications, it is suitable to use the halogen detector in room air with no special atmospheric control whatever. In those cases where control of atmospheric halogen is necessary, it can usually be accomplished in a simple manner at little expense.

Halogen Leak Testing of Cryogenic Plate Coil

The importance of the configuration of the part being leak tested is illustrated by halogen leak testing of a large cryogenic plate coil about 1.8 × 2.4 m (6 by 8 ft), with long, restricted internal passages (Fig. 32). This cryogenic plate coil was contained in a simulator vacuum chamber with leaks that prevented attainment of the required vacuum. The leak was determined to be in the cryogenic plate coil panels because the leakage stopped when they were evacuated. Repeated attempts to locate the leak were unsuccessful. Obviously, the system was both long and restricted. Because leak testing was done under vacuum, it was a severe case of time lag and signal smear, as illustrated top half of Fig. 31. A pressure test was then performed after pumping out the panels and backfiring them with dichlorodifluoromethane to 350 kPa (50 $lb_f \cdot in.^{-2}$ gage). Probing for

FIGURE 31. Leak detector response time curves.

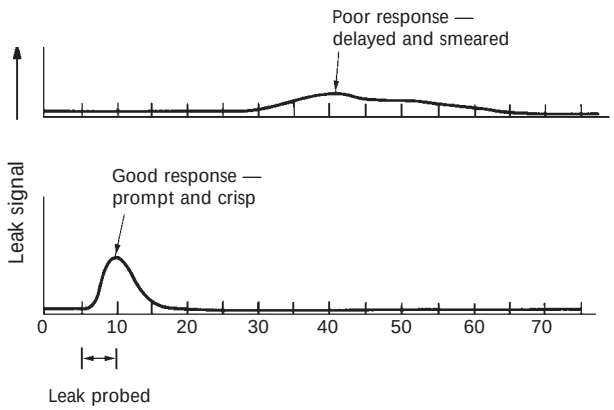
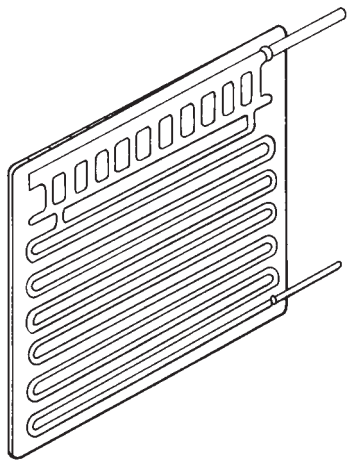


FIGURE 32. Cryogenic plate coil.



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leaks with the halogen detector located five leaks in the 10^{-6} to 10^{-8} Pa·m^3·s^{-1} (10^{-5} to 10^{-7} std cm^3·s^{-1}) range within a few minutes. The leaks were repaired by welding and the repaired joints were retested. When the system was put in operation, there was no further problem with leaks.

Techniques for Purging or Pressurizing Long or Restricted Systems

The application of Fig. 32 illustrates an important characteristic of systems of this type. Because of time lag and signal smear (Fig. 31), vacuum leak detection in systems of this configuration is difficult and sometimes completely unsatisfactory. Likewise, pressurization of this type of system with tracer gas requires the proper technique to ensure that the system is uniformly filled with tracer gas at all places. If tracer gas is merely applied to one end of the system, the air in it will be compressed at the far end and will contain little or no tracer gas. The most reliable way to ensure uniform filling is to evacuate most of the air before pressurizing. Another way, suitable for single path systems, is to purge the air out of the far end of the system as tracer gas is admitted. A pressure test is usually better for long, restricted systems.

Halogen Leak Testing of Household Refrigerator Unit

Small refrigeration and air conditioning units are another outstanding example of severe restriction in a system that must have an accurate leak test for proper field performance. As illustrated in Fig. 33, the high pressure side (warm) is connected to the low pressure side (cold) by a liquid expansion device, typically a meter length of small tubing with an inside diameter of about 1 mm (0.04 in.). Because of the compressor valves, any leak in the high side has to pass through this tube if a vacuum test is used. If a pressure test is used, the tracer gas must be filled and removed through this tube.

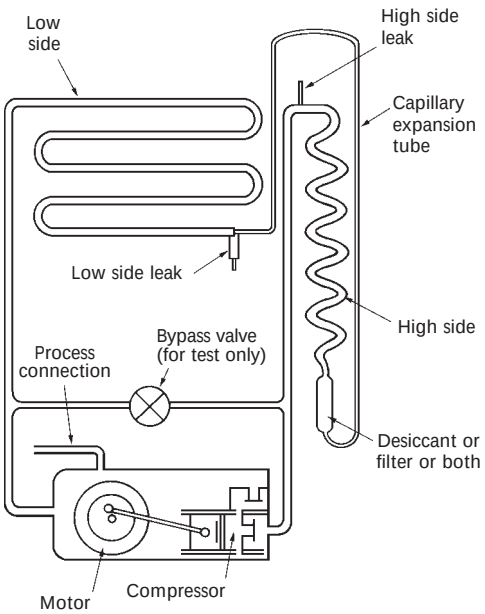
Tests were conducted with 1.2×10^{-6} and 1.2×10^{-5} Pa·m^3·s^{-1} (1.2×10^{-5} and 1.2×10^{-4} std cm^3·s^{-1}) calibrated leaks installed respectively on the high side and low side of the refrigeration system. With the refrigeration system pressurized with refrigerant-12 to 175 kPa gage (25 lb$_f$·in.$^{-2}$ gage), these leaks, which were many times larger than the operational specification leak size, could not be detected. Examination showed that the calibrated leaks were plugged with water, which was removed by baking the leaks at 200 °C (400 °F) for 30 min. The leaks were then recalibrated and reinstalled on the refrigeration system, one on the high side and one on the low side.

A vacuum leak test was performed first. Response to the low leak, which has a 5 mm (0.2 in.) inside diameter passage leading to the refrigerator process connection, began in about 20 s and was complete in 5 min. Response to the high side leak took about 15 min to start and had a leak response time constant of about 1 h.

A pressure leak test was then performed. The unit was evacuated to a pressure less than 1 kPa (10 torr) and back filled with refrigerant-22 to a pressure of 800 kPa absolute (115 lb$_f$·in.$^{-2}$ absolute). This required about 40 s. Response time when probing the pressurized leaks was not less than 0.5 s. In addition, the magnitude of response was much greater due to the increased leak flow at higher pressure.

As shown in Fig. 33, a valved bypass was installed between high and low sides and another vacuum leak test was performed with the bypass valve open. Response to the leak tracer gas was now noted in 3 s, with a time constant of about 90 s.

FIGURE 33. Schematic diagram of a typical household refrigerator unit.



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Design of Products and Systems to Facilitate Leak Testing

Problems of leak testing should be considered during the design of a product, not left as an afterthought. Often the addition of a bypass or port, or a slight change in configuration, can make a tremendous difference in the ease and accuracy of leak testing. In the case of the refrigerator unit of Fig. 33, a process bypass tube, pinched closed after leak testing, would make a great improvement in vacuum leak testing the product and would speed proper filling with tracer gas for pressure leak testing. The type of leak test selected can make a vast difference in the time required for the leak test. Testing time should be considered when selecting the leak test technique to be used. The flow of gas through a leak increases (roughly) as the square of the pressure across the leak. This makes it advantageous to test at the highest pressure that is practical.

Avoidance of Prior Water Immersion of Components to be Leak Tested

Components and systems to be tested for leaks smaller than 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}) should not be previously immersed in water. As the refrigerator example of Fig. 33 shows, water is a wonderful temporary plug for small leaks. If a part must be immersed for rough leak testing, cleaning or the like, it should be vacuum dried, purge dried, or baked at a temperature well above the boiling point of the liquid to evaporate these liquid leak plugs before performing a more sensitive leak test.

Calibration of the Test System

Whenever applicable, the leak test performance from the leak detector, through to the suspected leak area should be checked by inserting a calibrated leak of specification size in the system being tested. The calibrated leak should be located at a point most remote or restricted from the sampling or pressurizing connection. By doing this, an actual leak in the system is actually detected and the sensitivity is known or can be determined.

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PART 5. Writing Specifications for Halogen Leak Testing

Good leak testing is an important part of the manufacture of many products but is of little value unless coupled with leak specifications that are realistic, concise and meaningful to all persons concerned. Specifications and procedures must be adequate to fulfill their needs. Education in the meaning and use of specifications will have to continue because the halogen detector plays an important role in industrial leak detection.

The first contact a manufacturing person usually has with leak testing is to receive a specification restricting the amount of leakage allowed in manufactured product. However, the one place in leak testing where more people go wrong than any other is in the writing and application of leak specifications. The chances are very good that the specification will fall into one of two categories: it will either be very simple but incomplete and unusable, or it will be very detailed and restrictive. It may say, "There shall be no leakage in this part." This creates a serious dilemma. No leakage when tested with which method, using which test medium, under what pressure? A bubble test will show leaks that an air pressure drop test will not. A helium or halogen test will detect leaks impossible to find with bubbles. The wise manufacturer will go back to the customer and request a more definitive specification.

Determining Halogen Vapor Leak Test Specifications

It is believed that all leaks in materials, whether from seams, joints, welds, pores or couplings, will pass a liquid or gas under certain conditions of pressure and time. How much leakage is present is the important thing to consider. It is meaningless to say that an enclosure shall have no leaks.

Defining Physical Leaks in Terms of Leakage Rates

The word *leak* as used in this book denotes a hole or aperture in a barrier. A pinhole, for example, is a large leak. A hole 0.1 mm (0.004 in.) in diameter can be quite objectionable in a refrigeration system. On the other hand, 1 cm^3 of gas may take as long as one million years to pass the barrier. For all practical purposes, this leak can be considered insignificant. The manufacturer, customer and the leak test operator are concerned, therefore, with how much gas or liquid passes through a barrier in a period of time, i.e., the leakage rate.

Defining Leakage Rates in Practical Terms

Leakage rates are expressed in a variety of ways. Typical leakage specifications have be given in units such as ounce per year, cubic centimeter per second, bubbles per minute, pressure drop per hour etc. Volume flow rates mean little unless pressure conditions are specified. It is important that one terminology be established and used consistently. The refrigeration and air conditioning industry in the United States in the twentieth century has used leakage rate units of ounce per year. Generally, all other leak testing procedures call for pascal cubic meter per second or standard cubic centimeter per second. Table 9 lists equivalent halogen leakage rates. All numbers on the same line (reading across) are approximate values of leakage rates for air and for refrigerants at the same pressure (through the same physical leak). For all practical purposes these relative rates of leakage may be used interchangeably.

Reasons for Specifying Leakage Rates

Specifications concerning leakage rates are necessary for many reasons.

1. There should be agreement between the manufacturer and customer on the leak integrity of the product;
2. The factory can cut production costs by finding only significant leaks.
3. Field service costs can be cut by being certain that all significant leaks have been found before shipment.

The first step in preparation for writing leak testing specifications is to decide how large a leak can be tolerated.

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Operational Leak Specification for Household Refrigerator

The designer of a typical household refrigerator may determine that the loss of 30 g (1 oz) of the refrigerant charge will result in unacceptable operation for a unit with maximum operating pressure of 1.25 MPa (180 $lb_f \cdot in.^{-2}$ gage). The marketing organization wants to give a 5 yr warranty. Thus, the maximum loss that can be tolerated is 6 $g \cdot yr^{-1}$ (0.2 $oz \cdot yr^{-1}$). Because 1 g of refrigerant at room temperature is equivalent to 18.8 $Pa \cdot m^3$, corresponding to a maximum allowable leakage rate of 3.6×10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (3.6×10^{-5} std $cm^3 \cdot s^{-1}$).

The operation specification will be for maximum total leakage of 6×10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (6×10^{-5} std $cm^3 \cdot s^{-1}$) gas (refrigerant-12) at 1.25 MPa (180 $lb_f \cdot in.^{-2}$ gage) to atmosphere. The gas or liquid whose leakage rate is specified should be the one that the device sees in service. This operational specification says that no refrigeration units should leak in excess of this amount when they are installed and are in operation at the customer's home. The mistake should not be made, however, of using this operational specification as a leakage test specification on the production line, where a safety factor should always be applied.

Safety Factor in Specifying Allowable Leakage Rates during Leak Testing

As with any other manufacturing or testing process, there are random variations, human factors and equipment variables that affect the accuracy of the leak testing work. For this reason, a tolerance or safety factor in leakage measurement must be assigned. The leakage safety factor provides reasonable assurance that an adverse combination of test variables will not result in shipping an anomalous unit. In this case, the designer might assign safety factors in leak testing as follows.

1. For variations in flow between test gas and halogen under operating conditions, allow a reduction factor of $5 \times 10^{-1} = 0.5$.
2. For operator variations in leak testing procedures, allow a reduction factor of $4 \times 10^{-1} = 0.4$.
3. For possible doubt in leak detector accuracy, allow a reduction factor of $5 \times 10^{-1} = 0.5$.

The overall reduction rate to be applied during leak testing is the product of these factors: $(5 \times 10^{-1}) \times (4 \times 10^{-1}) \times (5 \times 10^{-1}) = 0.1$. This means that the leak test on the production line should be 10× as sensitive as required to meet the warranty on the end product. This is equivalent to using a safety factor of 10× in specifying the maximum allowable leakage rate during leak testing.

Example of Leak Testing Specification for Total Leakage

Applying the tolerance or safety factor of 10× to the operational specification, a testing specification for total leakage of $3.6 \times 10^{-7} \cdot m^3 \cdot s^{-1}$ (3.6×10^{-6} std $cm^3 \cdot s^{-1}$) of test gas at 1250 kPa (180 $lb_f \cdot in.^{-2}$ gage) to atmosphere is obtained for the household refrigerator given in the prior example. The specification now satisfies the designer; it approximates operating

TABLE 9. Equivalent leakage rates at same pressure for air and refrigerant-12, refrigerant-22 and refrigerant-114.

Air				Refrigerant			
$Pa \cdot m^3 \cdot s^{-1}$	$in.^3 \cdot day^{-1}$ [b]	$\mu m \cdot ft^3 \cdot h^{-1}$	$\mu m \cdot L \cdot s^{-1}$	Mass ($g \cdot yr^{-1}$)	Mass ($oz \cdot yr^{-1}$)	Time[c] (yr)	By Volume[d] ($\mu L \cdot L^{-1}$)
1.8×10^{-3}	9.46×10^{1}	1.72×10^{3}	1.37×10^{1}	3.0×10^{3}	1×10^{2}	1.6×10^{-1}	2.29×10^{4}
1.8×10^{-4}	9.46	1.72×10^{2}	1.37	3.0×10^{2}	1×10^{1}	1.6	2.29×10^{3}
1.8×10^{-5}	9.46×10^{-1}	1.72×10^{1}	1.4×10^{-1}	3.0×10^{1}	1	1.6×10^{1}	2.29×10^{2}
9.0×10^{-6}	4.73×10^{-1}	8.5	7.0×10^{-2}	1.5×10^{1}	5×10^{-1}	3.2×10^{1}	1.145×10^{2}
1.8×10^{-6}	9.46×10^{-2}	1.72	1.4×10^{-2}	3.0	1×10^{-1}	1.6×10^{2}	2.29×10^{1}
1.8×10^{-7}	9.46×10^{-3}	1.7×10^{-1}	1.4×10^{-3}	3.0×10^{-1}	1×10^{-2}	1.6×10^{3}	2.29
1.0×10^{-9}	5.6×10^{-5}	1.0×10^{-3}	7.6×10^{-6}	1.8×10^{-3}	6×10^{-5}	2.7×10^{5}	1.3×10^{-2}
1.0×10^{-11}	5.6×10^{-7}	1.0×10^{-5}	7.6×10^{-8}	1.8×10^{-5}	6×10^{-7}	2.7×10^{7}	1.3×10^{-4}

a. Refrigerant-12, refrigerant-22 and refrigerant-114, under the same conditions of pressure and temperature, will pass through a given leak at about the same volumetric rate.
b. Under standard atmospheric conditions.
c. Time for 0.45 kg (1 lb) to leak.
d. Assumes probe flow of 3 $L \cdot h^{-1}$ (0.1 $ft^3 \cdot h^{-1}$) and a leak of 100 percent refrigerant.

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conditions, which is desirable, and will allow only adequately tight units to be shipped.

Example of Leak Testing Specification for Each Leak

The factory may be set up to test most effectively for leak location by the probing method. Thus, there may be several leaks, each smaller than the total allowable leakage but which together will add up to more than this amount. What allowance should be made for this? The probability that all leaks smaller than a given size will add their leakage to give a total leakage rate more than two or three times the given size is quite small. However, the designer should assign a factor based on an evaluation of the product. In this example, a factor of 0.5 will be assigned to account for the possibility of several small leaks. When combined with the 10× factor for uncertainties listed previously, this corresponds to an overall leak test safety factor of 20×. This gives a value for each individual leak of only 1.8×10^{-7} Pa·m^3·s^{-1} (1.8×10^{-6} std cm^3·s^{-1}) test gas at a pressure of 1.25 MPa (180 lb$_f$·in.$^{-2}$ gage) leaking to atmosphere for the prior example of the household refrigerator.

Estimating Liquid Leakage Rates from Gas Leakages

Experimental data indicate that no visible water will leak when dry air, at the same pressure, leaks at a rate as great as 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}). To be on the safe side, it is believed that enclosures that are to contain liquids such as water or oil should have no leaks at rated pressure that are larger than 10^{-6} Pa·m^3·s^{-1}. The volume of helium leakage is about the same as leakage for air and refrigerants for leaks of about 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}). For leaks smaller than this, the volume of helium leakage will be somewhat greater than air through the same physical leak at the same pressure difference.

Enclosures that are to contain liquids such as water or oil can be tested under gas pressure, thereby possibly eliminating the problems and mess of hydrostatic testing. Thus, if the vessel is merely to contain water with no significant leakage, it should be specified that the vessel will have no gas leaks at rated pressure larger than 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) of refrigerant-12 gas. The rated pressure is the pressure the tank will be expected to withstand in normal usage. If the tank is an open or vented tank, this pressure below the top of the liquid is always above atmospheric pressure, 100 kPa (14.7 lb$_f$·in.$^{-2}$ absolute), because the weight of the liquids adds to the atmospheric pressure. Thus, all such testing should be done at an appropriate pressure above atmosphere such as 140 kPa (20 lb$_f$·in.$^{-2}$ absolute).

In some cases, the leakage rate is specified for an equivalent pressure of 5/3 of the working pressure, or as 1.5 times the working pressure. This level tests for safety of vessel plumbing as well as for leakage. This information should be included with the test specification. Consider safety when pressurizing tanks with a gas.

Specifying Pressure and Tracer Gas Percentage

Where an enclosure is to be pressurized in usage, the specification should always call for leak testing at a safe specified pressure, provided the test pressure does not exceed the design pressure. It should also state whether 100 percent tracer gas is to be used or whether part of the volume can be tracer gas and the balance air or nitrogen. Mixing tracer gas with air or nitrogen is nearly always possible and can represent a considerable saving in cost of tracer gas.

When the vessel to be leak tested has a relatively large volume, say 30 L (1 ft^3) or more, and when the leakage rate specification will allow, it is possible to use less than 100 percent refrigerant tracer gas and save money. Equation 4 shows how to compute the percentage of tracer gas to use. A 10× safety factor should be added to allow for operator technique, element stability, element sensitivity and normal variations in factory conditions.

Specifying Rating and Adjustments of Halogen Reference Standard Leaks

The calibrated leak reference standards are the go/no-go gages of leak testing. They are absolutely necessary for careful leak testing and should always be specified. Leak standards are available either for use at a fixed leakage rate or as a variable rate. When specifying a halogen leak standard with a fixed leakage rate, it is important to always add a suitable safety factor. For example, if there is a maximum allowable leakage rate of 3×10^{-7} Pa·m^3·s^{-1} (3×10^{-6} std cm^3·s^{-1}), then the leak standard may be 10^{-7} Pa·m^3·s^{-1} or, even better, 3×10^{-8} Pa·m^3·s^{-1} (3×10^{-7} std cm^3·s^{-1}), 10× safety factor. The size of the reference standard

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leak should be specified. If the specification gives an allowable leakage rate, the manufacturer's procedure controls test variables and is usually subject to approval by the customer.

Variable leak standards will allow the user either to change the leakage rate test setting or to change the amount of safety factors. Always use a leak standard with a full scale leakage rate to cover the specified leak test rates.

Specifying Conditions in Halogen Leak Testing

An important part of any test specification is to state where the leak testing shall be done and what conditions shall exist in the test area. The specification should state that testing is to be done in an area that will probably be free of background halogen contamination (away from paint shops, degreasers or refrigerant charging area). A little prior thought and planning can save many problems later. It is not always possible to locate the test area in the best place in the plant. When the test area must be located near possible contamination sources, then other precautions must be taken such as special fans to bring in fresh air or an isolation booth with fresh air introduced to booth.

Background contamination is probably the most bothersome problem affecting leak testing. If the atmosphere contains too much halogen gas, it will mask the real leak and make it practically impossible to zero the leak detector. A halogen gas background concentration of 2 $\mu L \cdot L^{-1}$ is considered high, especially with leakage rate specifications of 10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-6} std $cm^3 \cdot s^{-1}$) or smaller. The leak detector can be used with a leak standard and a pure air supply to determine the background contamination. A regular check to determine background contamination should be specified. The leak detector indication due to background contamination should never exceed that expected from the minimum leak to be detected.

Responsibilities for Implementing Leak Test Specifications

Ideally, a leak testing specification should express only (1) the maximum allowable leakage rate, (2) test pressure and (3) total leakage or any point leakage. The person doing the testing then has the responsibility of making sure that conditions are the best possible to do the testing.

The specification writer, however, does have the basic responsibility to point out unusual problem areas to the leak test operator. For example, a device containing glass envelope electronic tubes should not be pressurized with helium because the helium may diffuse through the glass. On the other hand, the conditions of the test such as the testing location, allowable background contamination, test booth and test equipment are the responsibility of the testing organization. The test operators have the basic responsibility to ensure that these items are selected and controlled so as to conform to specified conditions.

Examples of Concise Specifications for Halogen Leak Tests

One cannot overemphasize the importance of writing easily understood specifications. The following specification samples may be helpful to the user in this regard.

Example 1

The clean, dry tank shall be pressurized to 400 kPa (60 $lb_f \cdot in.^{-2}$ gage) with refrigerant-134a tracer gas. All detected leaks larger than 10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} std $cm^3 \cdot s^{-1}$) shall be found and repaired. The maximum test pressure shall be specified. The following points shall be considered in performance of this specification.

1. The normal operating pressure of the tank shall be 400 kPa (60 $lb_f \cdot in.^{-2}$ gage) at 24 °C (75 °F).
2. 100 percent tracer gas shall be used requiring an evacuation of the tank to a low level; 750 Pa (5 torr) is usually sufficient.
3. A leak standard with a full scale rating of 1×10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-5} std $cm^3 \cdot s^{-1}$) shall be used to calibrate the leak detector. It shall be set for 5×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-6} std $cm^3 \cdot s^{-1}$) as the reject point.

Example 2

The clean, dry tank shall be pressurized to a level of 350 kPa (50 $lb_f \cdot in.^{-2}$ absolute) with a 10 percent mixture of refrigerant-134a and nitrogen and sealed off. Leakage shall not cause the pressure to drop more than 35 $kPa \cdot yr^{-1}$ (5 $lb_f \cdot in.^{-2} \cdot yr^{-1}$). (Conversion of SI units should be done by the specification writer.) The volume of the tank is 85 L (3 ft^3). The amount of test gas required is $0.085 \times 350 = 30$ $Pa \cdot m^3$ (300 std cm^3), of which 10 percent or 3 $Pa \cdot m^3$ is refrigerant-134a.

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Specifications for Halogen Leak Testing of a Tank

A typical specification for halogen leak testing of a tank might be to reject a tank unit with any one leak larger than 3 g (0.1 oz) of refrigerant-134a per year when unit is pressurized to 350 kPa gage (50 × gage) after being evacuated to 10 Pa (0.1 torr) and back filled to 350 kPa gage (50 $lb_f \cdot in.^{-2}$ gage or 65 $lb_f \cdot in.^{-2}$ absolute).

1. To provide a safety factor of 2, set leak standard at 1.5 g (0.05 oz) refrigerant-134a per year or 9×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (9×10^{-6} std $cm^3 \cdot s^{-1}$).
2. Adjust sensitivity and/or set the scale range of leak detector so that when sampling the leak standard, set at 1.5 $g \cdot yr^{-1}$ (0.05 $oz \cdot yr^{-1}$), the panel meter pointer on the halogen leak detector will go to 5 on a scale from 0 to 10 (midrange on the meter scale).

Preparation for Halogen Leak Testing of Tank

Preparation for leak testing and calibration of the leak detector are very important and must be done properly for useful results. The unit tested must be clean and dry. Oil, water or other liquids will plug a small leak temporarily and prevent detection. To establish 100 percent refrigerant-134a in the tank (or system) it must first be evacuated to about 1 kPa (10 torr). This will also evaporate volatile liquids. If the units leaks so much that the vacuum cannot be established in a reasonable time, add refrigerant-134a until a positive pressure is established in the unit. Then locate and repair the gross leaks. Then evacuate to 1 kPa (10 torr) and backfill with pure refrigerant-134a for final test.

It is important to remember that tanks that have contained flammable liquids or explosive gases or vapors must be thoroughly purged to remove all traces of these materials before leak testing with the heated anode halogen leak detector.

Procedure for Halogen Leak Testing of Tank

Equipment required for locating a leak in a container by halogen leak tests includes (1) a halogen leak detector, (2) a reference leak such as a leak standard that can be preset to leak over a wide range of rates and (3) a supply of tracer gas such as refrigerant-134a.

The procedure for halogen leak testing of a tank includes the following steps.

1. Evacuate and pressurize the clean, dry tank with refrigerant-134a to 350 kPa (50 $lb_f \cdot in.^{-2}$ gage).
2. With the leak detector probe just touching the tank, move it along seams (or areas that may leak) at a speed of about 20 $mm \cdot s^{-1}$ (50 $in. \cdot min^{-1}$).
3. If the probe passes over a leak, there will be a leak signal.

If the signal is equal to or greater than when detector probing the leak standard, the unit should be rejected (to be repaired). The leakage point should be marked for subsequent repair. One can pinpoint the leak by moving the detector probe tip to the location that produces the maximum leak signal.

Technique for Halogen Leak Testing by the Accumulation Method

The accumulation technique of leak testing has been found to be extremely useful when the following conditions exist: (1) where there are several possible leak points but it is not feasible or economical to test every point; (2) where the leak to be measured is smaller than the maximum sensitivity of the leak detector; (3) where the background contamination is severe, making it difficult to stabilize the leak detector for detector probe testing; and (4) where a reading of total leakage is wanted.

In the accumulation leak testing technique, the device to be tested is placed in a leaktight chamber and leaks are allowed to accumulate for a period of time. The accumulation is then sampled by detector probe and the pointer deflection of the panel meter on the detector is noted. When this deflection is compared to a standard and the accumulation time is factored into the formula, the size of the total leakage can easily be determined. At this point the location of the actual leak is still unknown, but it is known whether the device meets the total leakage specification. If its leakage exceeds the total leakage specification, the individual leaks will have to be found and repaired.

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11

CHAPTER

Acoustic Leak Testing

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PART 1. Principles of Sonic and Ultrasonic Leak Testing

Airborne ultrasound detection is an inspection technique applied to locate leaks in pressurized or evacuated systems and high frequency discontinuities in mechanical and electrical components. Fluid leakage generates sound waves when the fluid flow through the leak is accompanied by turbulence, cavitation or high velocity flow. These sonic disturbances can be transmitted through the medium of the pressurizing fluid, through the containment structure or through the atmosphere surrounding the leak location. Airborne ultrasound can be detected at a distance from its source with directional scanning microphones or acoustic probes. Leak testing and location from a distance through air or other fluids involves remote scanning of suspected leak areas with a directional probe and coordinating the direction of leakage's characteristic hissing sound with the relative sound intensity. Certain precautions must be observed if the sound source or leak location is to be reliably determined. These involve (1) avoidance of sound path blocks or sound absorbing materials that create sound shadows between the leak and the acoustic sensor and (2) recognition of possible sound reflectors such as flat, hard surfaces that provide sound echoes from directions other than that of the original leak source.

Figure 1 shows a portable ultrasound leak detector that can be used both in the active and passive modes. The system permits a wide range of applications including (1) detection of gas leaks in pressurized systems, (2) detection of gas leaks in vacuum systems, (3) detection of rubbing contacts, (4) detection of electrical discharges and (5) detection of leak conducted sound from artificial sources.

The most common application is the detection of gas leaks with the scanning probe (see Fig. 2). When a gas penetrates a leak opening, its molecules are moved by the pressure difference. The molecular agitation causes a sizzling noise with an extensive frequency spectrum that can be detected easily. The sensitivity of the ultrasound leak testing device is much better than the sensitivity of the human ear. Agitation is produced when lighting a match or rubbing hands near the scanning probe. Examples of applications include the checking of all kinds of gas lines, valves, pistons, pressure lines, compressed air brakes, tires, bolted joints, tank systems, steam systems, compressors,

FIGURE 1. Ultrasound leakage detection kit.

FIGURE 2. Gas leakage is detected with an airborne module.

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pneumatic control systems, tools operating from compressed air, sprinkler systems, leaks in exhaust systems etc. or any objects under pressure.

Ultrasound detection devices are used fundamentally for the detection of sound emanating from materials in stressed structures. It is possible to locate very large leaks with the unaided ear. Human hearing, using both ears, is stereophonic. Experience enables the human to react quickly to locate the source of audible noises. In the past, a medical stethoscope has been used as a probe to aid in leak location. Other hearing aids include a simple hose pressed to the ear and directed toward the sound source and a wooden stick pressed against both the ear and sound source. Obviously, with such simple equipment, the sensitivity of detection of leaks is lower and the effect of noisy backgrounds is much greater than with more elaborate leak testing equipment. Little discrimination between significant leak signals and background noises is possible with crude detectors of audio frequency acoustic leak signals. When directional acoustic receivers (transducers) are used, sharper directivity patterns are attainable in the higher frequency ranges. Low frequency sound usually propagates spherically in air (in the absence of absorbers or reflectors). High frequency sound, whose wavelength is short compared to the size of the source, often tends to propagate more nearly in a beam with stronger directional characteristics.

Some types of acoustic leak testing equipment operate in the sonic frequency range. These instruments typically consist only of audio amplifiers that increase the level of the sound without conversion to another frequency. Certain passive leak detectors used to detect leaks in buried pipelines operate in the sonic frequency range because transmission of vibrations in earth materials typically dampens small amplitude, high frequency stress waves very quickly.

Specially designed acoustic probes that amplify any air turbulence help the operator to locate leaks in fluid flow or pressurized systems. They can be used to search for leak locations in components such as valves, regulators, pipes, gaskets and packing, pressure tanks and vessels, mufflers, manifolds, compressors and vacuum systems.

Classification of Fluid Leaks in Terms of Their Acoustic Emission

Leaks may be classified as internal or external to the structure — in either case they are undesired. Leakage may be classified further as acoustically passive or active.

Acoustically Active Leakage

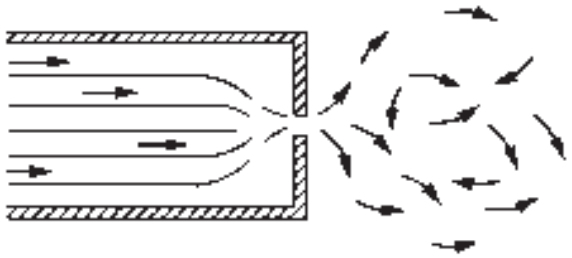
Active leakage emits sounds generated by turbulent leakage. Ultrasonic energy produced by turbulence that occurs in the flow of a fluid provides a detectable and measurable acoustic signal useful for leak location. Such signals can be generated when fluid flows through leaks in systems containing vacuum, liquids or gases. Sonic energy can be produced by the turbulence that occurs in the transition from laminar to turbulent flow of gases. The vibration at ultrasonic frequencies of gas molecules as they escape from an orifice is the source of the leak signals (see Fig. 3). Well known theories of generation of ultrasonic vibrations when fluids pass over solid surfaces and edges are applicable to musical instruments, sirens, whistles, edge tones and various types of sonic power generators used in industry. These are analogous to acoustic noise generated from leaks. The position of a leak is established by locating the source of its sound emission.

Acoustically Passive Leakage

Passive leaks are leaks with the characteristic of a streamline flow known as laminar flow leakage and do not produce acoustic signals related to leakage. Passive leaks can sometimes be detected with an artificial ultrasonic frequency tone generator transmitting signals through the leakage path to an external ultrasound detection probe.

Leak testing depends on the sensitivity and selectivity of the ultrasound detection probe. For example, a leaking heart valve may be defined as an internal leak within the human body. It would be identified as *acoustically active leakage* if the doctor is able to hear the leakage with the mechanic's stethoscope. However, if this leak can be detected only by tracer chemistry and fluoroscopy and not with

FIGURE 3. Turbulence caused by fluid flow through an orifice provides ultrasound signals of leakage.



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the stethoscope, the leakage may be classified as *acoustically passive*. The same terminology may be applied to fluid leaks on or about mechanical structures.

Factors Influencing Acoustic Emission Detection of Leaks

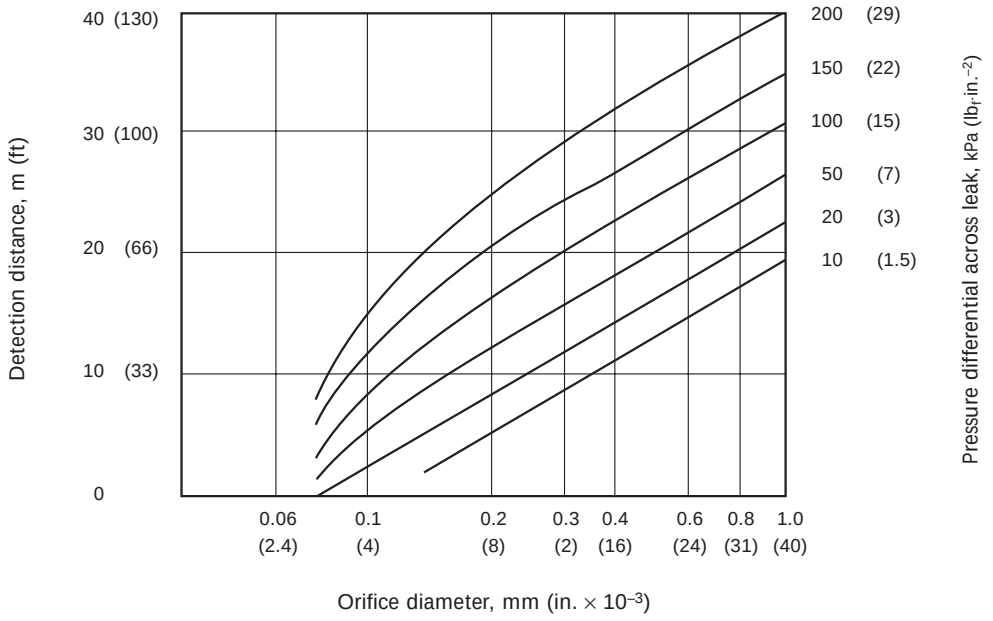
The value of an ultrasonic probe for leak testing depends also on the fluid viscosity, velocity, pressure differential across the leak and the physical size of the leak. Thus, ultrasonic detectability of leakage depends on the following seven parameters: (1) ultrasound detection sensitivity, (2) ultrasonic frequency detection selectivity, (3) acoustic shadowing, (4) viscosity of fluid, (5) velocity of fluid, (6) pressure differential and (7) leak size.

Detection of a leak depends on an ultrasound detection probe's sensitivity and selectivity and to what degree the leak is isolated from the ultrasonic transducers. The single most significant factor to be noted here is the frequency distribution of ultrasonic energy from leakage. All leak spectra possess energy in the 30 to 50 kHz region. At the lower pressures of 480 and 70 kPa (70 and 10 $lb_f \cdot in.^{-2}$), it is seen that there is a distinct maximum around 40 kHz. At the higher pressures (13.8 MPa or 2×10^3 $lb_f \cdot in.^{-2}$), there is a broad energy distribution with the prominent 40 kHz peak showing at the lower flow rate.

A conduit may pass a liquid internally without an apparent leakage of liquid, but a coupling may allow air or any gas present to penetrate into the fluid through a leak orifice in the coupling. Such leaks are frequently described as viscosity dependent leaks. The high velocity, low pressure liquid flow creates a condition that permits a low velocity, high pressure gas to be drawn into the leak orifice. Under these conditions, it is unlikely that an ultrasound detection probe would detect the location of the leak. However, under some conditions, the probe may detect gas entrapped in the liquid from the resulting cavitation or turbulence noise.

The technique of ultrasound detection and locating of leaks is applicable as long as there is sufficient pressure differential acting across the leak to produce the turbulence that generates sonic energy. The only requirement for applicability is that the leakage be large enough, 10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-3} std $cm^3 \cdot s^{-1}$) at 0 °C (32 °F) or higher, to generate noise during turbulent leakage. Ultrasonic signals transmitted by orifice leaks into the surrounding air can be easily detected at distances of more than 30 m (100 ft). There are not restrictions as to the fluid that generates the sound, but it is essential that the fluid flow be turbulent because a laminar flow leak does not generate sound. Figure 4 illustrates typical

FIGURE 4. Airborne ultrasonic probe distances at which orifice leaks can be detected, as a function of orifice size and pressure differentials.



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ultrasound leak testing distances as a function of different pressure levels. Figure 5 shows the orifice conditions necessary to achieve the results shown in Fig. 4.

Contact Acoustic Sensors for Structure Borne Leakage Signals

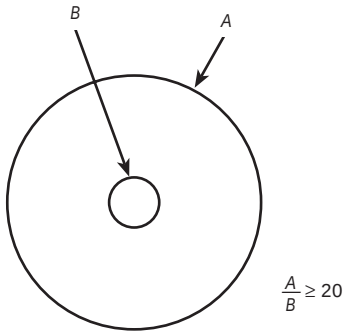
If the leak acoustics generate broad band random noise, the acoustic detection system can be quickly adapted to maximize sensitivity and selectivity to these sounds. Because leak noise generates surface vibrations in the structure where the leak is located, undamped ultrasonic piezoceramic transducers having a strong radial response are particularly sensitive to leakage noise.

In addition, instruments for most acoustic emission test systems have bandpass filters, which tend to have a slight peaking effect at the edges of the bandpass frequency range when driven with random frequency (noise) signals. If the bandpass is set for the radial resonance of the sensor, a very high sensitivity can be obtained for leakage signals. The contact sensor is frequently much more sensitive to leakage noise than are air coupled sensors. Therefore, leaks that may be classified as passive because the air coupled sensor lacks the sensitivity required for their detection may prove to be violently active with a contact sensor.

Coupling Ultrasound Sensors to Structures during Leak Testing

With acoustic emission systems, contact sensors coupled indirectly to structures with ultrasonic wave guides will not have the sensitivity of sensors ultrasonically coupled directly to the subject structure. Ultrasonic coupling means that the sensor face is coated with an oil, short fiber grease, resinous material or an adhesive and pressed into intimate contact or affixed to the structure for the purpose of eliminating an air interface between the sensor face and subject structure. However, a word of caution is in order — some coupling fluids may be somewhat reactive with the test material and cause corrosion or erosion acoustic emission (noise) that may remotely resemble intermittent leakage noise. The fluid causing the corrosion or erosion noise may be either the couplant or the fluid contained by the structure. Such noise is usually detectable by only the most sensitive vibration sensing instrumentation.

FIGURE 5. Orifice conditions necessary to permit ultrasonic leak detection at distances shown in chart of Fig. 4, where *A* is the cross sectional area of approach to the orifice and B is the cross sectional area of the orifice.



Airborne Acoustic Signals for Leak Testing

When structures cannot be monitored by direct coupling of sensors to their surfaces, air coupled or water coupled microphones can be used to detect ultrasonic emissions generated by leakage. Air coupled sensors are convenient but are most usable on active, external leaks. The sensitivity and directivity of remote microphonic detectors can be enhanced by addition of parabolic reflectors. Leaks at high velocity generate broad bandwidth amplitudes that center at frequencies of about 40 kHz (40 000 vibration cycles per second). Structures immersed in (or filled with) liquids are also observed to generate about 40 kHz peak signal amplitudes at the onset of high velocity, low volume leaks. Ultrasound leak detectors are often designed to respond to this 30 to 50 kHz signal frequency range and signals at other random frequencies are suppressed. This reduces interference from machinery or other ambient noise sources.

Artificial Sound Sources for Leak Testing in Large Containers

Large containers can be leak tested without internal pressurization by placing an ultrasonic sound generator in the fluid inside the container. An ultrasound detector is then moved about the outside of the container until a sudden increase in the ultrasonic signal amplitude is observed. Leak testing by this active sonic technique depends on transmission of the ultrasonic waves through the leaking fluid to the external atmosphere, rather than on the surface waves of the enclosing structure.

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Acoustic Detection for Locating High Voltage Electrical Leak Sources

Another use made of ultrasonic leak testing equipment is that of locating sources of radio and television electrical interference, corona discharges and high voltage insulation breakdown and arcing. The ultrasonic energy emitted by these electrical phenomena is similar in its frequency characteristics to the sounds such phenomena create on radiofrequency interference locators or in a portable or car radio. Each of these electrical phenomena is associated with leakage of electricity from bare or insulated conductors and with the resultant local ionization and heating of air or surrounding fluids.

Remote Directional Sensing by Ultrasound Leak Detectors

Because of the directional sensing capability of ultrasound leak testing units used from airborne signals, they can also be aimed to point to the sources of electrical leakage conditions producing acoustic emissions from considerable distances. For example, electric power utility inspectors use ultrasound detection units to scan overhead transmission lines and pole mounted electrical hardware to detect sources of corona or arcing that lead to interference or to indicate dysfunctional equipment or insulation. Similar acoustic detection techniques have been applied to detection of shorted cable pairs within telephone cables.

Instrumentation for Detection and Conversion of Ultrasound Leak Signals

Instrumentation used in various applications of ultrasound leak testing is of similar design. The sensor detects either an acoustic wave traveling over a solid structure or a longitudinal sound wave radiated into a gas or liquid. A preamplifier with a frequency band that extends about from 20 to 300 kHz is used to amplify the received sonic signals. (In some devices, the preamplifier is assembled with the sensor to operate at a distance from the display or loudspeaker unit.) A second amplifier receives the preamplified signal and mixes this amplified signal with the output of a tunable oscillator. Four output frequencies result from mixing: (1) the original ultrasound leak signal frequency, (2) the original oscillator signal frequency, (3) the sum of these two frequencies and (4) the difference between these two original signal frequencies. The audible difference frequency is selected and amplified to drive headphones or a loudspeaker. In this way, the inaudible ultrasound leak signals are converted to sound signals within the range of human hearing (30 Hz to 16 kHz). The originally detected ultrasound leakage signal is amplified to drive a sound intensity indicating instrument because the indicating meter is more sensitive to varying sound levels than is the human ear.

Active and Passive Techniques for Ultrasound Leak Testing

Sonic leak testing techniques may be divided into two classes, active and passive techniques. In the active sonic leak testing technique, sound from an artificial source other than the leak is injected into the fluid contained within the system under test. The leak is then detected because the fluid in the leak conducts sound from within the system to an external ultrasound detector or vice versa. This technique is used when it is not possible to pressurize an item and a leak location is needed.

Two distinct techniques are included in the class of passive ultrasound leak testing: (1) detection of the sonic signal transmitted though air from the test object and (2) detection of the sonic signals by direct contact with the surface of the test object.

Directional Ultrasonic Transducers for Airborne Leakage Signals

Ultrasonic wave transmission modes are different in air and in solid materials, and two different types of detection techniques are needed in leak testing. When the sonic signal is transmitted through air from the leak location to the sonic detector, use is made of a directional airborne signal transducer (with a horn or parabolic reflector) whose signal output is highest when it is pointed toward the source of the noise signal. The short wavelengths of the ultrasonic frequencies make it possible to design highly directional horns that are small in size and convenient to use. A parabolic dish permits a directivity pattern of a fraction of a degree.

Contact Ultrasonic Transducers for Structure Borne Leakage Signals

If the ultrasonic signal is transmitted entirely within metal or solid materials, a

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surface contact ultrasonic probe or detector is used. The vibration sensor is held in direct contact with the part or contact is made through an ultrasonic probe that can conduct the sound vibrations directly from the solid structure to the detector. In most cases involving inspection of pressure boundaries, the detector is moved to scan the test surface, weld seam or mechanical joint. When the characteristic sound of rushing fluid is encountered, the operator moves the detector to maximize the leak signal amplitude. As the ultrasonic probe approaches the leak from several directions, the leak is located when the maximum signal confirms the source of sound.

Acoustic Emission Electronic Analysis Systems for Leak Testing

Electronic acoustic emission nondestructive testing systems are used fundamentally for the detection of sound emanating from solid materials in stressed structures. However, the systems are well suited for leak testing and signature analysis.

The techniques for performing leak testing with acoustic emission test equipment are similar to the technique used with the ultrasonic contact leak test probe. The ultrasonic contact leak detector differs in three ways from acoustic emission test equipment.

1. A contact piezoelectric sensor (transducer) is ultrasonically coupled to the device suspected of having a leak with a waveguide in the ultrasound detection system. In acoustic emission test system, the sensor needs to be in direct contact with the test item.
2. In acoustic emission systems, the oscillator frequency is adjustable, enabling the user to tune in the frequency generated by the leak. Not all ultrasonic contact sensors have frequency tuning capability.
3. In acoustic emission systems, the bandpass is adjustable to allow selection of the frequencies unique to the leakage frequencies but to discriminate against artifact noise sources. Utrasonic contact sensors do not usually have this feature.

The flexibility of adjusting acoustic emission test and monitoring systems for operation in various environments makes these systems very attractive for isolating the particular sound sources of interest to an inspector. With both systems, the detected sound may be further analyzed with a spectrum analyzer to differentiate between the normal machinery noises, i.e., sound emanating from rotating bearings, sleeves etc. and sound resulting from leaks or hydraulic flow noise. Improvements in airborne ultrasonic instruments have aided in eliminating competing background noise.

Specialized Techniques

Liquid Leak Amplification

For extremely low level leaks, ranging from 10^{-6} to 10^{-7} std $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} to 10^{-6} std $cm^3 \cdot s^{-1}$), when minimal turbulence is produced. One manufacturer recommends using a *liquid leak amplifier* as an ultrasonic bubble test. The liquid with a low surface tension is faster and more reliable than classic bubble tests. The bubbles do not have to be seen for leak testing. As bubbles form and collapse they produce strong ultrasonic signals, which are easily detected by the ultrasound detection device. Bubbles form and collapse almost instantly so waiting time for bubbles in low level leaks is markedly reduced.

Ultrasound Detection of Leakage in Immersion Bubble Tests

One manufacturer has inaugurated a semiautomatic ultrasonic signal system for a bubble testing immersion system using a detergent in the water bath. An ultrasonic transducer is suspended above the water bath and the electronic circuitry serves as an acoustic signal amplifier to detect bubble emission by ultrasonic noises. In additional to the rapidity of the test procedure, ultrasound detection of bubble emission offers several advantages. The automatic alarm feature eliminates human judgment and the tedium of attempting to observe bubble formation in water or soap solutions. The system provides a sensitivity to leaks smaller than 2×10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (2×10^{-3} std $^3 \cdot s^{-1}$).

Sonic and Ultrasonic Frequency Ranges of Acoustic Leak Signals

Because many fluid leak acoustic signals are broad banded and include a wide range of frequencies, it is usually possible to detect these signals in either the sonic or the ultrasonic ranges of frequencies. Sonic leak signals are those whose frequencies can be sensed by the human ear. The sonic frequency range is typically described as extending from about 30 Hz

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to about 16 kHz, depending on the sound intensity. Ultrasonic frequency leak signals are those whose frequencies are above the range of human hearing or above about 20 kHz. The range of ultrasonic frequencies extends from 20 kHz to many megahertz.

Cavitation noise and other sounds associated with steady flow of fluids in pipes also include wide ranges of sonic and ultrasonic frequencies. Noise from machinery, when transmitted from some distance, tends to have greater energy in the lower frequency ranges. However, whenever gas escapes through a leak, it does so by means of turbulent flow. When the frequency spectra of turbulent flows are examined, it is found that leak generated acoustic emission frequencies include the range from 30 to 50 kHz. Thus, most ultrasound leak testing instruments are designed selectively to receive acoustic signals within a frequency band near 40 kHz.

Advantages and Limitations of Ultrasound Leak Testing

Versatility

Probably the greatest advantage of ultrasound leak testing is that this technique can be used with any fluid (liquid, gas or vapor) if the physical conditions for sound generation are met in the leak. This versatility eliminates the need for any special tracer gases. When the leak conditions generate sound in ambient air, leaks can be detected at distances up to and beyond 30 m (100 ft). This offers advantages when extended structures are to be inspected. For example, with leaks in overhead air conditioning ducts or pipes carrying gases or liquids, it is often possible to scan the systems from the ground or floor. Particular attention is paid to the welded joints, spiral lock seams and gasketed flanges. Ultrasound detectors can be used effectively for locating leaks in new pipeline installations if pressurization and leak testing are conducted before backfilling the pipeline trench. After the trench is filled, ultrasound leak testing from above ground is more difficult because granular earth materials rapidly attenuate the high frequency mechanical vibrations.

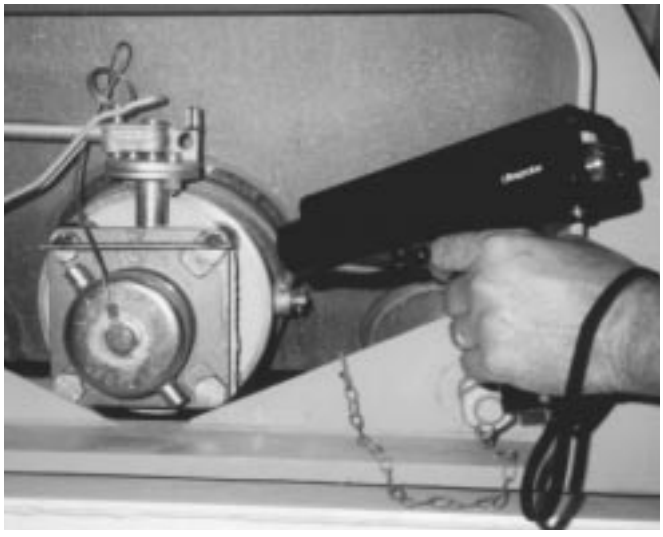
Intrinsic Safety in Hazardous Environments

Figure 6 shows an acoustic emission leak testing system that is field portable and certified as intrinsically safe for use in hazardous environments (Factory Mutual Class I, Division 1, Groups A, B, C, D for the United States and CENELEC EEx ia IIC for Europe).[1,2] It is designed for detection of internal leakage in a variety of valves, including ball, relief, gate, globe and butterfly valves, with sizes ranging from 25 to 450 mm (1.0 to 18.0 in.). With a pressure range of 0.05 to 14 MPa (0.5 to 140 bar), the instrument was developed for gas based systems and can also be used for liquid based systems with operating pressures greater than 300 kPa (3 bar). Data for up to 300 test points are recorded in the internal memory and can be downloaded to a personal computer for analysis and estimation of leak rate and economic impact.

Specialized Technique for Sonic Detection of Laminar Leaks

When the airborne ultrasonic signal directional detector is used, no physical contact is required between the detector and the leaking structure if the fluid is escaping turbulently from the leak into ambient air. If the leak is a laminar flow leak, liquid leak amplifier may be used. Liquid leak amplifier bubbles form and collapse allowing ultrasonic signals to be detected with the scanning module. Bubbles are emitted due to the bubble internal pressure exceeding the sum of the atmospheric pressure above the liquid, the gravitational pressure head of the liquid and the pressure head due to surface tension. It is not necessary for the inspector to visually see the bubbles burst, the ultrasound detector immediately hears the bubbles burst allowing the inspector to identify leak location.

FIGURE 6. Ultrasound translator leak detector for use in hazardous environments.



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On the other hand, if the fluid flows through the leak into another chamber, a contact stethoscope module must be coupled directly to the second chamber. In all situations, leakage through porous material or labyrinthine leaks may be laminar and not possess the turbulence required for the leak to be readily detected ultrasonically (Fig. 7) without liquid leak amplifier. Gas leaks may find paths along screw threads, however, and may be readily detectable at low differential pressures.

Sonic Leak Testing with Ambient or System Noise

Operating difficulties can arise in ultrasound leak testing when the sound from the leak is drowned out by noise in the area where the leak detector is being used. Sometimes the normal flow of fluid through a pipe will provide an acoustic signal input to the instrument, making leak testing impractical unless flow is stopped and the pipeline remains pressurized under static conditions. The ease with which ultrasonic energy is reflected from hard surfaces to produce echoes and multiple sound transmission paths sometimes poses a problem in establishing the exact leak location, if the operator cannot get close to the leak. Some operator experience is required to recognize quickly whether the sensor is intercepting a direct beam or a reflection. Fortunately, the ability to discriminate between direct and reflected waves can be acquired readily.

Sensitivity of Sonic and Ultrasonic Leak Testing

The sonic leak testing technique has a maximum sensitivity of about 10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-3} std $cm^3 \cdot s^{-1}$) at 0 °C (32 °F), but this sensitivity may not always be achieved, for a laminar flow leak does not

FIGURE 7. Example of diffused or labyrinthine leak, which may lack the turbulence conditions required to produce ultrasonic signals, depending upon the fluid viscosity, pressure and other characteristics.

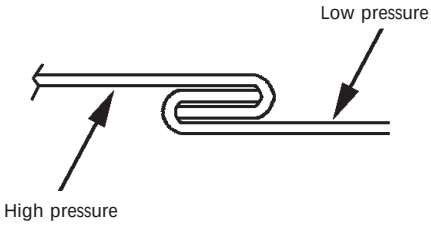
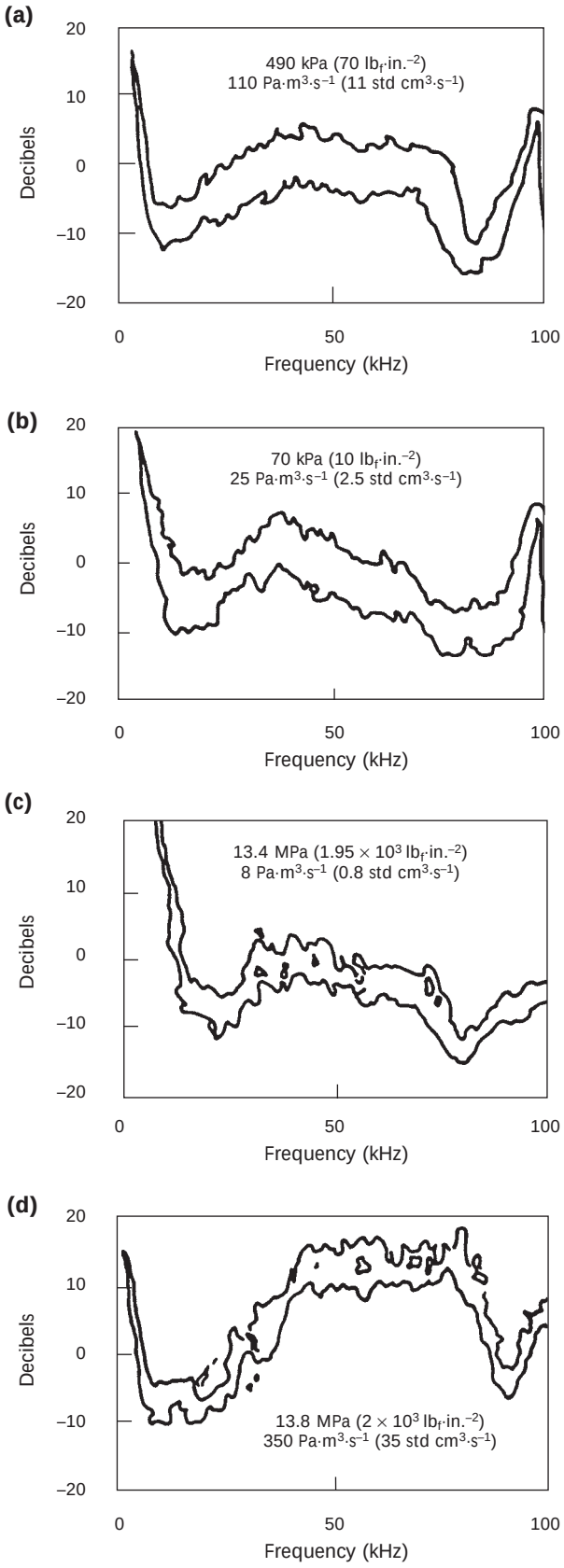


FIGURE 8. Acoustic energy spectra of typical gas leaks. Leakage signals may be single frequency for constant pressure and constant orifice size.



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generate sound. The sonic technique is intended for large leaks only.

Ultrasonic mechanical vibration signal energy is converted to electrical signal energy by an appropriate transducer. By restricting instrument response to the region of maximum ultrasonic energy emission from leaks, it is possible to suppress noise in the audible range that would otherwise mask the weak audible sounds of small leaks. The same conditions are valid for both airborne and conducted sounds.

Detection Distances for Airborne Ultrasound Leak Signals

Orifice leaks are easily detected by airborne signals at distances of up to 30 m (100 ft). Figure 4 illustrates typical ultrasound leak testing distances as a function of orifice size for different pressure levels. Figure 8 shows the sonic energy spectra for several different pressures and leakage rate conditions.

The single most significant factor to be noted here is the frequency distribution of ultrasonic energy from leaks. All leak spectra possess energy in the 30 to 50 kHz region. At the lower pressures of 490 and 70 kPa (70 and 10 $lb_f \cdot in.^{-2}$), it is seen that there is a distinct maximum around 40 kHz. At the higher pressures (13.8 MPa or 2×10^3 $lb_f \cdot in.^{-2}$), there is a broad energy distribution with the prominent 40 kHz peak showing at the lower flow rate.

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PART 2. Instrumentation for Ultrasound Leak Testing

Circuitry and Functions of Typical Ultrasound Leak Testing Instrument

Figure 9 shows the block diagram of electrical circuitry and operational functions for a commercially available ultrasound leak testing instrument. This instrument converts ultrasonic leak signals of 20 to 100 kHz to audible frequency range. To eliminate leakage ambient sounds in the audio frequency range below 15 kHz, the output electrical signal from the detector transducer is subjected to filtration. The airborne ultrasound detector or contact probe receives the high frequency ultrasonic mechanical vibrations generated by leaks and converts these leak signals to high frequency electrical oscillations.

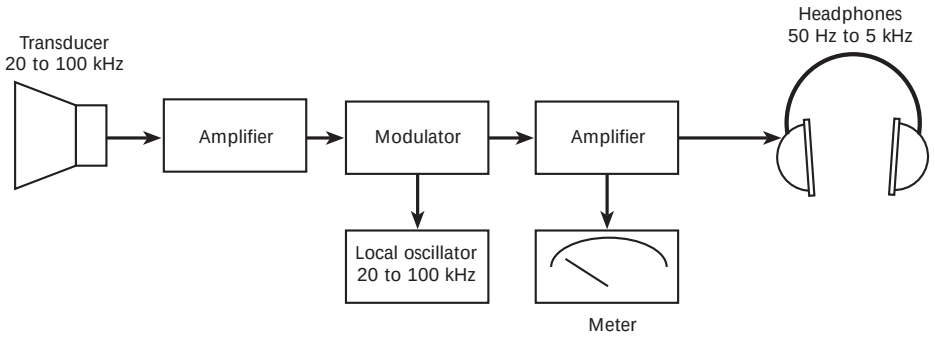
These electrical signals are then amplified at their high ultrasonic frequency range of 36 to 44 kHz, which results in an amplitude modulated signal with a central or carrier frequency near 40 kHz. This stage corresponds to the radio frequency amplifier stage of the conventional amplitude modulated radio receiver. The amplified high frequency input signal is then mixed with a frequency derived from an internal oscillator to provide a different frequency signal in the audio frequency range. The airborne ultrasound detector or contact probe receives the high frequency ultrasonic mechanical vibrations generated by leaks and converts these leak signals to high frequency electrical oscillations. These electrical signals are then amplified and then heterodyned. Finally, the audio frequency signal is amplified and reproduced on a loudspeaker or by headphones. This audible leak signal is interpreted by human listeners as the typical sounds of hissing leaks on vibrating objects. In other words, the ultrasound leak detector is only slightly different from a radio receiver, in that the original frequency ranges is ultrasonic (near 40 kHz) rather than in the conventional amplitude modulated broadcast frequency range (0.55 to 1.4 MHz). From these audible signals it is possible to analyze the amplitude and characteristics of the ultrasonic signals that are received from leaks.

Inspection Modules

Two distinct inspection techniques of detecting passive ultrasound leaks are: (1) ultrasonic scanning probe is used for detection of the sonic signal transmitted though air from the test object and (2) ultrasound detection contact stethoscope probe used in the detection of the sonic signals by direct contact with the surface of the test object. The ultrasound detection scanning probe can be used as an ideal system with which to do leak testing and signature analysis.

The techniques for performing leak testing with an ultrasound detection scanning probe are similar to the technique used with the ultrasound detection contact (stethoscope) leak test probe. In addition, improved scanning probes have adjustable frequency controls enabling the user to tune in the frequency generated by the leak. The bandpass is adjustable to allow selection of the frequencies unique to the leakage

FIGURE 9. Block diagram of an ultrasound leak detector.



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frequencies, but to discriminate against artifact noise sources. The contact probe differs from an ultrasound detection scanning probe in which the contact piezoelectric sensor (transducer) is mechanically coupled to the device suspected of having a leak.

The flexibility of adjusting ultrasound detection systems for operation in various environments makes these systems very attractive for isolating the particular sound sources of interest to an inspector. In addition, the detected sound may be further analyzed with a spectrum analyzer to differentiate between the normal machinery noises, i.e., sound emanating from rotating bearings, sleeves etc. and sound resulting from leaks or hydraulic flow noise. Improved airborne ultrasonic instruments have aided in eliminating competing background noise.

Scanning Module for Airborne Leakage Signals

Ultrasonic wave transmission modes are different in air and in solid materials, so two different types of detection techniques are needed in leak testing. Although air coupled sensors are certainly convenient and fast for the detection of many leaks, they are most usable on active, external leaks. When the sonic signal is transmitted through air from the leak location use is made of a directional airborne signal transducer with a scanning module. The scanning module is characterized by its directional sensitivity; therefore, unwanted noise outside its acceptance angle tends to be suppressed.

The scanning module contains amplification circuitry for remote sensing. The ultrasound detector is basically an ultrasonic super heterodyned receiver amplifying frequencies in the range of 20 to 100 kHz and mixing them with an internal ultrasonic frequency. The scanning module for airborne leak signals of Figure 10 has a conical directivity pattern with a 60 degree included angle (at the ±3 dB or half power points) for sensing leak locations from ultrasonic waves transmitted through the atmosphere. Leaks at high velocity generate broad bandwidth acoustic signals. Airborne leak sounds have peak amplitudes that center at frequencies of about 40 kHz (4×10^4 vibration cycles per second). Structures immersed in (or filled with) liquids are also observed to generate about 40 kHz peak signal amplitudes at the onset of high velocity, low volume leaks. Ultrasound leak detectors are often designed to respond to this 40 kHz signal frequency range; signals at other random frequencies are suppressed. The resulting intermediate frequency has a frequency spectrum with the characteristics of the ultrasonic signal picked up by the probe. For this reason the characteristic noise of a gas leak, for example, is preserved. Selected frequency capability reduces interference from machinery or other ambient noise sources.

Response of the ultrasound detection probe is highest when the probe is pointed toward the source of the noise signal. The short wavelengths of the ultrasonic frequencies make it possible to design highly directional probes that are small in size and convenient to use. Available accessories include a parabolic dish ultrasonic wave reflector (Fig. 11). A parabolic dish permits a directivity pattern of a fraction of a degree. It can be attached to this probe for enhanced convenience in location of leaks on overhead structures from the ground using parabolic reflectors.

Stethoscope Probe for Structure Borne Leakage

Airborne ultrasound leak testing test techniques can also be applied when fluid leaks generate sound transmitted through solid material and structures such as walls and supports of the system under test. Such sonic disturbances often cause the walls of the leaking structure to vibrate in

FIGURE 10. Scanning module for detection of airborne ultrasound.

FIGURE 11. Parabolic detection module.

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random Lamb wave modes. Ultrasonic transducers, in contact with the surface of the structure, are sensitive to turbulent leakage through the leak orifice for pressure leaks down to about 25 µm (0.001 in.) in diameter at 40 kPa (6 $lb_f \cdot in.^{-2}$ gage) pressure. For good coupling, the structure must be mechanically solid and have no intervening acoustic impedance discontinuities whose impedance mismatch could cause total signal reflection, since it is the structure (rather than the fluid) that carries the sound of the leak to the acoustic sensor.

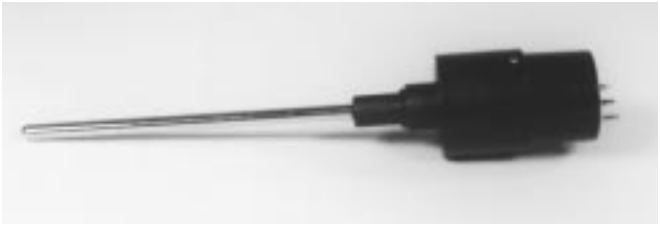
The ultrasonic contact probe of Fig. 12 is a mechanical contact vibration detector used to detect ultrasonic noise originating inside a mechanism or enclosing structure. Metallic friction and rubbing metals parts generate ultrasonic noise. The contact probe contains very sensitive components and thus should be protected from any hard blows. The pointed end of this probe is pressed firmly against the object to be measured to ensure good sound transmission. The highest sensitivity is obtained when pressing the probe perpendicular to the surface of the object that transmits ultrasound from leaks or rubbing surfaces.

The contact probe is also used for the location of internal leaks in hydraulic systems. The ultrasonic energy produced by the fluid flow is conducted through the walls of the valves, tubing and other components. This makes it possible to detect leak flows and changes in flow conditions that are otherwise difficult to locate without disconnecting lines and partially disassembling the system under test. For example, leaks in water pipes buried in structural concrete walls and floors can often be detected acoustically without damage to the structures. Acoustic emission sensors are placed in contact and coupled with the concrete floor until a peak signal amplitude is recorded from the leaking pipe.

In acoustic emission systems, contact sensors (transducers) coupled indirectly to structures with ultrasonic wave guides will not have the sensitivity of transducers ultrasonically coupled directly to the subject structure. Ultrasonic coupling means that the transducer face is coated with an oil, short fiber grease, resinous material or an adhesive and pressed into intimate contact or affixed to the structure for the purpose of eliminated an air interface between the transducer face and subject structure.

However, caution is in order. Some coupling fluids may be somewhat reactive with the test material and cause corrosion or erosion acoustic emission (noise) that may remotely resemble intermittent leak noise. The fluid causing the corrosion or erosion noise may be either the couplant or the fluid contained by the structure. Such noise is usually detectable by only the most sensitive ultrasound detection device.

FIGURE 12. Structure borne detection module.



Construction of Contact Stethoscope Module

The 140 mm (5.5 in.) long probe of the contact stethoscope module features a plug-in type, insulated probe with radio frequency shielding. The probe tip is conical for uniform surface contact. The stethoscope module detects wavelengths of 20 to 100 kHz from the acoustic signal. The contact stethoscope module stylus responds to the mechanical vibrations conducted from the ultrasonic source through the structure. This mechanical energy is in turn conducted through the stylus to a piezoelectric crystal within the probe housing. The crystal transducer converts the mechanical energy into an electrical signal. Solid state circuitry within the probe amplifies the signal for introduction to the ultrasonic translation electronics within the main instrument. A three piece segmented metal rod lets the operator increase the stethoscope contact range to 500 mm (20 in.) and 760 mm (30 in.).

Rubber Focusing Module

A rubber focusing module can be slipped over the scanning or stethoscope module (Fig. 13), which reduces its opening to about 8 mm (0.3 in.) in diameter. Thus, ultrasonic noise outside the sensitive area is reduced and the directional sensitivity of the scanning module is enhanced. A noise source has to be located exactly within the beam to be detected. The scanning module is used alone for gross detection of ultrasonic sources. The rubber focusing module pinpoints the exact spot from which the noise originates.

Parabolic Microphone

A parabolic microphone doubles the detection distance obtainable with a conventional scanning module while narrowing the sound beam. The parabolic microphone has less than a five degree

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beam spread compared to the scanning module with a beam spread of about 60 degrees. Unique features of the parabolic microphone let an inspector focus on a target up to 30 m (100 ft) away.

Artificial Ultrasonic Frequency Tone Generator

An artificial ultrasonic frequency tone generator can be used when leakage fails to generate ultrasonic signals. The tone generator provides a conical sound beam. Place the tone generator anywhere within a hollow test object because the sound is present practically throughout the enclosed volume. For example, the tone generator of Fig. 14 emits a frequency modulated signal in the range of 36 to 44 kHz. The heterodyned frequency is in the audio frequency range; this signal is responsible for the sound heard when the device is switched on. The ultrasound generated by the tone generator passes through small openings, cracks and leaks. It can be detected by the ultrasound detection probe on the opposite side of the object. However, for detecting extremely small leaks, the tone generator output beam should be aimed at the ultrasound detection probe. This may be achieved by keeping the tone generator and ultrasound detection probe simultaneously in the direct line with each other while the pair are scanning along opposite sides of a welded seam. Solid material reflects the 36 to 44 kHz airborne ultrasonic signal and is not received by the ultrasound detection probe. The search for leaks with the tone generator is based on both of these facts.

FIGURE 13. Rubber focusing attachment.

Applications Using an Artificial Ultrasonic Frequency Tone Generator

Airborne ultrasound emitted by an artificial ultrasonic frequency tone generator is detected using a ultrasound detection probe. This procedure allows detection of porosity ruptures and other leaks in containers and objects that may not be subjected to pressure. The tone generator is switched on and placed outside or inside of the object under test. The ultrasound detection probe can now be used on the other side of the containment wall to detect any possible leaks.

It is important for the operator to keep in mind the fact that ultrasonic frequency is reflected by hard surfaces and could be picked up by the ultrasound detection probe indirectly. To prevent false measurements, all natural openings have to be sealed before the test. Checking for leaks of windshields, window seals on buildings and vehicles, door seals, trunk seals, pressure tanks and storage bins, hulls, switching cabinets, walls, ceilings, heated pipes, refrigerator door seals and ventilation ducts are all applications using an artificial ultrasonic frequency tone generator.

FIGURE 14. Ultrasonic tone generator.

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Types of Small Portable Ultrasound Leak Detectors

Several other sources provide portable ultrasound leak detectors with a variety of capabilities. For example, one manufacturer provides the following versions: (1) hand held, gun shaped sound probe, with earphones (Fig.15) and sound level meter; (2) model designed for the telephone industry for detecting leaks in pressurized cables to the exclusion of wind and vehicular traffic noise etc.; (3) models for vacuum and pressure leak testing; (4) contact model for acoustically determining mechanical malfunctions such as worn shafts, bearings, engine valves, gears and hydraulic systems; (5) model for spotting electrical leaks in power transmission systems; and (6) tone generator to assist in locating leaks in nonpressurized areas such as the cold box of refrigerators.

All of these units either detect or generate frequencies in the ultrasonic range.

Control Adjustments of Ultrasound Leak Detector Instrumentation

Various configurations of ultrasound leak detectors are available for incorporation in numerous industrial systems and applications. The adjustable signal gate and alarm lengths can be preset for high speed production leak tests such as on aerosol packages or pressure valves. A typical example of a quality assurance program is the inspection of volume production, two way relief valves certified for opening at pressures as low as 3.5 kPa (0.5 $lb_f \cdot in.^{-2}$ gage). A built-in signal attenuator gives the instruments the flexibility to perform a variety of quality assurance tests. The signal required to trigger the alarm can be set to any level in an 85 dB range above inherent noise with fine attenuation continuously adjustable over a 20 dB range. Examples of the uses of this feature are the inspection of ultrahigh pressure hydraulic and pneumatic systems in which a photographic record of the intensity meter during pressure testing constitutes adequate documentation of system integrity. Another example is pressure testing of plastic fabricated enclosures to various specifications.

FIGURE 15. Leak detection in a gas distribution system.

Sensitivity Control

The sonic leak testing technique has a maximum sensitivity of about 10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-3} $std \cdot cm^3 \cdot s^{-1}$). This sensitivity may not always be achieved, for a laminar flow leak does not generate sound.

A ten-turn sensitivity adjustment control dial covers a wide range of ultrasonic signal reception so that users may detect subtle mechanical and leak problems as well as focus in on gross signals for accurate analysis.

Frequency Control

The frequency tuning control allows the operator to select the specific frequency of a problem sound while reducing interference from competing ultrasonic signals. Because many fluid leak acoustic signals are broad banded and include a wide range of frequencies, it is usually possible to detect these signals in either the sonic or the ultrasonic ranges of frequencies. Sonic leak signals are those whose frequencies can be sensed by the human ear. The sonic frequency range is typically described as extending from about 30 Hz to about 16 kHz, depending on the sound intensity. Ultrasonic frequency leak signals are those whose frequencies are above the range of human hearing or above about 20 kHz. By tuning from 20 to 100 kHz, an operator is able to easily recognize leakage in gas and liquid systems when the ultrasound detection device is able to heterodyne the frequencies to 100 Hz to 3 kHz audio. In addition, information regarding wear patterns in operating equipment can be noted by plotting changes in selected frequencies.

Cavitation noise and other sounds associated with steady flow of fluids in pipes also include wide ranges of sonic and ultrasonic frequencies. Noise from machinery, when transmitted from some distance, tends to have greater energy in the lower frequency ranges. However, whenever gas escapes through a leak, it does so by means of turbulent flow. When the frequency spectra of turbulent flows are examined, it is found that leakage generated acoustic emission frequencies include the range from 20 to 100 kHz

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Calibration

Under certain circumstances the industrial user of ultrasound leak testing instruments may not have the advantage of a formal demonstration before initial use of the equipment. If this is the case, it is recommended that a typical leak be simulated to enable the operator to get the *feel* of the instrument. It is desirable to have a means of adjusting both the size of the leak and the internal pressure. A few minutes spent in this manner will assist the user in learning what to anticipate with the equipment in actual leak testing. Tests with artificial sources of ultrasonic signals can be useful training aids.

A calibrator for the sonic leak detector may be a small plastic squeeze bottle. When this bottle is quickly squeezed and released, the air passing though its nozzle generates an airborne ultrasound test noise signal. This artificial leak signal can be detected by the microphone probe of Fig. 10 at distances of up to 20 m (65 ft). Although this device will not provide a quantitative calibration, it enables the operator to determine if the airborne sonic leak testing instrument is qualified for leak location. If this signal produces no indication on the leak detector meter, the battery may need replacement (or major instrument service is required). This provides an easy way to find out whether the device is switched on or off. The jingling of keys and coins is often used for the same purpose.

A qualitative calibrated reference standard can be fabricated by using a pressurized clean and dry air source, pressure regulator, flow indicator and orifice nozzle. Details for the fabrication of this calibrated reference standard can be located in ASTM E 1002[3] as well as the calibration procedure.

Calibration of Ultrasound Detector with Tone Generator

The tone generator produces an ultrasonic signal when switched on. This signal should be detected without any difficulties by the ultrasound detector probe over a distance in air of about 15 m (50 ft). Failure to meet this test implies the need for a new battery or major instrument service. An ideal calibrating source is obtained by putting the tone generator within a pipe with an internal diameter of 100 mm (4 in.). This pipe should have a hermetically sealed bottom and a height of about 500 mm (20 in.). The sound opening of the tone generator has to point towards the top opening of the pipe. A sliding cover placed over the top opening of the pipe is provided with a small hole perhaps 0.5 mm (0.02 in.) in diameter. The tone generator (equipped with a fresh battery) is then switched on.

During calibration, the tone generator is pressed directly onto the hole in the pipe cover. The ultrasound detection probe can then be preset to provide a certain instrument deflection by adjusting the volume control knob. This reference calibration setup should be maintained and its conditions recorded to be able to reproduce the calibration later. This reference calibration technique can be used to repeat leakage measurements on a periodic schedule to detect wear, aging or other causes of leaks.

Qualification of Airborne Ultrasound Testing Personnel

The effectiveness of airborne ultrasound testing depends on the capabilities of the personnel who are responsible for and perform testing. Airborne ultrasound can be performed by personnel who are not formally trained, much as visual testing is performed daily by personnel not formally trained in visual testing. However, industry requirements that focus on qualification and certification have changed the way the world does business. Airborne ultrasound testing personnel should be qualified to perform inspections. This not only gives personnel the knowledge and understanding of the test technique but also gives the inspector the confidence needed to perform the inspection to the highest level of success.

Personnel being considered for qualification and certification should complete sufficient organized training to become thoroughly familiar with the principles and practices of airborne ultrasound related to the level of certification desired and applicable to the processes to be used. The training should include sufficient examinations to ensure that the necessary information has been comprehended. Airborne ultrasound training should consist of sufficient references and technical source material. The employer who purchases outside training services is responsible for ensuring that such services are in accordance with governing requirements.

Interpretation of Airborne Ultrasound Leak Signals

Ultrasonic sound waves can be generated by the interaction of higher velocity air or gas molecules with the atmosphere. Pressurized systems leaking gas under turbulent conditions provide the types of acoustic signals to which directional probes can respond from various

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distances. Hissing sounds are used to pinpoint leaks in pressure or vacuum systems. Pulsed or rhythmic sounds indicate mechanical malfunctions such as faulty valves or gaskets. Frying sounds characterize electrical leakage phenomena such as corona causing radio interference or high voltage (arc or spark) insulation breakdowns in anomalous bushings or cables.

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PART 3. Ultrasound Leak Testing of Pressurized Industrial and Transportation Systems

Locating High Pressure Leaks by Ultrasound Leak Testing

There are compelling reasons for maintaining the integrity of pressure distribution systems in industrial plants, power plants and transportation systems. Of course, leakage in poisonous, noxious or explosive gas systems must be immediately located and stopped. Ultrasound leak testing units have been used with considerable success for several years in the maintenance of a wide variety of gas pressure systems. The following discussion contains general recommendations for ultrasound leak testing in high pressure systems and some suggestions for specific applications.

Technique for Ultrasound Detectors for Pressure Leaks

For detecting leaks from pressurized industrial systems during operations, the airborne ultrasound signal detector's volume control should be set at maximum range for initial scanning of an area for leaks. Reducing the volume control setting sharpens the response cone of the detector and minimizes interference from large adjacent leaks. A preliminary test should be conducted before inspection. The inspector can simply point the scanning module toward the inspector's face and breathe in and out. The ultrasound detector will detect the sound immediately.

Before entering an area such as a factory, the inspector should stand at the entrance and scan the entire area for an indication of leaks; otherwise, the technician may follow an apparent reflection from a leak behind the technician's back. Large overhead leaks often create sonic reflections that give a false indication of a leak. The leak source is always louder than its reflections. Always verify leakage by eliminating the possibility of sonic reflections.

Procedure for Locating Large Leaks with Ultrasound Leak Detectors

In facilities such as refineries, chemical plants and locomotive repair shops, large steam and compressed air leaks are often encountered. These leaks can create an extremely high ultrasonic background noise level, which tends to limit the effectiveness of the detector. Proper airborne ultrasound technique can eliminate or minimize the effects of competing ultrasound. It is necessary to adjust the volume control to minimum and hold the probe close to the object under test. In severe situations, it is important to use the rubber focusing extension to mask the probe from such high level acoustic energy. It is usually advisable for the inspector to wear sound insulating earphones in all areas where there are audible hissing sounds such as those omnipresent at refineries, chemical plants etc. One manufacturer has incorporated hearing protection in their standard equipment so that the inspector can concentrate on ultrasound and audible sounds are minimized.

The operator also must be aware of the location of pneumatic controls and valves that have intentional bleed systems. To minimize the effect of large leaks and reflections, field practices have proved the advantage of approaching each suspected leak area from another direction. When a multiple leak situation if found, it is generally helpful to seal major leaks temporarily with tape or clamps and then to continue to search for the remaining leaks. All significant leaks should be marked, reported and brought to the attention of those responsible for replacement or repair of leaking parts.

Leaks in Exotic Gas Supply Systems

Manufacturers producing sophisticated electronic components have used ultrasound leak testing to aid in maintaining exotic gas supply and manifold systems with minimum disruption of critical production processes. The time required to inspect the integrity of newly installed exotic gas manifolds was cut 80 percent by a portable

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ultrasound detector during reconstruction of the gas distribution system throughout a 3200 m^2 (35 000 ft^2) two-story facility. Reorganization of the plant's argon, forming gas, hydrogen, nitrogen and oxygen lines had to be done with minimal curtailment of production operations. Supply system gage pressures ranged from 750 kPa (110 $lb_f \cdot in.^{-2}$) down to 100 kPa (15 $lb_f \cdot in.^{-2}$) for exotic gases and 20 kPa (3 $lb_f \cdot in.^{-2}$) at service drops. The more than 600 m (2000 ft) of multiple line distribution manifolds are located with all other utilities in a 1.5 m (5 ft) tall loft between the first and second floors. To inspect this gas pressure system, the inspector merely aims the probe along the course of pipes or tubing (Fig. 16), paying particular attention to known trouble sources such as valves, regulators and joints. When the detection unit's hissing sound is loudest, the precise leak location is determined and marked for repair or replacement.

It is estimated that the ultrasound leak location system is at least five times faster than the previously used bubble test. Limited access and poor visibility often limit observation for bubble leak tests. Ultrasound leak testing does not require the thorough cleaning that must precede bubble tests. With ultrasound leak testing, complete gas systems leak testing can be scheduled, a task that was previously nearly impossible in large gas systems. Before the ultrasound leak detector, maintenance of the gas system was done only during plantwide shutdown.

Advantages of Ultrasound Leak Testing during Purging of Gas from Systems to Be Repaired

At best, hot work on flammable gas pressure networks is necessarily time consuming and disruptive. In this area, ultrasound leak testing has provided a faster inspection technique. For example, the welding of a new valve for a plant revision still requires purging the flammable gas with nitrogen. While the welder is standing by, the nitrogen purge gas itself serves as a testing medium for ultrasound leak testing. This eliminates the delay required for hydrostatic weldment tests.

FIGURE 16. Typical acoustic emission leak testing instrument.

Pinpointing Gas Leaks beneath Insulation

Whereas the gas leaking from an insulated pressure conduit may seep underneath the insulation for 30 m (100 ft) or more before making its presence detectable in the atmosphere, the leak's precise location emits ultrasonic energy, which is usually detectable through the insulation. Industrial plants using this detection technique report substantial savings in the cost of insulating materials such as magnesia compounds as well as labor cost to reinsulate extensive conduit lengths.

High pressure air has been used successfully as a testing medium instead of conventional hydrostatic tests. Examples are inspecting shipboard plumbing and other industrial plant plumbing networks. Repairs can be effected immediately and rechecked ultrasonically, which compares favorably with the steps required with hydrostatic testing.

Process Piping and Equipment

Both contact and air coupled ultrasound leak detectors and acoustic emission leak test systems are being used in electric generating plants to locate leaks in piping, ductwork and pressure vessels where air is used as a testing medium. Testing of the various powerhouse systems can become quite involved and expensive if conventional tests such as air pressurization and bubble tests or hydrostatic tests are used. Scaffolds or ladders must be used to gain access to high places for bubble testing of welded joints when air is used for pressurizing the system. When water is used, the leaks must be located and the system drained for repairs.

The building heating system piping in an electric power plant, which for the most part is located near the ceiling and at other rather inaccessible locations, may be pressurized with air to about 700 kPa (100 $lb_f \cdot in.^{-2}$ gage) and tested using an ultrasound leak detector. Another piping system tested with an ultrasonic leak detector is the fuel oil system. This system was also tested for leaks using air at

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700 kPa (100 $lb_f \cdot in.^{-2}$ gage). The fuel oil piping between large storage tanks and the powerhouse can also be tested for leakage.

The leak detector is also used for testing the boiler internals and welded tubing panel walls and roof. The boiler consists of two identical furnaces. Each furnace enclosure is made up of welded tube panels joined into a structure forming a rectangular, upright box 19 m wide × 9 m deep × 18 m high (62 ft × 29 ft × 59 ft). The boiler pressure parts are pressurized with air to about 700 kPa (100 $lb_f \cdot in.^{-2}$ gage) and the furnace enclosure is pressurized to about 5 kPa (0.75 $lb_f \cdot in.^{-2}$ gage) for testing for leakage.

Other items tested for air leaks by the ultrasound leak detector are the condensers. The condenser housings can contain over 72 000 tubes, measuring 22 mm (0.88 in.) in diameter, that are rolled and expanded into the tube sheets. These sections and the condenser housing are tested for leakage using ultrasound leak detectors. Because these operate under a vacuum, this provides an excellent source for leak testing. The ultrasonic tone generators can be used when it is not possible to draw a vacuum or when thin wall tubes need to be located.

Instrumentation Air Systems

Ultrasound is used in both "as needed" and "scheduled" inspections of control instrumentation, process regulation, air and logic control systems. If a lag in control actuation is noted, the air lines and valves are checked and leaks in plastic covered tubing and internal valve bypassing are readily detectable. One plant that conducts an annual inspection of several thousand kilometers of air instrumentation networks reports that one person completes the tests during the 10 day long shutdown. Before ultrasound leak testing, it took 14 people to perform equivalent inspections.

Leaks in Air Conditioning Ducts

Reports from air conditioning contractors indicate reduction in contractor checkout time as great as 80 percent by using ultrasound detection to inspect the integrity of high pressure duct work. Generally the technique is as follows. Using a low volume blower, a section of ducting is pressurized until the manometer water column reaches 380 mm H_2O (3.8 kPa or 0.5 $lb_f \cdot in.^{-2}$ gage). A manometer coupled to the test gage and to another test point in the ducting is calibrated. The actual leakage rate is compared to permissible air leakage rates. When the leakage exceeds the permissible rate, the ultrasound test begins immediately. To inspect the ducting, which ranges from 100 mm to 1.2 m (4 in. to 4 ft) in girth, the test operator merely walks at floor level and aims the probe along the course of the overhead ducting. Particular attention is paid to welded joints, to spiral lock seams in which gaps in the sealant may exist and to gasketed companion flanges.

Locating Leaks in Larger Flanges

It is still common in many facilities such as refineries, either on construction checkout or on resuming onstream production after shutdown, to seal large flanges with masking tape and then to puncture the masking tape to insert a hydrocarbon detection probe or to apply film solution to a puncture in the masking tape. Bubble testing can reveal only that a leak exists, which in the case of a 1.5 m (60 in.) diameter flange is of little help to the repairman. A number of refineries have eliminated entirely the time and cost of masking flanges by pinpointing the location of such leaks ultrasonically.

Pressurized Fermentation Systems

The practice of inspecting the integrity of critical air pressure levels in biological fermentation systems provides an example of the economics of ultrasound detection. One company reports that ultrasound detection conducted during the start of a culture growth cycle effects an 80 percent reduction in the time over the previous technique, which required a technician to keep a continuous watch over pressure indicators on the culture tanks and associated plumbing networks.

Hydrogen Cooled Electric Power Generator

A major electric power utility company uses ultrasound detectors to detect and pinpoint leaks in hydrogen cooled generator casings and hydrogen supply systems. The ultrasonic system has been adopted in addition to the customary detector probe and bubble testing techniques. Acceptance standards set by electric utility and manufacturer require that the generator casings maintain 415 kPa (60 $lb_f \cdot in.^{-2}$ gage) air pressure for 24 h with no more than 0.55 m^3 (20 ft^3) loss from 48 m^3 (1690 ft^3) volume casings. Normal pressurization with hydrogen during operation is at 310 kPa (45 $lb_f \cdot in.^{-2}$ gage). Maintenance procedures at the electric plant also call for ultrasound tests in the event the hydrogen system pressure gages indicate a leak. Speed in locating and repairing leaks is essential. Plant safety and the cost of leaking hydrogen are critical considerations. The same

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procedure for ultrasound leak testing is followed for both new equipment checkout by construction engineers and maintenance by plant engineers.

With the airborne ultrasonic signal detector, the engineer guides the probe along the surface of flanges and seams of the generator casing and along valves, couplings and flanges of the hydrogen supply piping. When the detector emits a hissing sound, identical to the familiar sound of a punctured inner tube, the engineer determines the precise leak location by aiming the probe in the direction of the sound's greatest intensity. The device does not respond to audible sonic energy, so the ambient hissing noises ever present in a power station do not affect the operation. Similarly, the engineer can also discriminate against ambient ultrasonic sounds by noting their direction.

The detector's responsiveness to leaks depends of course, on the size and shape of the leak orifice and the pressure and type of the gas. With 415 kPa (60 $lb_f \cdot in.^{-2}$ gage) pressure air used in construction inspections, the detector can pinpoint a leak smaller than 0.1 mm (0.004 in.) diameter located 3 m (10 ft) from the probe. With 310 kPa (45 $lb_f \cdot in.^{-2}$ gage), this leak could be detected from 2.5 m (8 ft) distance. The ultrasound leak testing system is faster and, especially in three-story piping assemblies feeding hydrogen to generators, far easier to use than bubble testing.

Petroleum Refinery Equipment

With earlier refinery inspection techniques, leaks were not detectable until the process went onstream. When leaks were present, this could delay resumption of production. Flanges were covered with a masking tape to accumulate any leaking hydrocarbon vapors. As the system went onstream, the masking tape was punctured to insert a hydrocarbon probe. Hydrocarbon leak testing, under the supervision of the fire department, detected the presence of gas but not the precise location of the leak. Knowing the location of a leak in a 1.2 m (4 ft) diameter flange is almost as important as knowing it exists, especially when time is important.

Detecting leaks in pressure and vacuum systems by ultrasound is now standard plant inspection procedure. The most intensive use occurs during and immediately following shutdowns for catalyst regeneration or repairs. During the shutdown, an entire hydrogen vessel and piping, such as a 3200 m^3 (20 000 barrel) per day isocracker, is actually ultrasonically surveyed three times. The majority of small leaks and all gross leaks are located before going onstream, either during evacuation or during purging with nitrogen. The inspector merely scans the course of the vessel and piping with the probe, concentrating of course, on flanges. When leaks are pinpointed the maintenance crew takes immediate action.

A final ultrasonic check for minuscule leaks that appear under high pressures is now made as the process goes onstream. With pressures generally exceeding 3.4 MPa (500 $lb_f \cdot in.^{-2}$), the detector can pinpoint a leak 0.02 mm (0.001 in.) in diameter at distances more than 1.5 m (5 ft) from the probe. The device also has been used successfully to locate air leaks in a vapor recovery system, both to improve efficiency and to eliminate leakage of exhaust pollutants.

Compressors in Chemical Plants

An ultrasound detector may permit a chemical plant to reduce downtime on a compressor from 32 to about 16 h per month. Time is saved because a particular valve, valve packing or part that is causing trouble can be located immediately without a full teardown and internal inspection of each part. Formerly, it took about 8 h to tear down the machine to find which valve or part was not operating properly. Because the process is continuous, the decreased downtime permitted by the ultrasound leak detector saves a sizable amount of money.

The two-stage compressor is used as part of a recovery system for hydrocarbons. Inlet suction pressure is about 350 kPa (50 $lb_f \cdot in.^{-2}$ gage) and discharge pressure is 3.3 MPa (480 $lb_f \cdot in.^{-2}$ gage). The stream has a considerable amount of sticky material or foreign matter that causes trouble with valves and rod packing. When operating, the machinery creates both audible and ultrasonic sounds. Incipient leakage generates ultrasonic sounds analogous to later audible sounds created by leaks so bad that possible breakdown may be imminent. When translated to audible frequencies, these ultrasonic emissions are immediately recognizable by experienced maintenance personnel who can easily discern between normal and malfunctioning machines. Thus, the compressor can be inspected internally during operation with the ultrasound detector.

In an infrared analyzer room where toxic material is handled at 200 to 2100 kPa (30 to 300 $lb_f \cdot in.^{-2}$ gage) in 6 mm (0.25 in.) stainless tubing, the leak detector is used about once a week. With the bubble test, this weekly test took about 8 h; it can be performed in 1 h by the ultrasonic technique.

The ultrasonic unit prevents losses of nitrogen in a system handling the gas at

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2.1 to 4.2 MPa (300 to 600 $lb_f \cdot in.^{-2}$ gage). Leaks that could have been overlooked with bubble testing are found quickly with the ultrasonic procedure. The ultrasound leak detector is also used for testing control valves and pump bearings.

Pipelines

It is virtually impossible to precalculate acoustic transmission efficiency, signal attenuation and environmental noise levels for large structures and pipelines because of the large number of unknown parameters involved. However, acoustic signal attenuation is often similar for similar types of structures. In this case, it is possible to make reliable estimates of the attenuation of signals in test components by correlation with past experience with similar components that have been instrumented for leakage monitoring. This similarity often holds true for typical pressure vessels, tanks and other industrial process control system components. However, pipelines are an exception to this general approximation rule based on the assumption of similar acoustic attenuation for similar components.

Signal Transmission Characteristics of Pipelines

The attenuation of acoustic emission signals of turbulent leakage in pipelines is influenced by the following factors: (1) pipe dimensions; (2) types of pipe welds and welded attachments; (3) acoustic properties of fluids transported through the pipeline; and (4) materials surrounding and in contact with the outside surface of the pipeline, including insulation, earth and water.

Although extensive measurements have been made of the acoustic attenuation characteristics for specific pipelines, there are still no reliable empirical correlations available from which to make predictions. Consequently, to predetermine appropriate spacing along a pipeline for leakage acoustic emission sensors, it is necessary to perform tests on a short length of the pipe to determine its attenuation characteristics.

Figure 16 shows a typical acoustic emission leak testing instrument, which can be used readily to make pipeline attenuation measurements on a short length of pipeline. This instrument can concurrently monitor and compare signals from two channels connected to sensors at a fixed distance from each other along the length of the pipe. The instrument contains electronic circuitry to process both leakage and noise signals and to compute leak location, as well as to measure signal attenuation in decibel units. The leak noise discriminator provides separate signals to signal lights that indicate either leakage signals, noise signals or both.

When it is desired to install a permanent leak testing system on a pipeline (using periodically spaced permanent monitor sensors or waveguides), the measured attenuation values are used to determine the total number of sensing stations (and the optimum distance between adjacent sensor stations) required for the pipeline leakage monitoring system. Thus, signal attenuation conditions have a strong effect on the number of sensors and signal channels required and this has a strong effect on the cost of the pipeline leakage monitor system installation and equipment costs. For this reason, the pipeline attenuation measurements should be made in advance of the design and installation of permanent pipeline leakage monitoring equipment.

Ultrasound Leak Testing of Underground Pipelines

Ultrasound detection can be used most effectively for locating leaks in new pipeline installations if pressurization and ultrasound detection are conducted before backfilling. After the trench is filled with earth, ultrasound leak testing becomes more difficult. High velocity fluid leakage from underground pipelines may be detectable by ultrasound leak detectors under most conditions. Leak signal detection from buried systems is dependent on each material and packing depth to cover and fluid characteristics such as viscosity etc.

Steam Systems

The generation of steam is costly and steam leaks in a system lower its efficiency and raise operating costs. Ultrasound detection and location of leaks in high pressure steam systems is a simple matter of using the airborne ultrasound probe to locate the source of the leak. As a matter of fact, many such leaks can be spotted by audible sound alone without recourse to instruments. Even with audible leaks, however, the ultrasound detector contributes ease of detection by the directional response of the microphone probe. Additionally, because it is not sensitive to audible sound, the directional probe simplifies the problem of locating leaks in noisy areas.

Steam leaks in low pressure systems, such as those normally used for heating buildings are very hard to locate because the sound level of the leak is

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correspondingly lower. Here, an ultrasound detector is almost indispensable. It can detect a leak far smaller than that audible to the ear and at the same time ignores audible noise in the area.

Locating High Temperature Leaks

Ultrasound leak testing has proved particularly effective in pinpointing leaks occurring in flanges and leaking fittings of high pressure and high temperature gas systems. One chemical plant, for example, reports that ultrasound detection is a mandatory test procedure on stainless steel flanges on reformers carrying hydrogen at 730 °C (1350 °F). Fin tube exchangers and other irregular fittings common to chemical processing have shapes unsuited for temporary repairs or bubble testing techniques. Here again, ultrasound leak testing is an effective maintenance procedure in the location of leaks.

Ultrasonic Identification of Split Boiler Tubes

A recent practice developed by power plant engineers permits the precise determination of the location of a split tube during boiler operation. By detecting the ultrasonic energy released by the tube leak, the operating engineer is warned of the presence of a leak far in advance of chemical analysis. Furthermore, by picking up the presence of a leak and identifying its general location, ultrasound testing speeds up isolating the leaking tube during hydrostatic tests.

During boiler operation, the engineer surveys the boiler wall surfaces with the airborne ultrasonic signal probe and places a chalk mark at the position of maximum sonic intensity. During shutdown for hydrostatic tests, the engineer enters the manhole with the ultrasonic translator detector and locates the high frequency acoustic energy emanating from the invisible high pressure mist at the rupture tube. The two-stage ultrasound testing affords a considerable reduction in personnel hours for boiler repair as well as providing advance warning of early stage leaks to prevent unscheduled downtime.

Steam Trap Inspection

A less obvious source of leaks, but very costly ones, are faulty steam traps. In any steam system, a steam trap is located on the downstream side of each load. Its purpose is to exhaust condensate from time to time. When condensation builds up in the trap, a valve opens and condensate is ejected into the return line. Some traps are also designed to operate whenever the trap temperature falls below a preset level. The trap is designed to eject moisture and is in series with the load. If the trap should stick open, the steam loss is tremendous. On the other hand, if the trap were to stick shut, the condensate flow may be prevented. If this occurs, for example, to one of a bank of heating jackets, the loss in efficiency may be great without the stoppage being obvious to maintenance personnel.

In the past, various techniques have been used to spot anomalous or leaking steam traps, ranging from listening for the sound of passing gases in high pressure systems (often clearly audible if there is not a high ambient noise level) to the use of pyrometers or thermomelt crayons on the upstream and downstream sides of each trap. This lengthy, messy approach has often been only partially effective. Listening for audible leak sounds is of no use in low pressure steam systems (not enough sound is generated) and is useful in high pressure systems only in quiet environments.

Ultrasound provides the only positive test technique. The contact probe is not disturbed by a noisy environment and clearly detects the sound of passing steam in the trap. Normally, the meter will be seen to jump sharply to very nearly full scale whenever the trap exhausts. If sound is present all the time, the trap is stuck open. If sound is never present, the trap is stuck shut.

Steam Plant Heat Exchangers

The following typical condenser tubing leak testing procedure is becoming increasingly common among electric utilities. On noting a reduction in condensate purity, the appropriate side of the condenser is drained and a vacuum on the order of 1 kPa (0.3 in. Hg) is attained. The control engineer then earmarks the leaking tube by aiming the ultrasonic probe at the bank of tube ends facing the manhole access and zeroing in on the noisy tube or tubes. Utilities report the procedure generally takes substantially less than the setup and cleanup time required to use a foam generator type of leak testing fluid. A number of utilities using salt water to fresh water condensers have used a similar ultrasound testing technique and have reported substantial time savings. One end of the tube is masked and the condenser jacket is pressurized with air to a maximum of 28 kPa (4 $lb_f \cdot in.^{-2}$ gage) pressure.

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Aerospace and Aviation Industry

Rocket Nozzle Fuel Line Inspections

For certain rocket nozzle tests using combustible gases such as hydrogen, the gas supply lines are pretested with helium to 4.2 MPa (600 $lb_f \cdot in.^{-2}$). These pressurized components are then safety inspected with an ultrasound leak detector. Under 4.2 MPa (600 $lb_f \cdot in.^{-2}$ gage) helium pressure, the sensitivity is such that it will detect a leak smaller than 0.01 mm (5×10^{-4} in.) diameter at close inspection range.

Aerospace Rocket Test Vacuum Chamber

Testing for leakage into a 5.5 m (18 ft) long aerospace test vacuum chamber and its related components requires less than 5 min by using ultrasound detection. A five-stage steam ejector pumping system, powered by two 1.1 MW (100 boiler horsepower) steam generators, produces within the 5.5 m (18 ft) long test chamber a continuous vacuum flow condition ranging from 0.01 Pa to 100 kPa (50 mtorr to 1 atm) absolute. Ultrasound leak testing significantly increased the speed of inspecting weldments, without resorting to potentially dangerous artificial pressurization.

In addition to access ports, flanges and vacuum instrumentation fittings, a varying number of pneumatic power lines actuating the test equipment enter the chamber. To ensure the integrity of the system, all of these items are inspected, before each firing series, with a portable ultrasound leak detector. The ultrasound test is a typical standard checkout procedure before tests simulating the heating of reentry vehicles and rocket nozzle materials. Ultrasound detection precludes the necessity for artificially subjecting vacuum equipment to pressurization for leak testing by other techniques. The ultrasound leak testing device is also used in the manufacture of missile handling containers, in which a rubber enshrouded pad of shock foam must be evacuated to permit insertion between two steel tubes.

Inspecting Aircraft Fuel Systems

The applications of ultrasound testing of high pressure systems include the maintenance of cabin oxygen systems aboard transport aircraft, cockpit pressurization inspection, aircraft fuel tank and fuel supply system inspections and safety inspection of high velocity, high temperature shock tunnels. Also, hydraulic systems on board aircraft are tested with contact sensors for correct operation, including leaktight seals etc. The ultrasonic method is now used for detecting and locating leaks in fuel systems and pinpointing malfunctions in hydraulic systems of aircraft.

With ultrasound detectors, airframe mechanics listen for the high frequency, inaudible sounds created by air molecules escaping from leaks even within wings and listen for the ultrasound of hydraulic fluid bypassing dysfunctional valves. Previous techniques of detecting aircraft fuel system leaks involving detector probes often provided the information only on the existence of a leak, not its precise location. The time required for tracer fluids to permeate fuel systems was also a problem.

Most aircraft use bleed air to supply air to subsystems designated as high heat systems. The temperature of the bleed air depends on which stage the bleed air is removed from the engine. Bleed air temperature can be from 385 to 774 °C (725 to 1425 °F) and at pressures of 21 to 75 kPa (3 to 25 $lb_f \cdot in.^{-2}$ gage). Airborne ultrasound is used to identify bleed air leaks between the aircraft engine and these subsystems.

Progressive aircraft reworking calls for overall examination of fuel systems on some aircraft after specified total periods of flight time During progressive aircraft reworking, the aircraft's entire system of fuel tanks and lines is pressurized to 24 kPa (3.5 $lb_f \cdot in.^{-2}$ gage) with air. With the ultrasound detection leak testing instruments in hand, the mechanic guides the probe along the surface of tanks and lines exposed for testing as well as along wing surfaces. When the detector emits a hissing sound, the mechanic aims the probe in the direction of the sound's greatest intensity. Marking this leak, the mechanic proceeds over the entire system. At 24 kPa (3.5 $lb_f \cdot in.^{-2}$ gage) pressure, the detector can pinpoint a leak smaller than 0.07 mm (0.003 in.) diameter from 450 mm (18 in.) away.

Aircraft Oxygen System Inspection

A low cost, portable ultrasound detection system has reduced the time required to inspect aircraft emergency oxygen systems by 50 percent, according to maintenance officials of commercial airlines. The ultrasound detector is used during overhaul and tests aboard all aircraft. To reveal leakage during overhaul, the passenger aircraft oxygen system is brought to its emergency operational mode. In some aircraft, the distribution manifolds throughout the aircraft and

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cabin are under constant pressure, permitting inspection without this preliminary step. With the ultrasound leak detector, airframe mechanics at the maintenance facility listen for leaks, even within cabin panels (see Fig. 17). The ultrasonic system has reduced the time required to inspect an aircraft's entire oxygen system from as long as three days to an average of 3 h. Before ultrasound leak detectors, aircraft maintenance practice called for tedious inch-by-inch applications of bubble test solutions. This required equally time consuming removal of all paneling.

Because of the short wavelengths of ultrasonic energy, the sonic waves from a leak, even from tubing having only low pressures, penetrate the cabin panels. With the ultrasonic instrument in hand (Fig. 18), the mechanic guides the probe along the full extent of the cabin paneling, tracing the approximate course of the cabin oxygen system. The cabin paneling is not removed. When the detector emits a hissing sound, the mechanic finds the location of the leak by aiming the probe in the direction of the sound's greatest intensity. When the mechanic finds leak noise behind a panel, the mechanic removes the panel, pinpoints the leak, makes the repairs and continues the mechanic's inspection. Similar inspection, of course, is performed at the oxygen supply within baggage holds (and above the flight compartment entrance way for the crew's oxygen system).

FIGURE 17. Detecting oxygen leaks in aircraft panels.

Inflatable Aircraft Escape Slide Inspection

The height of modern aircraft necessitates inflatable airplane escape slides and ramps that are long and do not buckle under heavy loads. There are many types in use, designed for particular aircraft and loads. Essentially, these escape slides consist of several inflatable tubes fastened together to form ramps, slides or chutes for emergency escape.

Testing escape slides for leaks can be a problem. The ultrasound leak detector is useful for finding such leaks. Additionally, most aircraft have pressurized cabins. If there is a suspicion of a leak in a cabin, an ultrasonic transmitter can be placed inside the cabin and the exterior of the aircraft can then be examined with the microphone probe to find the leak.

Vacuum Bags for Autoclave Bonding

Vacuum bags used for autoclave bonding of metal-to-metal sandwich and honeycomb aircraft structures are inspected for leaks ultrasonically. This reduces the inspection time from as long as 3 or 4 h to an average of 10 to 15 min. The ultrasonic system is used for inspection of nylon bags for leaks impairing vacuum integrity on such bagged assemblies as wing skin panels, trailing edge wedges and leading edge sections. The test is effective on structures for missiles, helicopters and airplanes.

Common aerospace industry manufacturing quality control standards prohibit bags from proceeding to the autoclave if they allow as much as 1.5 kPa (0.5 in. Hg) change in vacuum level from 88 kPa or (26 in. Hg). Even a 0.02 mm

FIGURE 18. Detecting oxygen system leaks in aircraft.

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(0.001 in.) diameter leak in the bags would permit an air return that would affect a uniform autoclave bonding pressure. Failure to bond properly could necessitate rejecting the assembled aerospace structure. For doublechecking each component before bonding, both bagging personnel and autoclave operators use ultrasonic systems to scan the entire vacuum bag assembly by moving the probe within about 40 mm (1.5 in.) of the nylon surface. At the scanning distance at which the probe is normally held, the operator will detect a leak smaller in diameter than 0.02 mm (0.001 in.) in the bag at the 88 kPa (26 in. Hg) vacuum level.

Vacuum Material Handling System Inspection

Some aircraft manufacturers conduct a daily inspection of vacuum actuated material handling equipment. Operating under 85 kPa (25 in. Hg) vacuum level, the grip system convey 1.6 mm (0.06 in.) steel plates measuring 1.5 × 6 m (5 × 20 ft). All possible leak sources such as fittings, flexible hose connections and hoses to the eight vacuum grips are inspected ultrasonically before overhead conveying starts.

Before day shift production commences each morning, the manufacturing supervisor surveys the vacuum system with a battery operated ultrasound leak detector. After establishing normal 85 kPa (25 in. Hg) operating vacuum, the foreman inspects each vacuum grip system merely by aiming the directional airborne probe at all possible leak sources, such as fittings, flexible hose connections, gage fittings and hoses leading to the eight vacuum grips. When the unit emits a hissing sound, like that of a punctured inner tube, the supervisor establishes the leak's precise location by coordinating the intensity of the sound with the direction of the probe.

Mechanical Inspection of Aircraft Hydraulic Systems

The application of ultrasound leak testing to aircraft maintenance is typified by another large scale leak testing program. Presently, the system is in use by both military and commercial aviation organizations. In overhaul or progressive aircraft rework, the entire hydraulic system is checked under linear flow pressurization. As each subsystem (e.g., flaps, landing gear) is put through its operating regime, the air frame mechanic applies the ultrasonic contact probe to the specific subsystem's valve components. Bypassing of fluid through a faulty valve, of course, is instantly recognizable. Reports from one large aircraft overhaul facility reveal that the ultrasonic elimination of step-by-step trial and error replacement of aircraft valves has reduced inspections from as long as 8 h to a maximum of 20 min. Similarly minimized are hydraulic component inventories and hourly operating costs for both the linear flow equipment and its operator.

Ultrasound detection also serves to speed periodic operational inspections of aircraft. One airline provides an interesting example of the application of ultrasound leak testing units. Part of this line's fleet is comprised of aircraft using pneumatic power systems. These are inspected with the general purpose probe for leaks at each operational check by a single mechanic in 90 min. Testing took up to eight work hours with previous techniques. Other aircraft hydraulic systems are inspected at required intervals with the contact probe and also after notification via pilot reports.

With a contact probe that responds to the 36 to 44 kHz ultrasonic bandwidth through solid conductors, the airframe mechanic can locate internal bypassing in hydraulic systems. When a system indicates malfunctions under lineal flow pressure, the mechanic applies the probe, like an ultrasonic stethoscope, to the specific system's valves. On hearing a "rushing water" sound when there should be silence, the mechanic can locate the dysfunctional valve.

The ultrasound detector is now often the only instrument used to check hydraulic systems. It has eliminated the previous step-by-step replacement of valves that took as long as 4 to 8 h per faulty system. The ultrasonic test requires only 15 to 20 min. Its use has reduced the inventory required for components and similarly reduced linear flow equipment costs and operator time. The device is also used to check the integrity of nitrogen pressurized engine shipment containers and in the overhaul of engines.

Mechanical Inspection of Compressor Valves

Locating dysfunctional valves and valve plates in multivalve cylinders is a straightforward application of ultrasound detection. Cracked or broken valve plates in compressors can be located without the contact probe but the contact probe permits much wider use of ultrasound detectors in machinery maintenance. To pinpoint dysfunctional valves, the mechanic places the probe against the valve plate, head or rocker box cover and listens for the pulsed whistling of compression bypass. Many maintenance mechanics become so adept that they can

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predict which quadrant of a valve plate is leaking before disassembly.

The ultrasonic contact probe is used whenever a compressor fails to maintain rated pressure levels. By holding the contact probe at right angles against the valve head, the operator can troubleshoot which valve plate, for example, is cracked within a multivalve head. The diagnosis of valve bypassing is simple; the mechanic needs only know the compressor's timing sequence to know when there should be periods of silence. The translated rushing sounds of valve bypassing are indications of trouble.

Marine Industry

Although ultrasound leak testing techniques in marine propulsion system applications are the same as those discussed previously, the problems of locating condenser and boiler tube leaks have historically proved vexing enough to warrant special discussion. One major producer of single plane marine propulsion units reports a 50 percent reduction in time required for leak testing compared with the time required to perform hydrostatic tests. Hydrostatic testing involves preparatory steps such as blanking the flange between low pressure and high pressure turbines. Although two-plane marine propulsion systems require less preparatory blanking, ultrasound testing during the inspection phase is equally effective and considerably faster.

After salinity indicators have revealed a leak in axial flow condenser units, the water boxes are drained and manhole covers at both ends are removed. Next, the steam side of the condenser is isolated, which merely entails securing all lines into and out of the condenser, such as dumps, drains, suctions and exhausts. The last preparatory step is to pressurize the condenser (through the steam side) to about 14 kPa (2 $lb_f \cdot in.^{-2}$ gage) maximum by using the ship's air system. A pressure gage control valve and relief valve are temporarily installed to regulate pressure within the condenser. Using a 25 mm (1 in.) air hose, it takes about 15 min to achieve satisfactory pressure within an about 27 m^3 (950 ft^3) condenser at a gage pressure of 14 kPa (2 $lb_f \cdot in.^{-2}$).

Pressure and Vacuum Leak Testing

Welding Ships and Large Chambers

A portable ultrasonic system to detect and pinpoint leaks has been used to inspect air pressure and venting systems at a major shipbuilding yard. Used on virtually a daily basis during the progress of ship building and repair, the firm has developed several techniques of ultrasound detection for leak testing.

Prior leak test techniques familiar to inspectors of ships and large closed chambers typically were application of bubble testing or of evacuating the structure and having an inspector move a candle along the course of all welded seams and penetrations to observe the flame's flickering to indicate a leak. According to one study made by techniques and standards analysis at a large shipyard, the cost per compartment of previous leak testing technology was high, whereas inspection processes using the ultrasonic system provided a savings of about 24 percent over previous techniques. The hazard of the candle test was demonstrated by the fire in the Brown's Ferry Nuclear Plant, which destroyed the signal cables to the control room.

Ultrasound leak testing has proved to be an effective means of inspecting large welded fabrications. One continuing application is inspecting the integrity of watertight compartments, skegs, rudders, tanks and other structures during shipbuilding and repair. Other similar applications have included the inspection of combustion chambers during the construction of an electric generating plant. Enclosures as large as 6400 m^3 (225 000 ft^3) have been tested by using the following procedures.

The initial test phase consists of pressurizing the enclosure to 3 to 15 kPa (0.5 to 2.0 $lb_f \cdot in.^{-2}$ gage) and, in the case of skegs and rudders, to 70 kPa (10 $lb_f \cdot in.^{-2}$ gage). Obviously, on smaller fabrication (e.g., small shipboard compartments etc.), if the gage indicates constant pressure for 10 min, there is no need for further inspection. However, when the gage indicates pressure reduction and/or the enclosure is so large as to preclude accurate pressure determination, then the ultrasound testing commences. Typically, the rubber extension is placed over the airborne directional probe and the inspector surveys all weldments. In the case of shipboard use, particular attention is applied to cable piping and ventilation penetrations.

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Field experience has shown that in compartments pressurized to 14 kPa (2 $lb_f \cdot in.^{-2}$ gage), the ultrasound detector can pinpoint a leak smaller than 0.1 mm (0.004 in.) diameter from a distance of 0.6 m (2 ft) even through 25 mm (1 in.) thick fiberglass insulation. The ultrasonic unit's standard earphones are normally worn during these tests because of the likely presence of high ambient audible noise levels. Ambient ultrasonic energy such as that released by pneumatic tools can easily be discounted by the operator by noting its direction.

Ship Air Conditioning Systems

In detecting leaks in air conditioning systems aboard ships, an airborne ultrasound leak detector can locate leaks through insulation ranging from 20 to 100 mm (0.75 to 4.0 in.) in thickness. Because of the short wavelengths of ultrasonic energy, the sonic waves from a leak — even from ducting having only flow pressures — penetrate the fiberglass blanket and can be detected from distances up to 1 m (40 in.). The airborne signal ultrasound detector response to leaks depends, of course, on the size of the leak orifice and the pressure differential across the leak. Three typical shipboard examples are cited. In air conditioning duct or compartment testing, a 0.1 mm (0.004 in.) diameter leak under 14 kPa (2 $lb_f \cdot in.^{-2}$ gage) pressure can be detected from 600 mm (2 ft). In 960 kPa (140 $lb_f \cdot in.^{-2}$ gage) pressure air systems, and at the same 0.6 m distance, the device can detect a 0.02 mm (8×10^{-4} in.) diameter leak. In 20 MPa (3×10^3 $lb_f \cdot in.^{-2}$ gage) high pressure systems, at the same 600 mm (2 ft) distance, it could detect a leak only 2 µm (8×10^{-4} in.) in diameter.

Ship Hull

Ultrasound tests performed on a refrigerator vessel being repaired revealed a leak in a refrigerated hold filled with perishable merchandise. This leak had leaked water into the refrigeration insulation when the temperature was raised for deicing. When the ship was in dry dock, the inspector scanned the hull and was able to pinpoint the leak within the hold's diffused vent ducting. The diffuser room was closed and pressurized. The leak was actually detected from the outside main deck at 15 m (50 ft) distance and from the shear strake plating at a 6 m (20 ft) distance.

Steam Turbine Exhausts

Ultrasound leak testing is used in production leak testing of the exhaust systems on steam turbines ranging in size from less than 0.1 m^3 (a few ft^3) to those 6 m (20 ft) tall and 9 m (30 ft) in diameter. With the turbine in place on its test stand, the system is evacuated to 85 to 95 kPa (25 to 28 in. Hg) vacuum gage level. The test operator ultrasonically scans the exhaust system, scrutinizing gaskets, bolted flanges, weldments and other possible leak sources. Such an inspection on a 2.2 MW (3×10^3 horsepower) class turbine takes about 30 min. This compares favorably with test times as long as four days by various other leak testing techniques. Optical comparator measurements by the test engineering staff indicate that leaks as small as 0.01 mm (4×10^{-4} in.) in diameter have been detected ultrasonically.

Ultrasound Leak Testing of Ship Compartments with Artificial Ultrasonic Frequency Tone Generator

An artificial ultrasonic frequency tone generator can be used when leakage fails to generate ultrasonic signals. The tone generator provides a sound beam with a conical beam. Place the tone generator anywhere within the ship compartments in which sound would have a free path to suspected leak area. The tone generator emits a frequency modulated signal in the range of 36 to 44 kHz. The heterodyned frequency is in the audio frequency range and this signal is responsible for the sound heard when the device is switched on. The ultrasound generated by the tone generator passes through small openings, cracks and leaks. It can be detected by the ultrasound detection probe on the opposite side of the object. However, for detecting extremely small leaks, the tone generator output beam should be aimed at the ultrasound detection probe. This may be achieved by keeping the tone generator and ultrasound detection probe simultaneously in the direct line with each other while the pair are scanning along opposite sides of a seam or suspected leak area. Solid material reflects the 36 to 44 kHz airborne ultrasonic signal and will prevent the ultrasonic signal from being received by the ultrasound detection probe.

This technique has become the preferred one for compartment tightness testing and bulkhead penetration leak location by the United States Navy. It is extremely difficult to pressurize the large bulkheads on an aircraft carrier, so testing for penetration leaks has not been done frequently. With the ultrasonic tone method, this task can be performed without any disruption to normal

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activities. This has saved many work hours during major overhaul of the ship.

Pressure and Vacuum Leaks in Automotive Industry

Leaking Valves in Automotive Engines

Leaking intake valves in auto and truck engines may be checked by using the contact probe and the headphones with the engine running. All valves should emit quite similar sound patterns or signatures when the probe is placed on the intake manifold opposite the intake port. The valve or valves out of pattern can then be positively checked, when the engine is not running, by bringing that particular cylinder to full compression and placing the contact probe on the valve stem. The hiss of gas escaping across the valve seat will be distinctly audible on the leaking valve. Exhaust valves can be checked by using the same procedure. Signal intensity will be much greater from exhaust valves than from intake valves.

Air in Automotive Cooling Systems

The detection of air pockets in liquid cooling systems of trucks and automotive equipment is receiving great attention, particularly in diesel engines. Air pockets can cause hot spots, which burn liners and heads in a very short time. Liquid cooling systems are under pressure so coolant must escape in order for air to enter. Aeration detection equipment gives the signal that air is in the system but does not locate the leak. Using ultrasonic contact probes in suspect areas near head gaskets or around water pumps and injectors will usually detect the exact sources of leakage.

Ultrasound tests should be conducted with engines idling. The bubbling of air in a cooling medium gives a very noisy detector signal on the headphones and usually permits exact determination of the problem. Ability to single out a certain unsuspected trouble point can save unnecessary labor such as engine head removal. Internal leaks may also be pinpointed with the contact probe immediately after engine shutoff when internal pressure is greatest.

With the engine idling, the inspector can clearly hear the pulsation in the auto exhaust system with the contact probe. Any leak points will give off a much louder and sharper sound. If required, the fine probe can be used to locate the exact leak point. The headphones are essential.

Leak Testing of Diesel Engine Fuel Suction

The trucking industry has applied ultrasound leak testing to troubleshooting of fuel suction systems on diesel engines. As the engine is running, the probe is moved along the supply suction lines, flange, gaskets and bolts in the fuel filter, the main fuel line and at other points such as the tachometer drive shaft fitting to detect leaks that should be repaired.

Leak Testing of Truck Tires

Tire personnel and mechanics at highway auto transport companies can use the portable ultrasound leak detector to locate tire leaks. Routine maintenance, of course, calls for first checking each tire with a pressure gage. When a reading indicates pressure loss, the next step is a fast visual test for noticeable valve leakage or casing cuts. If this step does not produce a satisfactory solution, the ultrasound leak detector is used. With the probe in hand, the mechanic scans the tire in much the same fashion as aiming a flashlight around its surface during visual testing. When the detector emits the hissing sound of a leak, the mechanic finds the precise location of the leak by coordinating the direction of the probe with the intensity of the hissing sound.

Because of the penetrating ability of sound waves in the higher (and shorter wavelength) frequency, the device responds to these ultrasonic sounds even when the leak is confined to a break in the inner tube. The ultrasonic energy created by the leak in a truck tire inflated to 670 kPa (95 $lb_f \cdot in.^{-2}$ gage) will penetrate the casing. Because the device does not respond to audible frequencies, the operator can discriminate between any change in ambient ultrasonic sounds created by pneumatic tools by noting their direction. Earphones are standard equipment for extremely noisy environments.

Tubeless tires mounted at each shop can also be inspected for air leaks, bad valves and cracked rims. Repaired tires are checked around tread areas after mounting. Tires with borderline pressure drops can be tested, marked and repaired. Higher pressure tubeless tires for trucks and buses are inspected ultrasonically for bead and rim leaks at a growing number of commercial fleet repair shops. Even fleets using tube tires report satisfactory tire leak location without submerging the tube. These truck operators report savings in work hours and marked reduction in highway tire failures. However, air trapped between the tube and tire on newly inflated tires can produce false leak indications.

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Leak Testing of Truck Brake Systems

Another use for the ultrasound detector is inspecting truck air brake systems to locate the leak or source of internal air bypassing that would not be indicated on the system air gage but could be in violation of governmental regulations. The air brake system of a large truck can be checked for both internal and external leaks in 10 to 15 min. Because the system requires no further analysis than the judgment and experience of qualified mechanics, who can recognize the sound of a leak or a valve that is not shutting sufficiently, training in detector use takes only a short time. In one truck repair station, some 40 mechanics and lubrication personnel are adept in ultrasound testing of air brake systems. Each needs only to check out the detector from the stockroom, thereby saving hours of laborious bubble testing.

To inspect an air brake system, the unit's pressure is built up and the brakes are applied to check storage and application pressure at the same time. With the ultrasound detector, the mechanics scan the brake system's tubing valves and fittings from underneath (either by creeper or at the grease pit). A leak will produce a pronounced hissing noise. An instance of valve malfunction will produce a flowing sound where there should be silence.

Inspection includes the entire course of tubing, hose, fittings, tanks and valves. When the instrument emits a hissing sound, the probe is zeroed on the precise location and the fault is marked for repair or replacement or, if possible, corrected at that time. Similar inspection techniques are used on refrigerated trucks, especially on the flexible tubing and fittings located beneath the trailer. The device also is used to check for and locate fuel system leaks.

Fuel Injector Performance Testing

As fuel injectors become dirty or worn, the spray pattern will change and the fuel will not be atomized properly.

With an unobstructed nozzle, the fuel will be atomized into a very fine particle size. This optimizes the combustion process and provides maximum fuel economy. If the contact probe is placed on the fuel injector body or on the fuel inlet line to the injector, ultrasonic readings can be taken and comparisons made. The good injector spray pattern will produce a more turbulent flow and will have higher amplitude. The poor spray pattern will have a lower amplitude due to the less turbulent flow. These tests can be performed while the engine is in operation, which can save many work hours.

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PART 4. Ultrasound Leak Testing of Evacuated Systems

Locating Leaks in Evacuated Systems

An ultrasound detection unit has proved a valuable supplement to the mass spectrometer for inspecting the integrity of vacuum enclosures and locating gross leaks in high power electron tube water cooling circuits. In this application, ultrasound detection provides a fast means for locating leaks on the order of 0.1 mm (0.004 in.) in diameter in these evacuated electron tube assemblies. These have leakage rates large enough to saturate the mass spectrometer leak detector. This example demonstrates the leakage range where each leak testing technique is best used. Ultrasound detectors are used to locate big leaks. Helium mass spectrometers are used for locating small leaks and for measuring leakage rates.

The exhaust supervisor conducting tests on electron tube water circuits uses the ultrasonic unit to detect leaks ranging from those immediately apparent to the eye or touch to those approaching the sensitivity limit of a mass spectrometer. When this range of vacuum leak situations is present, the supervisor disconnects the mechanical vacuum pump fitting and pressurizes the water cooling circuitry to 140 to 280 kPa (20 to 40 $lb_f \cdot in.^{-2}$) with the plant's central nitrogen supply. After establishing this pressure, the exhaust supervisor scrutinizes the circuitry by aiming the airborne ultrasonic signal probe at close range toward all possible leak sources such as fittings, flanges and, of course, weldments.

The device does not respond to audible frequency level, and the operator distinguishes leakage sounds from the chance ultrasonic sounds released by nearby exhaust processes by noting the direction of their sources. At the less than 20 mm (0.8 in.) detection range used, the ultrasonic airborne probe can pinpoint a leak in the electron tube water cooling circuitry smaller than 0.02 mm (8×10^{-4} in.) diameter when pressurized with nitrogen to about 200 kPa (30 $lb_f \cdot in.^{-2}$ gage) pressure. The ultrasound method takes one person only 3 to 5 min. After repair, mass spectrometry is again used for helium leak tests.

Ultrasound detectors also serve to ensure against leakage in the 20 000 m^2 (200 000 ft^2) manufacturing facility's nine-line exotic gas manifolds and to detect boiling in heat exchangers. In the latter use, the instrument responds to the ultrasonic energy released by steam formation within stainless steel collector units to provide engineers with an immediate indication of the efficiency of heat exchangers during the design evaluation of high power electron tubes.

Basic methods of leak testing of vacuum systems and evacuated components are described in detail elsewhere in this volume. With ultrasound leak detectors, leaks in vacuum systems are located by detecting the acoustic energy released by the interaction of air molecules activated by the pressure differential across the orifice. A rubber focusing extension on the airborne probe is recommended for vacuum systems; otherwise the techniques are quite similar to those used for locating leaks in pressure systems.

Techniques for Ultrasound Leak Testing of Large Vacuum Systems

Large vacuum systems such as those used at arc plasma facilities and tunnels simulating high altitude provide good applications for ultrasound leak testing. Because of their size and complexity, many of these systems are not designed to withstand both vacuum and pressure stressing. This precludes pressurization testing. Engineers in these fields have developed two distinct techniques for using ultrasound leak detectors.

One research facility operates a 5.4 m (18 ft) long test chamber with continuous vacuum flow condition ranging from 0.1 Pa to 100 kPa (1 mtorr to 1 ktorr). In this installation, research engineers inspect the chamber before each firing series by holding the rubber focusing extension tube of the ultrasonic leak detector probe against a solid surface of the housing, with the instrument set to almost full gain control. Having established this audible response and meter reading for a *no leak* condition, the inspector then proceeds to test all access port seals, flanges, vacuum instrumentation fittings and pneumatic power line penetrations for higher

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intensity signals typically representing leaks.

The second example of aerospace developed techniques is an ingenious technique of amplifying the ultrasonic energy released by a leak in a low vacuum system, where a pressure differential of 14 kPa (2 $lb_f \cdot in.^{-2}$) or less may exist. Distilled water or ether is applied by brush to the outside of the vacuum chamber, fittings and other components. When this liquid agent enters the vacuum leak, its cavitation releases energy of sufficient level to permit leak testing from a distance as far as 7 m (23 ft) from a puncture measured by National Aeronautics and Space Administration scientists at 0.01 mm (4×10^{-4} in.).

One of the larger vacuum chambers known to be maintained ultrasonically is a 4800 m^3 (170 000 ft^3) high altitude tower, operating during tests at 800 Pa (6 torr). The 30 m (100 ft) tall tower simulates 30 km (19 mi) high altitudes. The ultrasound detector is used before and during each test to ensure the integrity of numerous instrumentation penetrations, electrical conduits, observation ports, evacuation plumbing to the steam ejector plant and inflatable seals on a 30 t (65 000 lb) steel door. Such previously conventional techniques as artificially pressurizing the vast structure or smoke or candle flame techniques are obviously impracticable. The same aerospace facility also uses ultrasound to test laboratory equipment operating at 0.1 mPa (1 µtorr) absolute pressure.

Wet Chlorine Gas System

A 90 m (300 ft) long low vacuum network transporting wet chlorine gas has been inspected for leaks on a regular, weekly basis by ultrasound detection. Transporting wet chlorine gas from cell plants to drying towers, the 0.6 m (24 in.) diameter and smaller vacuum lines operate under a low vacuum level of 3.7 kPa (15 in. H_2O or 380 mm H_2O). Each of the three vacuum lines extends for more than 90 m (300 ft).

When excessive air levels in the system are noted, the ultrasound leak test begins. The engineer assigned the task scans the entire network, paying particular attention to flanges and leak band seals. In this manner, an entire network can be inspected and leak areas marked for tightening or repair in less than 15 min. The previous network inspection was by visual testing of the entire vessel. This was particularly arduous and costly in terms of work hours, especially in the case of leak bank seals. The ultrasound leak detector is also used to search out vacuum leaks in evaporator bodies and to maintain the integrity of the plant's pressure system. Another, more routine example of ultrasound leak testing is the plant maintenance man's regularly scheduled monthly foot patrol of the complex manifolding of compressed air, oxygen, acetylene and vacuum lines.

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PART 5. Ultrasound Leak Testing of Engines, Valves, Hydraulic Systems, Machinery and Vehicles

Ultrasound Leak Testing in Hydraulic and Pneumatic Systems

Figure 19 illustrates the operation of the ultrasonic contact probe in leak tests of fluid power networks. The fluid under pressure bypassing a valve releases ultrasonic energy readily detectable to the inspector with knowledge of the specific fluid circuit operation. The ability to pinpoint such a bypassing discontinuity without trial and error disassembly is a significant advantage. Beyond this immediate analysis, ultrasound leak testing also provides the design engineer with insight as to the efficiency of hydraulic systems by detecting cavitation.

Ultrasound Leak Testing in Design of Hydraulic Systems

A manufacturer of hydraulic shock absorbing devices uses ultrasound leak tests as a standard design analysis procedure. Prototype equipment and existing equipment that will be subjected to new loading requirements are checked ultrasonically during dynamometer tests. During the prescribed cycling, the hydraulic design engineer determines, by noting the intensity of the ultrasonic acoustic energy, if hydraulic valves operate at proper pressure settings on both compression and rebound strokes. Higher intensities indicate excessive control damping and valves with too weak a setting can be similarly isolated. In addition, the ultrasonic noise tests may reveal valve designs that create excessive sonic energy, which, for example, might transmit noise or cause resonance within the passenger compartment of a vehicle.

FIGURE 19. Detecting hydraulic system leaks.

Maintenance of Hydraulic Systems

There are numerous applications of ultrasound leak tests in the maintenance of hydraulic systems. Die casting machines afford a good example of the efficiencies possible through ultrasound leak testing. Many components within hydraulic systems of die casting machines are welded together or fitted under enormous torque. It is advantageous to locate a valve responsible for casting pressure contrary to specification while the machine is still operating and before maintenance disassembly. The maintenance engineer, on noting a pressure deficiency during operation, scans the hydraulic components ultrasonically. As an additional reference, comparative checks with the ultrasonic contact probe can be made on counterpart hydraulic components on identical machines within the same plant. It has been found that this practice gives the engineer a virtually 100 percent chance of isolating the difficulty before repair. Field reports conclude that dysfunctional components generally can be identified and located within a few minutes. This compares favorably with often weeklong procedures of step-by-step disassembly of hydraulic systems to locate sources of leakage.

A pretest of a hydraulic system using an inert gas as a pressure source will allow quick and effective leak testing with the airborne ultrasonic sensor. A large aircraft manufacturer uses this technique on the assembly line to eliminate the costly cleanup of hydraulic fluid that has leaked.

The airborne ultrasound leak signal probe has proved extremely effective in pinpointing external leaks in complex hydraulic systems. Such leaks often produce an oil pool 1 or 2 m (several feet) away from the actual orifice. Ultrasound detection locates the shrill hissing at the source of the atomized oil.

The airborne probe may detect ultrasonic vibrations of some thin walled metallic structures that vibrate, like diaphragms or drum skins. However, the

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airborne probe is unable to detect this energy unless the metallic surface is light enough to be displaced mechanically by the acoustic vibrations. Thin walled tubing or sheet metal structures are typical examples of vibration sources to which the airborne probe responds.

The contact probe for an ultrasonic translator leak detector is shown in Fig. 12. Its piezoelectric vibration sensor is coupled to the stylus, which is held in direct contact with structural surfaces to detect the ultrasonic vibrations transmitted through solid structures.

Heavier metallic structures such as cast fluid power components and generally all engine structures such as heads and bearing housings readily conduct ultrasonic acoustic energy. However, the mass of the structure prohibits its reverberating sufficiently to rebroadcast the acoustic energy through the atmosphere. The contact probe of Fig. 12 can detect these vibrations.

The ability to hear a distant train by putting one's ear to the railroad track provides a good analogy to the sonic conductance of a metallic mass. In this case, the ear is acting as a contact probe. This very phenomenon proves valuable in the practice of ultrasound detection. Because the contact probe cannot respond to acoustic energy transmitted through the atmosphere, its detection is limited strictly to ultrasonic energy released within the metal structure. Maintenance inspection with the contact probe is immediately pinpointed to the precise area of interest. Furthermore, knowledge of the precise inspection point allows repetitive, comparative inspection.

Mechanical Inspection

Ultrasonic noise (body noise) detected by the contact probe is generated by two rubbing or touching metal parts or surfaces, especially loud when lubricant has been lost by leakage. This fact allows checks of bearings and other mechanical moving parts for out-of-tolerance and poor lubrication conditions. A well running bearing whose lubrication is adequate may produce an ultrasonic noise that is transposed by the detector to a soft, whizzing tone. However, a bad bearing whose lubricant has leaked out, permitting metal-to-metal contact, produces a bad tone, significantly louder and often scratchy.

Applications of contact probes also include the checking of the sharpness of tools and application of cutting fluids on fast running, cutting machine tools, cam plates and eccentric plates, gears and all other mechanically moved parts rubbing or striking on a metal surface due to wear or poor lubrication. Other examples are the checking and detection of dry running ball bearings and friction bearings, noisy transmissions that have lost fluid, leaking valves and slide valves with internal leakage through the closed valve.

Bearing Analysis

Bearings can be analyzed with a structure borne contact probe. Based on research conducted by the National Aeronautics and Space Administration and many years of experience, the first indicator of a bearing going into a failure mode (microscopic degradation of the bearing wear surface due to lack of lubricant) is the rise in ultrasound. This rise in amplitude is heard through the ultrasound detector as a rough or raspy sound. The experienced operator can detect this anomaly immediately. The amplitude can be trended for further investigation. This will allow the bearing to be changed long before it fails completely. If the ultrasound detector is used during inspections before plant shutdowns, problems can be identified and corrected during the planned shutdown rather than causing an unplanned shutdown later. Also if the ultrasound detector is used during the lubrication procedure, it can help the operator to determine precisely when enough lubricant has reached the wear surfaces.

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PART 6. Electrical Inspection

Ultrasound Detection of Electrical Leakage and Arcing

Lightning is a good example of a large scale electrical leakage current conducted by arcing breakdown of the atmosphere. Current, often of the order of hundreds of kiloamperes, rapidly heats the air in its conduction path. This rapid expansion of the air acts like an explosion to generate intense, steep front sound waves that can be heard for considerable distances. The human ear can often detect these acoustic signals, which, if echoes do not interfere excessively, permit quick estimation of the direction and distance from the human observer to the sound source. Airborne sound at normal atmospheric pressures travels at speeds near 330 $m \cdot s^{-1}$ (1100 $ft \cdot s^{-1}$). Thus, a signal delay of 3 s corresponds to about 1 km (0.6 mi) of travel distance.

For years, the accepted procedure of detecting high voltage electrical leakage and arcing has involved two persons. One man remains on the ground to operate a standard radio frequency electrical signal detector while the other climbs a pole to probe suspect components with an electrically insulting *hot stick*. The object is to find a component of the transmission system that caused a noticeable change in radio frequency electromagnetic emission when probed. The ultrasonic mechanical vibration energy emitted by high voltage electrical corona, arcing and insulation breakdown phenomena is similar in its audible characteristics to the sounds such phenomena create on a radio frequency interference locator or on a portable or car radio. Each of these electrical phenomena is associated with leakage of electricity from bare or insulated conductors and the resultant local ionization and heating of air or surrounding fluids. In some stubborn situations, it is necessary to go over an entire local power distribution system, tightening all components when the exact noise source could not be located. This procedure consumed considerable time.

The airborne ultrasound detector has changed all this by eliminating one inspector and speeding up the whole operation. The radio detector is still used to locate the pole or immediate area of trouble, but once this has been accomplished, the inspector switches to an ultrasound detector with an airborne signal probe. Being light and compact, an ultrasound detector can be easily carried while patrolling a right-of-way. If windy conditions must be simulated, the pole is struck with a sledge hammer and the resulting corona response is noted with the detector.

Because of the directional focus capability of airborne ultrasound detection units, it is possible to locate the source of such conditions at considerable distances and with procedures ensuring the safety of the inspection personnel. Audio heard through the detector as a result of corona sounds much the same as through a radio frequency detector, but the ultrasonic unit is far more directional. The faulty component may be located exactly from the ground.

Ultrasonic Probe with High Voltage Cable Tests

A number of manufacturers of such products as polyethylene insulated conductor (PIC) have adopted ultrasonic quality control inspection procedures as a means of detecting the precise location of shorts and crossed circuits among paired wires. Once the wrapping of polyester, polyethylene terephthalate or similar initial wrapping on a given cable is completed, each conductor of up to 1.5 km (5000 ft) length is subjected to 3 to 10 kV direct current proof test voltages. The test voltage is determined by wire gage, which normally ranges from 0.4 to 0.9 mm (26 to 19 American Wire Gauge [AWG][4]). A failure on the high voltage insulation test identifies the reel of wire that must be segregated for ultrasound testing. Equal test voltages are then applied to the suspect reels of wire, with more sophisticated instrumentation including an electrical fault locator of the Wheatstone bridge type to indicate the approximate locations of leaks. Once the cable technician has unwound the reel to this indicated area, the technician reapplies the test voltage and begins ultrasonic scanning from a safe position beyond a safety barrier at least 1 m (3 ft) distant from the high voltage test circuitry.

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With the cable suspended between two reels about 6 m (20 ft) apart, the technician moves the probe along the length of the cable. The location of the most pronounced crackling or sputtering sound is the location of the fault.

After the technician removes the polyethylene terephthalate cable insulation cover, the technician can quickly locate the faulty color coded pairs of wires as indicated from initial high voltage testing and make the repairs with polyethylene sleeves. Following this, the cable is retested and made ready for shipment after aluminum and polyethylene sheathing is applied.

Interpretation of Acoustic Signals during High Voltage Electrical Tests

As the test voltage is increased to specified ratings of electrical cables and components, the inspecting technician listens for the following symptoms and causative leaks indicated by the ultrasound test device: (1) frying sound, culminating in a pronounced *click* indicating a contact arc; (2) continuous frying sound indicating internal corona caused by discontinuity in encapsulating resin bond; or (3) buildup of intensity of corona sound indicting progressive deterioration and culminating in silence indicating capacitor breakdown at a given test voltage.

Ultrasonic Monitoring While Inspecting High Voltage Transformers and Capacitors

One manufacturer of electronic measuring instruments inspects 60 Hz, 4 kV filament transformers for corona as both a design and quality control procedure. The inspection is semiautomatic in that the voltage levels are supplied by an automatic high potential test while the inspector scans each assembly with the rubber focusing extension on the ultrasonic contact probe.

Two more high quantity electrical components tested ultrasonically are high voltage pulse capacitors and toroidal and C-core transformers. Both tests use the ultrasonic contact probe. An atomic research facility regularly tests 50 kV pulse capacitors for corona threshold in less than 1 min each. A typical test consists of an application of 35 kV root-mean-square alternating current sine wave voltage to provide a peak voltage equal to 50 kV for about 5 s.

The low cost ultrasound detection setup has demonstrated the following superiority. There is not ambiguity about the test results — a satisfactory capacitor emits no ultrasonic energy during the test regime. Previous 100 kV direct current high potential tests were inconclusive, as were those using radio frequency electrical detection because acceptable corona discharges occur in the air around the bushing below test voltage. The contact probe does not respond to airborne bushing corona. Final benefit is the low cost of the test apparatus. It replaced a system that included costly corona-free transformers, power separation filters, oscillographs and related equipment.

Example Procedure for High Voltage Transformer Tests

One electronics firm subjects incoming high voltage transformers to three corona threshold tests. Both toroidal and C-core transformers typically are tested up to 14 kV before, during and after a 1 h long heat soak at +55 ºC (131 ºF). With the transformer on the test bench, a 1 kHz sine wave primary voltage is increased from zero until a secondary voltage of 14 kV is developed. The technician holds the ultrasonic contact probe against the transformer while wearing a 20 kV rated insulating glove and standing on a rubber pad with equal voltage insulation. Full safety precautions are required for this type of testing.

As the secondary voltage is increased to 14 kV (and often, as an extra precaution, to 16 kV) the technician listens for white noise. If between 14 and 16 kV the technician hears only a hum, the transformer is acceptable. If hash or frying sounds are detected before 14 kV, the unit is rejected. This test, only 5 min long, provides a rapid and successful measurement of transformer acceptability.

Electrical Substation Component Maintenance

It has become standard practice at a number of electric utilities to listen ultrasonically to substation components such as bushings, insulators and transformers. The contact probe is highly effective in locating internal arcing in transformers. The contact probe is merely held by the inspector at right angles against the grounded transformer case. For safety reasons it is essential to ground the transformer case first. The test operator must not expose himself to high voltage conductors and fields on open wires or bushings. Maintenance supervisors at substation facilities have attributed to ultrasound detection a reduction in the time necessary to isolate faulty hardware, as well as the prevention of serious outages. The techniques of pinpointing corona or arcing are identical, of course, to those for locating radio and television interference sources. Other

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substation inspections include periodic detection of pneumatic circuit breaker air pressure systems and bearing wear in transformer oil pumps.

Electrical Maintenance in Industry

A number of large industrial plants use their ultrasonic translator detectors also in the maintenance of plant electrical systems. Typical applications are the location of single source arcing grounds on distribution systems operating at potentials as low as 600 V; corona sources on lines, transformers and insulating bushings on secondary and primary distribution systems; and pinpointing arcing in corroded relay contacts on motor controls.

Pressure Insulated Flash X-Ray Equipment

Corona and High Voltage Breakdowns

A technique for the detection and location of corona and high voltage breakdown in capacitors within a 5 mm (0.2 in.) steel housing uses the 36 to 44 kHz ultrasonic acoustical energy released by electrical breakdown. This system is used as a quality control procedure during manufacture of high voltage X-ray and electron beam radiographic equipment. The ultrasound detection system gives immediate indication of the breakdown's nature and location to within a module of four capacitors among 80 in a 3.5 m (11 ft) long, 2 MV flash X-ray pulser assembly. An acrylic plastic sleeve is tightly fitted to the ultrasonic contact probe to locate discontinuities with corona discharges in the encapsulating resin of capacitor modules.

In addition to inspecting high voltage components, the ultrasound detection device is used to check the integrity of nitrogen and fluorocarbon gas pressure insulation vessels for flash X-ray apparatus. These radiographic devices use the Marx surge generator energy storage principle and range from portable 23 kg (50 lb) 18-module 100 to 150 kV models to 30-module 2 MeV units. Internal arcing caused by discontinuities in the encapsulating resin within a capacitor releases 40 kHz acoustic energy, which, when translated and amplified, is recognizable to technicians as the sputtering, frying sound of corona. A nitrogen or fluorocarbon gas leak through steel or fiberglass vessels sounds like the familiar hissing of a punctured inner tube. Because the contact probe is held firmly at right angles to the high voltage capacitor case, an electrically insulating acrylic plastic sleeve 1.5 mm (0.06 in.) thick and extending about 6 mm (0.25 in.) beyond the stylus tip is used. The transducer sensitivity is not impaired by the sleeve.

Ultrasonic Leak Testing of Flash X-Ray Pressure Systems

Smaller X-ray devices are pressurized with desiccant dried compressed air. Larger models have an inner fiberglass insulation vessel containing fluorocarbon gas and an outer welded steel vessel for nitrogen. Ultrasound testing is now standard for both types.

In this operation the inspector uses the directional airborne probe and scans weldments and the surface of fiberglass layups as the vessels are pressurized to 1.1 MPa (160 $lb_f \cdot in.^{-2}$) for nitrogen and 200 kPa (30 $lb_f \cdot in.^{-2}$ gage) pressure for fluorocarbon gas insulation. At the higher pressure, the instrument will produce a hissing sound to pinpoint a leak as small as 0.02 µm (7.5×10^{-4} in.) in diameter. The ultrasound leak testing takes an experienced inspector about 0.5 h for the largest pressure vessels, as opposed to 4 to 12 h required for leak testing by the bubble testing method and the resultant cleanup time.

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PART 7. Ultrasound Leak Testing of Pressurized Telephone Cables

Without ultrasound leak testing it has been necessary on aerial cable for the craftsman to apply bubble solution from the ground or a suspended platform and to attempt to watch for bubbles. For underground ducted cable, it was necessary to train personnel in sophisticated gradient measurement techniques to plot the leak location. However, the possibilities of error, even by the most technically oriented personnel, frequently resulted in much more extensive excavation than necessary merely to repair the cable sheath.

Modern telephone practice requires the installation of compressor/dryer units at central offices, with smaller pole mounted units supplying cable pressure in outlying areas. Flow indicators adjacent to these compressors provide telephone maintenance crews with constant readings as to the integrity of the cable. Additionally, contact terminals and pressure regulators are used throughout the plant.

The most common causes of leaks in cable plant are corrosion (particularly in coastal areas), electrolysis, squirrels, boring beetles, abrasion from wind and weather, hunters and outside workmen. Abrasion during installation and corrosion are the most frequent causes of cable sheath trouble in underground ducted passages.

Principles of Ultrasound Tests for Leaks in Telephone Cable Sheaths

The technique of ultrasound leak testing and location in telephone cables involves scanning the pressure system with the directional airborne signal probe and coordinating the direction of the characteristic hissing sound with its intensity (Fig. 20a). The aerial and underground pressurized cable plant of the modern telephone system is a large, low pressure system that lends itself to ultrasound leak testing during maintenance. All cable pressurization has resulted in overall reduction in outlay for cable plant maintenance. This is particularly true in the reduction of emergency repair time formerly encountered when rain entering the cable sheath resulted in widespread service disruption. Estimates by officials at various telephone operating companies as to the economies achieved by ultrasound leak testing range from 50 to 80 percent.

Although highly portable ultrasonic translator leak detectors have permitted telephone technicians to locate sheath damage in pressurized telephone cable from the ground, prudent supervisory management has established preinspection procedures to speed the operation further. It is typical practice, for example, for a splicer to perform the following preliminary steps on a cable failing to maintain the gas at nominal 70 kPa (10 $lb_f \cdot in.^{-2}$ gage) pressure.

1. Connect nitrogen cylinders set to give a gage pressure of 70 kPa (10 $lb_f \cdot in.^{-2}$)

FIGURE 20. Detection of leakage from telephone cable sheaths: (a) pressurized cable; (b) overhead cable.

(a)

(b)

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at strategic locations along the cable. Such cylinders often are allowed to remain connected for 24 h or longer to build up sufficient pressure.
2. Take cable pressure readings at selected points. This practice is particularly important on such cables as cross country toll lines that often traverse a line-of-sight right of way across precipitous terrain.
3. The readings taken at each pressure point are then plotted on graph paper. Each grid on the paper is selected by the inspector to represent a known distance as determined from the mechanic's cable plant maps.
4. An alternative means of narrowing down the point of the leak is with the cable pressurization computer or, as it is often called, the gas pressure slide rule.

Procedure for Ultrasonic Leak Testing of Overhead Telephone Cable

After the technician has determined the general location of the leakage to within the length of three sections or less, the technician would normally walk the route, using either a hand held probe or with a parabolic microphone hand held probe (Fig. 20b).

In certain instances, an areawide infestation of boring beetles will cause extensive damage and multiple leaks. The telephone splicing cable car is still required for ultrasound tests on cables where the cable traverses canyons or deep gullies. In locations where the cable can be as high as 60 m (200 ft) above the ground, the cable itself provides the only feasible path of locomotion.

Parabolic Microphone

A parabolic microphone lets the inspector effectively perform testing at a safe distance. The parabolic microphone doubles the detection distance obtainable with a conventional scanning module while narrowing the sound beam. Increased sensitivity of the parabolic microphone is due to an unique transducer assembly. The parabolic microphone has less than a 5 degree beam spread compared to the scanning module with a beamspread of about 45 degrees. Seven transducers enable the inspector to identify corona, tracking and arcing occurring in conductors, insulators, tie wires and bolts while the inspector is standing over 30 m (100 ft) away.

Training Personnel for Ultrasound Leak Testing of Telephone Cable

Training of personnel in ultrasonic leak testing is minimal. However, ability to hear sounds mimicking other, inaudible sounds is a new experience and it is recommended that cable maintenance personnel receive a brief introduction to the ultrasound detection instrument. Such an introduction can readily be set up by any telephone operating center. Many use cable vaults adjoining the central office. The instructor conducting the session will loosen air pressure valves to various degrees and then allow each of the students to find all of the leaks. The students are taught to coordinate the direction with the sonic intensity by reducing the gain of the airborne signal ultrasound leak detector as leaks are approached. This speeds the leak location process.

Protection of Ultrasonic Probe from Rain

In field operating conditions, rain falling into the ultrasound leak testing probe will temporarily decrease the unit's sensitivity. A simple way to prevent this loss of sensitivity is to remove the rubber screen cap from the end of the probe and place a thin sheet of plastic over the probe end. The rubber screen protector is then replaced. The plastic should not be stretched too tightly, as this would lower the probe's sensitivity. The addition of the plastic will lower slightly the sensitivity to distant leaks but has little effect on sensitivity to leaks closer to the probe.

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PART 8. Acoustic Emission Monitoring of Leakage from Vessels, Tanks and Pipelines

Acoustic emission (AE) can be used in a wide variety of applications to detect, locate and even quantify leaks. The escape of liquids or gasses through a pressure envelope may result in the generation of sound[5] that can be detected by one or more acoustic emission sensors and used to estimate the location of the leak source.

There are a variety of reasons why a leak may generate noise: (1) turbulent flow of the escaping gas or liquid, (2) cavitation during two-phase flow (gas coming out of solution) through a leaking orifice, (3) the pressure surge generated when a leak starts and stops and (4) backwash of particles against the surface of the equipment being monitored

The sound generated by a leak can propagate through the walls of a vessel as well as through any liquid inside. In general, it can be said that liquid inside a vessel or pipe will assist in the propagation of sound while liquid outside (such as moisture in the soil) has a tendency to reduce the detectable signal. Prediction of the actual acoustic waveform, generated by a leak, is very difficult. An example for a point leak in a buried pipeline has been reported.[6]

The frequency content of the leak signal can be considered broadband at the source. Various applications have been developed using a variety of sensors with sensitivities in the range of 1 to 400 kHz. Using lower frequencies implies that the leak can be detected from greater distances although effects of environmental background noise are more pronounced.

A typical low frequency application would be that of leak testing for buried pipelines where sensors are mounted so that they are no more than 15 to 30 m (50 to 100 ft) from any potential leak. A typical high frequency application would be that of internal leak testing for flare gas valves where a sensor is mounted in a location less than about 0.3 m (1 ft) from any potential leaks.

Feasibility of Acoustic Emission Leakage Monitoring

As discussed above, various applications require different frequency responses. leak testing for buried pipelines requires that a maximum sensor spacing be achieved. This drives the application towards the low frequency range where attenuation of the acoustic emission signal is not as severe as it is at higher frequencies. However, as the frequency is lowered, the effects of background noise become more pronounced, indicating that a compromise is required.

Internal leakage detection and assessment is performed on valves using acoustic emission testing. In this application, a sensor is placed on the valve so that it is less than about 0.3 m (“within inches”) of any leak site. Because the source-to-sensor relationship is so small, attenuation does not become a factor, thus allowing sensors that operate in the high kilohertz range, where background noise is minimized. By taking a measurement at higher frequencies, the content of the signal is dominated more by the leak than by background noise. This allows an accurate assessment of the leak rate to be made using the acoustic signal.

The effectiveness of the application and the design of the acoustic emission detection/monitoring system (for a given frequency range) depends on the following factors: (1) the amplitude of the leak signal at the leak source, (2) the background noise level, (3) the attenuation of the signal from the leak source to the detection sensor and (4) the need to characterize and separate the leak signal from other signals

Factors 1 and 4 can be investigated to some degree in the laboratory through simulation. If possible, it is best to investigate under real field conditions because this will provide the only opportunity for investigation of factors 2 and 3.

To best understand how all these variables are addressed and how applications are developed, applications are discussed in two categories below: (1) periodic proof testing and (2) continuous monitoring.

Examples of Periodic Proof Testing

Flare Valve Internal Leak Testing And Assessment

In 1982, a program was started by British Petroleum to develop leak testing and

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quantification for flare gas valve internal leakage.[7] The program involved a study of different types of valves ranging in size from 25 to 450 mm (1 to 18 in.).

Measurements were taken in service and then the valves were removed. In the laboratory, they were retested using a flow rig to simulate operating conditions to compare the leakage rate with the original acoustic emission signal measurement. After a database of over 800 valve tests was developed, a best fit correlation (between the acoustic emission signal and the leak rate) was generated.

As part of this developmental program, a system was fabricated that was simple and portable and could operate in an intrinsically safe environment (Fig. 16). This system can also be used for liquids with certain restrictions.

Factors having significant effect on the acoustic emission signal were found to include (1) valve type, (2) valve size, (3) differential pressure across the valve and (4) viscosity of the product (if it is a liquid) inside the valve

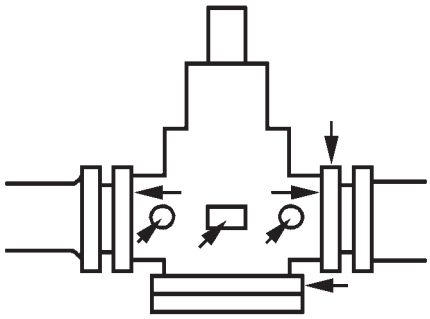
The operation of the instrument is simple.[8] The sensor is held in contact with a flat surface (Fig. 21), using a suitable couplant, on the valve to be tested. The current value of the signal level (dB) is noted. This may also be stored with a single keystroke in one of the 300 memory locations. If a leak is indicated by a reading greater than normal background (12 to 16 dB), then readings are taken on the pipe work upstream and downstream of the valve. As the signal level will be highest close to the leak and attenuate as the distance from the leak increases, these upstream and downstream figures will be lower if the valve is truly the source of the acoustic emission. The noted reading is then inserted into a personal computer spreadsheet along with the other relevant information: (1) valve inlet size, (2) differential pressure across the valve and (3) valve type.

This information is used in the spreadsheet by the predictive equation to calculate the loss rates. The spreadsheet is often modified to present the loss rate in convenient units such as metric ton (t) per year, cubic meter per day or even product value per period.

Some recent experiences include taking measurements on 20 valves on an offshore platform. The total leakage estimate was determined to be 85 $L \cdot s^{-1}$ (5.1 $m^3 \cdot min^{-1}$ or 2.5 $kt \cdot yr^{-1}$). In a refinery, a single 100 mm (4 in.) pressure relief valve was monitored and the acoustic emission signal level was determined to be 85 dB. In this case, this signal level equates to a leak resulting in product loss at a rate of 37.4 $L \cdot s^{-1}$ (2.2 $m^3 \cdot min^{-1}$ or 1.1 $kt \cdot yr^{-1}$). In a petrochemical plant, four 0.6 m (24 in.) valves were monitored. The results showed that two were leaking and that product was being lost at a rate of 85 $L \cdot s^{-1}$ (5.1 $m^3 \cdot min^{-1}$ or 2.5 $kt \cdot yr^{-1}$).

A 25 mm (1 in.) valve was detected leaking with product lost estimated at $34 000 per year. This valve was fixed on the spot just by adjusting the stop. The largest leaker found to date was a 0.6 m (24 in.) valve leaking at a rate estimated at 63 $L \cdot s^{-1}$ (134 $ft^3 \cdot min^{-1}$).

FIGURE 21. Flare gas valve. Arrows indicate points of interrogation for acoustic sensors.



Inservice Leak Detection for Aboveground Storage Tanks

A proprietary technology has been developed for inservice testing and assessment of tank bottoms for aboveground storage tanks. The development started with the desire to locate leaks in tank floors, during which time it became apparent that badly corroded floors, even when not leaking, made a lot of noise. The details of how to use acoustic emission for evaluating tank integrity and floor condition are given by Cole.[9]

The leak testing test is performed by instrumenting a tank with low frequency acoustic emission sensors. These sensors are designed to give the optimum performance when faced with high signal attenuation for large tanks and the possibility of background noise interference from environmental and mechanical noises.

Sensors are coupled to the outside of the tank wall, evenly spaced and mounted near the shell-to-floor interface. Before testing commences, calibration is performed to ensure that the sensors are properly coupled and that the instrumentation is functioning satisfactorily.

Because of low frequency sensor operation, the tank is allowed to still; pumps, agitators and valves are shut off; and piping attached to the tank is checked for possible extraneous noise sources. Weather permitting, data are collected in about 1 h. High winds, rain and hail generate considerable noise and

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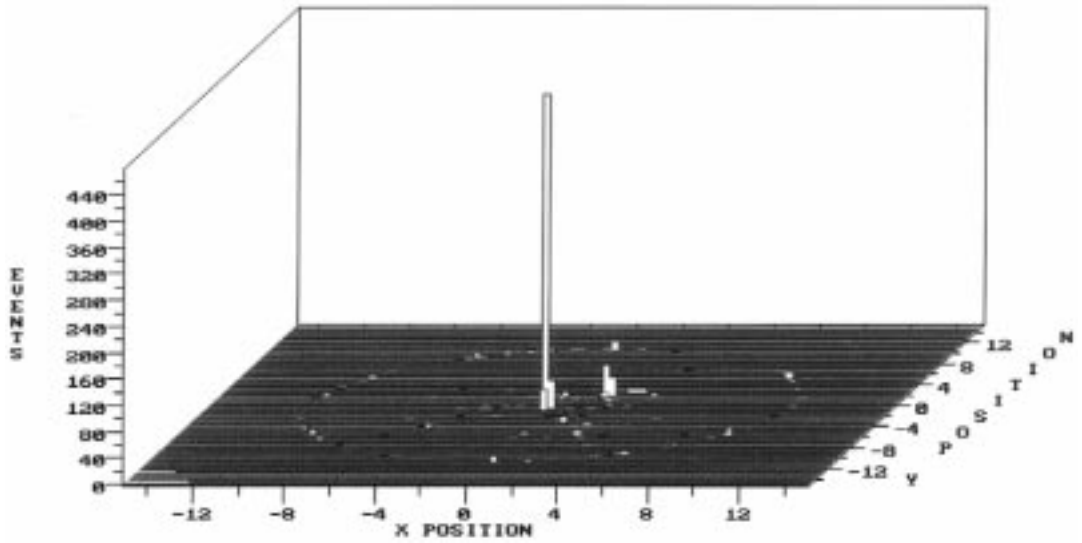
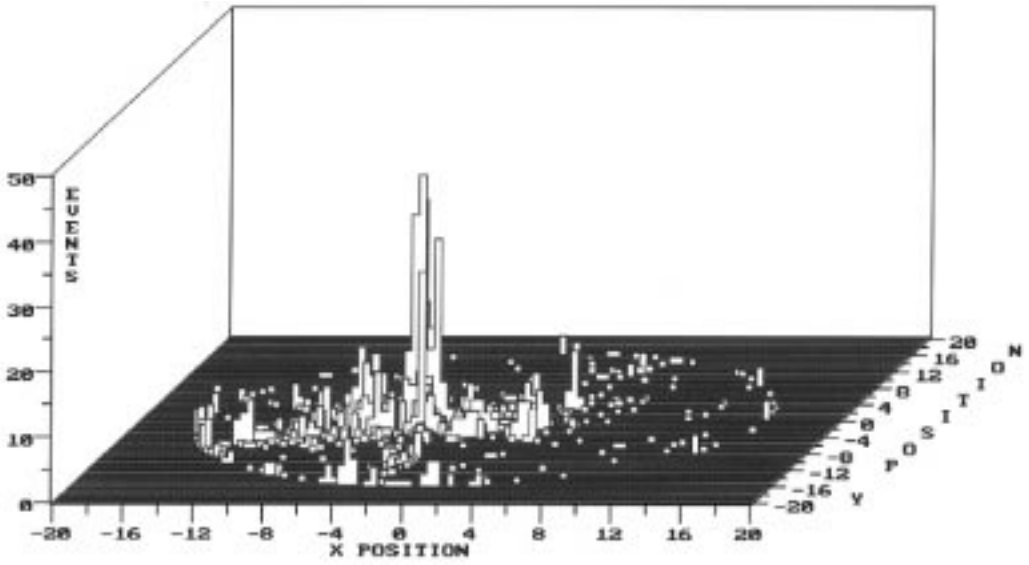
FIGURE 22. Computer generated maps of acoustic data: (a) 24 m (79 ft) diameter diesel fuel storage tank; (b) 38 m (125 ft) diameter naphtha storage tank; (c) glass reinforced plastic liner for 67 m (220 ft) diameter crude oil tank.

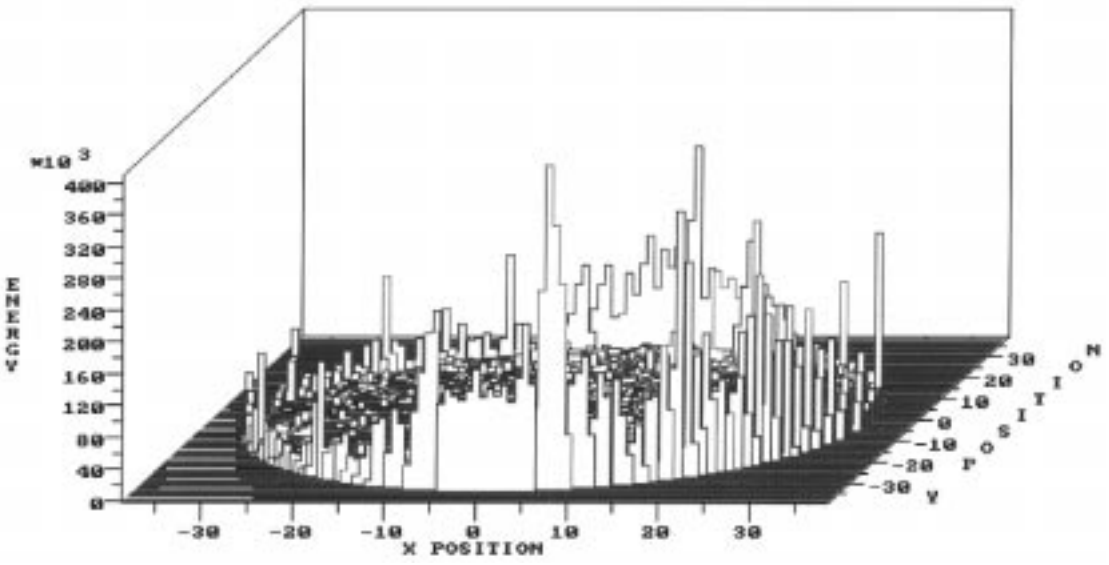
(a)



(b)



(c)



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are grounds for stopping or delaying the test. Tanks are generally filled with product to a prescribed level for this test.

Under normal conditions, one to two large diameter tanks may be tested per work day. More can be tested if they are smaller and close together.

Once data are collected, they are processed to determine where the noise sources originated. For determining the existence of a potential leak, the signature of the signal is examined and filtered to remove other possible noise sources. The filtered data are plotted on a location map to display potential leak locations.

Some times the process is rather straightforward, as shown in Fig. 22a. Here a 24 m (80 ft) diameter diesel fuel storage tank produced one location on the floor generating more than 400 locatable events. Internal inspection confirmed a 1 to 2 mm (0.04 to 0.08 in.) diameter pinhole where the epoxy coating had failed.

Similarly, a 38 m (125 ft) diameter naphtha storage tank was suspected of leaking about 90 t (100 ton) of product per day. The noise level from within the tank was so high that the test was performed at 2 percent of the normal sensitivity setting. Location of the leak (Fig. 22b) was within 2 m (7 ft).

In contrast, a 67 m (220 ft) diameter crude oil storage tank was tested producing the results shown in Fig. 22c. This tank had a glass reinforced plastic (GRP) liner that was found to be perfectly intact. Magnetic flux leakage testing indicated areas of greater than 60 percent of underside corrosion. Floor plates were cut out to confirm the presence of underside corrosion. The magnetic flux leakage results correlated very nicely with the acoustic emission location map.

Although the technology has evolved to where it is more useful as an overall condition assessment tool, it can still be effective in finding leaks. The results demonstrate that when the leak is the only noise source, its location can be identified with certain accuracy. There are concerns about accurate leak location when there are more than one leak source.

When the tank bottom is actively corroding, the noise from this type of source tends to overwhelm the data set, making it difficult or impossible to locate leaks. Therefore, this technology is best used as a surveying tool. When the activity from the bottom is considerable, it is time to enter the tank and perform internal inspection. If the tank is quiet, it is best to leave the tank in service rather than waste considerable cleaning and decontamination budget as well as internal inspection costs.

Examples of Continuous Monitoring

Application in Nuclear Industry

Westinghouse Electric Corporation has developed a leak testing system to monitor for leaks in the primary reactor coolant and steam piping systems in nuclear power plants. This system was developed in accordance with the guidelines provided in *Regulatory Guide 1.45* of the United States Nuclear Regulatory Commission.[10] Systems have been installed in several eastern European nuclear power plants.

The leak testing instrument is a personal computer based data acquisition system with online network communication to a central workstation that monitors the root-mean-square signal level for a maximum of 96 locations. The components of the system comprise sensors, signal processing instrument, workstation and an industrial personal computer with transmission control protocol and internet protocol network communications. The analog signal processing equipment and personal computer are shown in Fig. 23.

FIGURE 23. Data acquisition and processing system to monitor for leakage in nuclear power plants.

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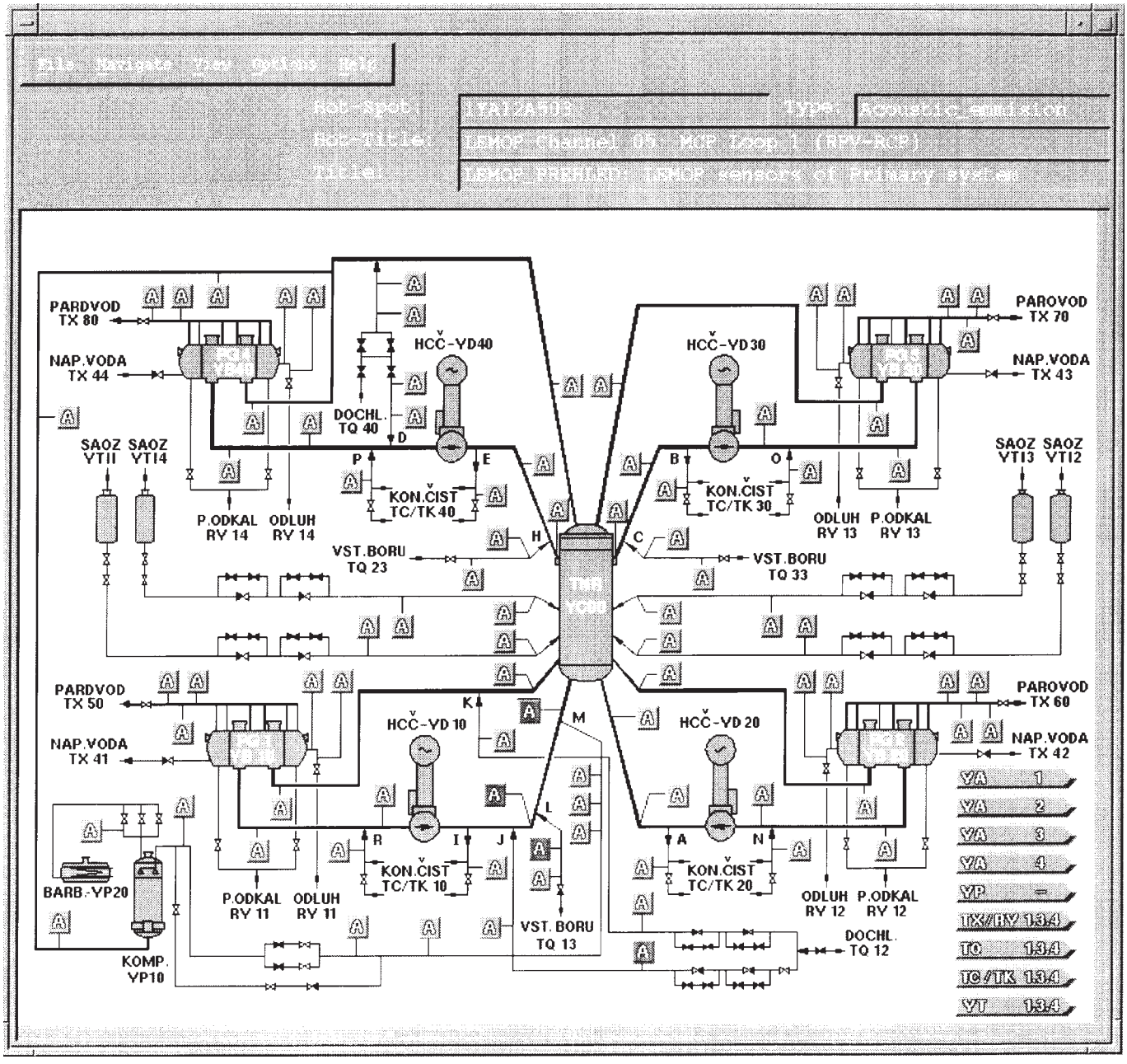
In the event that the root-mean-square level (for any given sensor position) increases above a preset threshold, the software resident on the workstation automatically determines if the increase in noise level is due to a leak or a change in the plant operating conditions, such as pumps starting and stopping, valves opening and closing, changes in power level etc.

The workstation has access to the plant data highway that provides the operating status of the various equipment in the plant. If no changes in the plant status are detected, then a leak is declared and the software validates the leak, begins to locate the leak on the piping system and quantifies the leak based on previous leak test data.

Figure 24 shows a screen display typically seen on the workstation. This display provides an overview of all of the sensor locations in the plant. The operator can navigate from this screen on the workstation to a more detailed view of the sensor locations. The workstation also has access to data from other plant monitoring systems, which include the main coolant pumps vibration data, loose parts monitoring data, pipe temperatures

FIGURE 24. Typical screen display of nuclear plant leakage monitoring system.



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and displacements and humidity monitoring systems. These data also provide valuable information for correlation with the changes in the root-mean-square levels and leak validation.

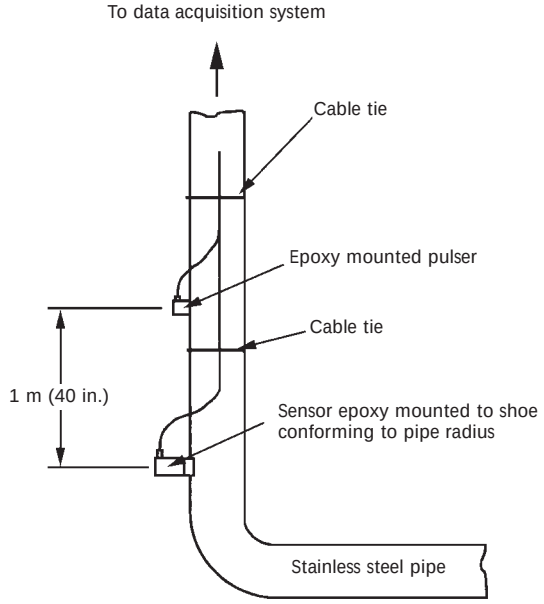
Continuous Leak Monitoring of Chemical Feed Supply Line

A continuous leak monitoring application has been developed for a chemical feed supply line. Sensors are mounted on a stainless steel pipeline at locations near potential leak sites such as elbows and valves. The sensors are adhesively bonded to a special shoe that conforms to the diameter of the pipe while providing a flat surface for sensor attachment. The shoe is also bonded to the pipe's outer surface.

Next to each sensor, a pulser is mounted within 1 m (3 ft) as shown in Fig. 25. Periodically, the pulser is driven with a high voltage spike so that it launches a sound wave that travels along the pipe wall. The sound is detected by the sensor and is used to verify system operation and sensor/pulser attachment.

No leaks have been detected during normal operation when the pipe is full of a liquid chemical. However, during a nitrogen purge, at 60 percent of the normal operating pressure, a valve was cracked to simulate a very small leak. The leakage was detected by a sensor located 5 m (15 ft) away and produced an energy level reading greater than 100 times that of normal operation. Another sensor located 30 m (100 ft) away detected the same leakage with a reading twice as high as for normal operations.

FIGURE 25. Mounting of acoustic emission pulser and sensor on stainless steel piping in chemical plant.



Leak Monitoring of Heat Exchanger in Chemical Industry

When hazardous or corrosive chemicals are used in chemical plants, acoustic emission leak monitoring can be integrated into plant controls to provide continuous feed back of pipeline or vessel integrity. Advance detection of very small leaks can prevent environmental incidents as well as the catastrophic loss of equipment and human life.

To develop an application of continuous leak monitoring, it is imperative to not only understand the physics of why a leak makes noise but also the variables associated with the process being monitored and the potential for false calls. An excellent example is given[11] for the development of a continuous leak monitoring system for a sulfuric acid plant.

This application focuses on a heat recovery system that extracts heat from concentrated acid contained in a stainless steel heat exchanger. If an internal leak occurs, water or steam will mix with the acid forming a highly corrosive dilute acid. The corrosive effect of the dilute acid is enough to destroy the equipment if leaking is not rapidly detected.

When a leak initiates and concentrated acid mixes with water or steam, a violent reaction follows that is audible and can be felt from the outside of the heat exchanger. The feasibility of detecting this event with acoustic emission was shown for a combination of instrument and sensors (Fig. 26) operating between 100 and 300 kHz. Because the operating temperatures reach 227 °C (440 °F),

FIGURE 26. Acoustic emission system used for leakage monitoring of heat exchanger in chemical plant.

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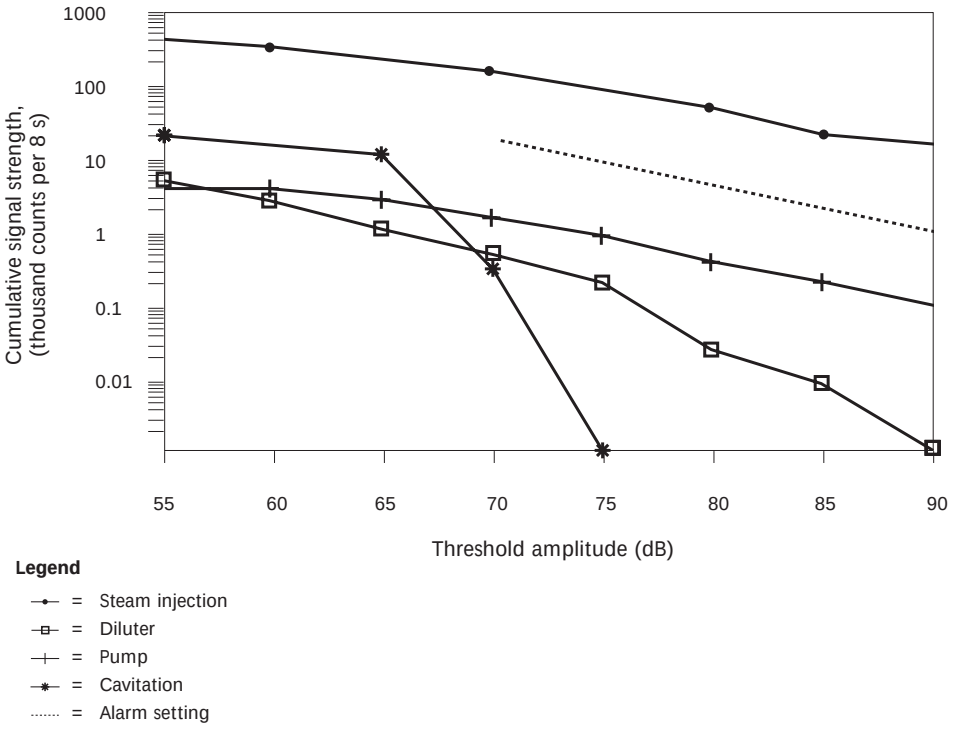
FIGURE 27. Combinations of detection thresholds and signal strength were defined for alarming on detection of leakage while ignoring extraneous emissions associated with startup, shutdown and other transient upset conditions.



waveguides were used to couple structure borne sound to the sensors.

During the feasibility study, several other sources of noise were studied to identify the potential for false calls In particular were possible noise sources due to the operation of a drain pump, noise transmitted from a diluter and cavitation of a valve. As a result of this study, combinations of detection thresholds and signal strength (as shown in Fig. 27) were defined for alarming on detection of a leak while ignoring extraneous emissions associated with startup, shutdown and other transient upset conditions.

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12

CHAPTER

Infrared Thermographic Leak Testing

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PART 1. Advantages and Techniques of Infrared Thermographic Leak Testing

A body emits thermal radiation, the largest part of which is in the form of infrared waves, with wavelengths in the rage of 1 to 50 µm.

Infrared thermographic leak testing techniques are accurate and cost effective processes for water, sewer, steam, petroleum, chemical and gas pipeline rehabilitation programs and for locating leak discontinuities in storage facilities and manufacturing programs.[1-3] These techniques have been used to test petroleum transmission pipelines, chemical plants, water supply systems, steam power plants, natural gas pipelines and sewer systems.

Thermographic technology makes it possible to inspect large areas, from remote distances, with 100 percent coverage. In addition, certain infrared thermographic techniques have the capability to locate voids and erosion areas surrounding buried pipelines, making their testing capabilities unique and highly desirable.

Infrared thermographic leak testing techniques can be divided into three main categories: (1) infrared emission pattern techniques, (2) infrared absorption techniques and (3) infrared photoacoustic techniques. The first two techniques rely upon using an infrared thermographic imager to image either the infrared energy emitted by a leak and the effect it has on its surroundings or to absorb a specific frequency of infrared energy. Both techniques have the following aspects in common.

1. They are accurate.
2. They are noncontacting and nondestructive.
3. They are used to inspect large areas as well as localized areas.
4. They are efficient in terms of both labor and equipment.
5. They are economical.
6. They are not obtrusive to the surrounding environment.
7. They do not inconvenience the pipeline's users or the production process.

The third technique is based on using a laser with a specific frequency in the infrared spectrum to cause leaking gas to emit an acoustic signal.

Their differences come into play on the types of leaks they are used for and the auxiliary equipment used with the basic infrared thermographic imagers.

The first category, based on infrared emission pattern techniques, uses an infrared imager to view large ground surface areas and lets the operator look for general thermal anomalies, either hotter or colder than the surrounding background surfaces, that could indicate subsurface pipeline leaks. This technique can be used with portable imagers, truck mounted imagers or helicopter and fixed wing mounted infrared imagers. The decision as to whether to look for anomalies hotter or colder than background is determined with knowledge of the type of leak being sought, the ambient conditions and the time of day. This technique has been used to investigate up to 800 km (500 mi) of pipeline daily for leaks.

The second category, based on the absorption of specific infrared frequencies in the thermal spectral bands, emitted from a combination infrared emitter and infrared imager, uses the infrared imager to view small and medium size areas and lets the operator look for areas where the image is black or missing, because of the absorption of the visualizing energy. Imagers can be hand carried or can be mounted on inspectors or trucks. This technique is specifically designed to locate leaks in a variety of situations, such as locating fugitive emission leaks in chemical plants or small gas leaks in manufacturing and assembly operations.

The third category is based on using a tuned laser to excite a specific leak testing gas in a repetitive manufacturing process, such as air conditioning heat exchanger testing. The excitation of the gas by the tuned laser causes the tracer gas to emit a specific acoustic signature that can be picked up by nearby microphones. From the information gathered, the exact location of the leakage can be accurately determined.

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PART 2. Infrared Leak Testing Using Emission Pattern Techniques

Principle of Operation

Effects of Subsurface Conditions on Temperature Measurement

An infrared thermographic imaging system measures the energy emitted from a ground surface only. But the temperatures that are measured on the surface of the ground above a buried pipeline depend on the subsurface conditions.

The subsurface configuration effects are based on the theory that energy cannot be stopped from flowing from warmer to cooler areas and that it can only be slowed down by the insulating effects of the material through which it flows. Various types of construction materials have different insulating abilities. In addition, differing types of pipeline discontinuities have different insulating values.

There are three ways of transferring energy: (1) conduction, (2) convection and (3) radiation. Good solid backfill should have the least resistance to conduction of energy and the convection effects should be negligible. The various types of problems associated with soil erosion and poor backfill surrounding buried pipelines increase the insulating ability of the soil by reducing the energy conduction properties without substantially increasing the convection effects. This is because dead air spaces or voids do not allow the formation of substantial convection currents.

An energy flow must start with an energy source. Because buried pipeline testing can involve large areas, the heat source should be both low cost and able to distribute heat evenly in the ground surface above the pipeline. The sun fulfills both of these requirements. Allowing the sun to warm the ground surface above the pipeline areas under test will normally supply all the energy needed. At night, the process may be reversed with the ground as the heat source and the night sky as the heat sink. This theory and methodology works best with pipelines carrying fluids at the same ambient temperature as the ground (i.e., natural gas, water or sewage).

For pipelines carrying fluids at temperatures above or below the ambient ground temperatures (i.e., steam, oil, liquified gases or chemicals), an alternative is to use the heat sinking ability of the earth to draw heat from the pipeline under test. The crucial point to remember is that the energy must be flowing through the ground. It doesn't matter what direction it is going.

Effects of Ground Cover on Temperature Measurement

The ground cover is a second important factor to consider for apparent temperature variations on the surface condition of the test area surfaces caused by emissivity changes.

It was mentioned earlier that there were three ways to transfer energy. Radiation is the technique that has the most profound effect on the ability of the surface to transfer energy. The ability of a material to radiate energy is measured by the emissivity of the material. This is defined as the ability of the material to release energy as compared to a perfect blackbody radiator. This is strictly a surface property. It normally exhibits itself in higher values for rough surfaces and lower values for smooth surfaces. For example, rough concrete may have an emissivity of 0.95 while a shiny piece of tin foil may have an emissivity of only 0.05. In practical terms, this means that when looking at large areas of ground cover, the engineer in charge of testing must be aware of differing surface textures caused by such things as broom roughed spots, tire rubber tracks, oil spots, loose sand and dirt on the surface and even the height of grassy areas and trees.

Effects of Environment on Temperature Measurement

The final system that affects the temperature measurement of a ground cover surface is the environmental system that surrounds the surface to be measured. Some of the various parameters that affect the surface temperature measurements are sunlight, clouds, ambient temperature, wind and moisture on the ground.

Solar Radiation. Testing should be performed during times of the day or night when the solar radiation or lack of solar radiation would produce the most

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rapid heating or cooling of the ground cover surface.

Cloud Cover. Clouds will reflect infrared radiation. This has the effect of slowing the heat transfer process to the sky. Therefore testing should be performed during times of little or no cloud cover to allow the most efficient transfer of energy out of or into the ground.

Ambient Temperatures. Atmospheric temperature should have a negligible effect on the accuracy of the testing because the important consideration is the rapid heating or cooling of the ground surface. This parameter will affect the length of time (i.e., the window) during which high contrast temperature measurements can be made.

Wind Speed. Wind has a definite cooling effect on surface temperatures. Measurements should be taken at wind speeds of less than 24 $km \cdot h^{-1}$ (15 $mi \cdot h^{-1}$).

Moisture on Ground. Moisture tends to disperse the surface heat and mask the temperature differences and thus the subsurface anomalies. Tests should not be performed while the ground has standing water.

Selection of Test Area

Once the proper conditions are established for imaging, a relatively large area should be selected for calibration purposes. This area should encompass both good and bad pipeline areas (i.e., areas with voids, delaminations, cracks or leaks). Each type of anomaly will display a unique signature depending on the conditions present.

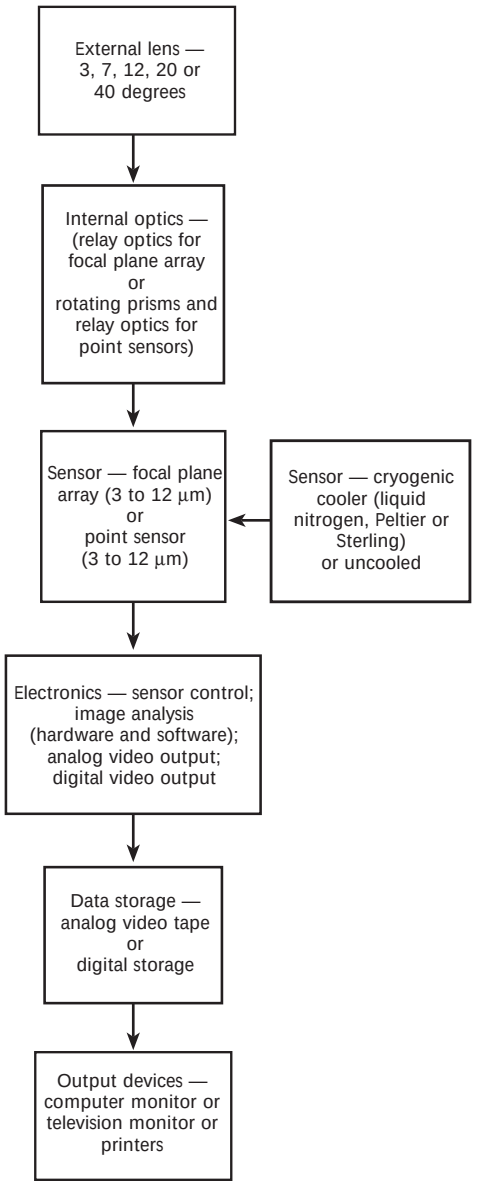
Test Equipment

To test ground cover for subsurface voids, pipeline leaks and other types of anomalies, all that is really needed is a sensitive contact thermometer. But, in even the smallest test area thousands of readings would have to be made simultaneously to outline the anomaly precisely. This means that to inspect large areas of ground cover efficiently a high resolution infrared thermographic imager is recommended. This type of equipment allows entire areas to be imaged and the resulting data to be displayed as pictures with areas of differing temperatures designated by differing gray tones on a black and white image or by various colors on a color image. A wide variety of auxiliary equipment can be used to facilitate the data recording, referencing and interpretation (Fig. 1).

The actual imaging and analysis system can be divided into four main subsystems. The first is the infrared sensor and optics head that normally can be used with interchangeable lens. It is similar in appearance to a portable video camera. The scanner's optical system, however, is transparent only to short wave infrared radiation in the spectrum field of 3.0 to 5.6 µm or the medium wave infrared spectrum field of 8 to 12 µm. These two spectrum bands are selected because outside of these ranges the thermal radiation emitted or reflected by objects is absorbed by the moisture in the atmosphere.

In addition, the imager's sensor is normally cooled to reduce the effects of background heating of the infrared sensor. Normally the infrared scanner's highly

FIGURE 1. Block diagram of typical Infrared scanner showing various major options.



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sensitive detector is cooled by liquid nitrogen or a mechanical Stirling cooler, to a temperature of –196 °C (–321 °F) and can detect temperature variations as slight as 0.05 °C (0.1 °F).

Alternate techniques of cooling the infrared radiation detectors are available which use either compressed gases or electric cooling. These last two cooling techniques may not give the same resolution because they cannot bring the detector temperatures as low as liquid nitrogen or the Stirling cooler. In addition, compressed gas cylinders may present safety problems while storing or handling.

The second major component of the infrared imaging system is a real time microprocessor coupled to a display monitor. With this component, cooler items being scanned are normally represented by darker gray tones, while warmer areas are represented by lighter gray tones. A color monitor may also be installed in the monitoring system to make the images easier to understand for those unfamiliar with interpreting graytone images. The color monitor will quantize the continuous graytone energy images into 2, 3 or more energy levels and assign them contrasting visual colors representing relative thermal energy levels.

The third major component of the infrared imaging system is data acquisition and analysis equipment. It is composed of an analog-to-digital converter, a digital computer with high resolution color monitor and storage and analysis software. The computer allows the transfer of moving instrumentation video tape or live images of infrared scenes to single frame computer images. The images can then be stored individually and later retrieved for enhancement and individual analysis. The computer allows the engineer in charge of testing to set specific analysis standards based on destructive sample tests, such as corings, and apply them uniformly to every square centimeter of ground cover. Standard off-the-shelf image analysis programs may be used or custom written software may be developed.

The fourth major component system consists of various image recording and retrieving devices. These should be used to record both visual and thermal images. They may be composed of video tape recorders, still frame film cameras with either instant and 35 mm or larger formats or computer printed images.

All of the above equipment may be carried into the field or parts of it may be left in the laboratory or office for additional use. If all of the equipment is transported to the field to allow simultaneous data acquisition and analysis, it is prudent to use an automotive van to set up and transport the equipment. The van should also include a technique to elevate the scanner head and accompanying video camera to allow scanning of the widest area possible, depending on the system optics used. The equipment may also be transported by fixed wing aircraft or helicopters, depending on the length of pipeline to be inspected.

Several manufacturers produce infrared thermographic equipment. Each manufacturer's equipment has its own strengths and weaknesses. These variations are in a constant state of change as each manufacturer alters and improves equipment. Therefore, equipment comparisons should be made before purchase.

Equipment Considerations

Items of major importance when selecting equipment include the following.

Thermal Resolution. The smaller the better.

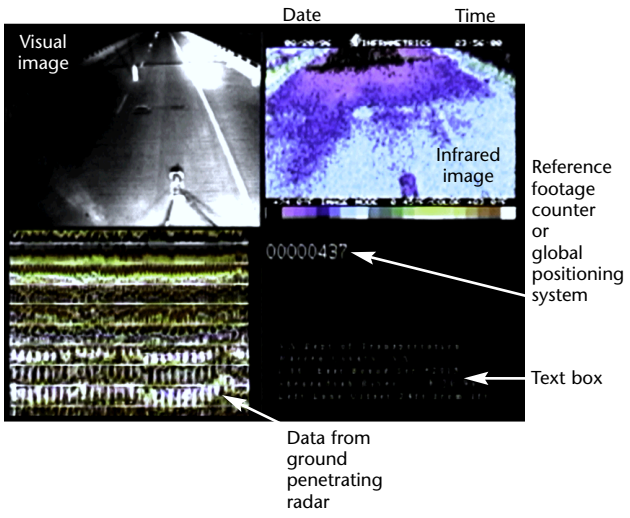
Spatial Resolution. The smaller the better.

Field of View. Appropriate to requirements of the job.

Data Collection Format — Analog or Digital. Analog lets more data be collected and stored at less cost but detail information may be lost in the storage process.

Data Synchronization between Data Sets. The data sets include infrared thermographic data, normal image data, reference data such as global positioning system (GPS) information, meter distance counters etc. (Fig. 2). Data synchronization is critical because at

FIGURE 2. Screen data collection system developed for thermographic inspection.



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normal video rates of data collection, 60 fields per second, if separate video and infrared thermographic recorders are used and are 1 s off in synchronization, then the images can be looking at areas thousands of feet out of synchronization, making the data worthless.

Software. Software is used for data analysis and presentation.

Leak Testing of Pipelines

Buried Water Pipeline

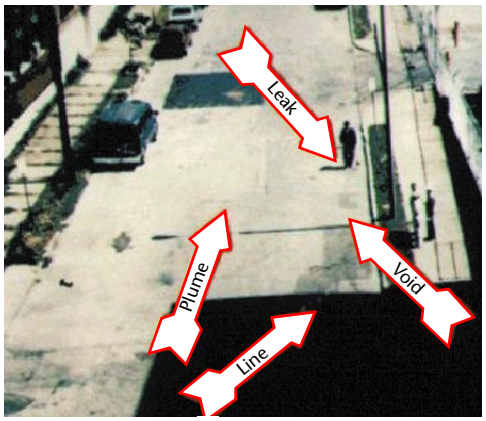
In 1983, an infrared thermographic leak and erosion void investigation was performed on Duncan street in midtown St. Louis. Before the inspection, crews from the Metropolitan St. Louis Sewer District had observed street pavement sinking up to 150 mm (6 in.) along a 183 m (600 ft) long section of Duncan Street. Visual inspections using both television cameras and crawl crews had located only three dime sized water infiltration points in the 1.5 m (5 ft) diameter sewer located about 4.0 m (13 ft) below the surface. Running alongside the sewer was a pressurized water line.

During the thermographic investigation a cool area was located perpendicular to the buried water pipe. It began at the water line and spreading outward toward the sewer line. It was determined that the cooler surface area was caused by the heat sinking ability of the water plume as it spread out from the water line leak and flowed down the outside of the nearby sewer pipeline. Some of the fresh water was entering the sewer line through the three dime sized holes that the crawl crew had located.

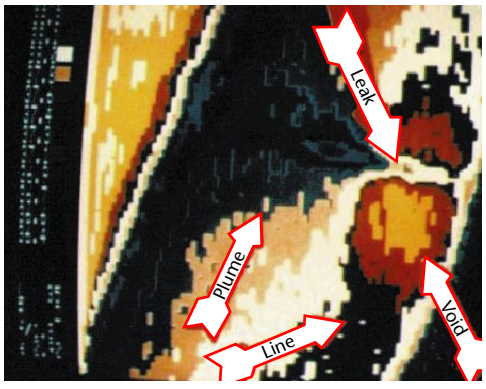
In addition to the water leak, the infrared thermographic investigation also located an erosion area above the water line. Evidently the water flowing from the water pipeline to the sewer pipeline was carrying soil, which was washing away down the sewer line. This void area had caused some of the pavement sinking and further street collapse was inevitable. The void above the water line was evidenced by a warmer signature in the thermographic image (Fig. 3).

FIGURE 3. Surface images showing water pipeline, water leakage, leakage plume and void area forming above pipeline: (a) visual photograph; (b) infrared thermographic image of surface.

(a)



(b)



Buried Drain Pipeline

In May 1990, at an airport in New England, the landing gear of a DC-10 carrying a full load of passengers fell through the taxiway pavement while approaching its unloading gate (Fig. 4). Damage to the airplane cost $500 000 and included areas of the landing gear, fuselage and fuel system leaks.

Upon removal of the passengers and containment of the leaking fuel, airport authorities removed the airplane. During the removal process, it was determined that a 1.8 m (6 ft) by 1.8 m (6 ft) by 2.4 m (8 ft) deep void had formed underneath the pavement because of leaks and

FIGURE 4. Airplane landing gear collapsing into a taxiway void caused by drain pipe infiltration leakage void.

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infiltration of the soil into a 40-year-old buried storm water drainage pipe. When it was determined that the drainage system was located throughout the entire airport pavement system, airport authorities and their consultants concluded that more drainage system leaks and erosion areas probably existed. Airport authorities then requested that the consultants determine a technique of locating the leaks and possible voids with 100 percent coverage, without interrupting airport traffic.

The inspection of over 185 800 m^2 (2 000 000 ft^2) of pavement was conducted by using infrared thermographic techniques at night, after 11:00 p.m. when air traffic was at a minimum. The entire investigation took three nights and uncovered twelve subsurface voids of varying sizes, some of which could have caused major damage to airplanes if they had collapsed (Fig. 5).

Buried Natural Gas Pipeline

In 1985, an investigation of 3.2 km (2 mi) of six lane concrete pavement was conducted through the main downtown area of Belleville, Illinois. The main purpose of this inspection was to locate any anomalies (i.e., utility pipeline leaks or voids beneath the city street) that could collapse or cause the need for repairs after a proposed street resurfacing project took place. Several utilities were located beneath the city streets including sewage, water and natural gas.

While inspecting the areas containing buried natural gas pipelines, the infrared thermographic equipment was set up to locate areas cooler than normal, under the hypothesis that pinhole leaks in a pressurized natural gas pipeline would cool the surrounding soil due to the venturi cooling as the gas escaped and expanded as it left the pipeline.

The entire field portion of the project took only one night and located, along with other anomalies, two natural gas pipeline leaks, one of which is shown in Fig. 6.

FIGURE 5. Leaking drain pipe at airport: (a) visual image; (b) thermogram.

(a)

(b)

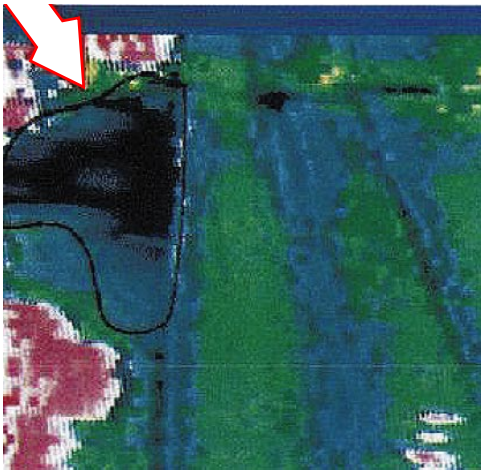
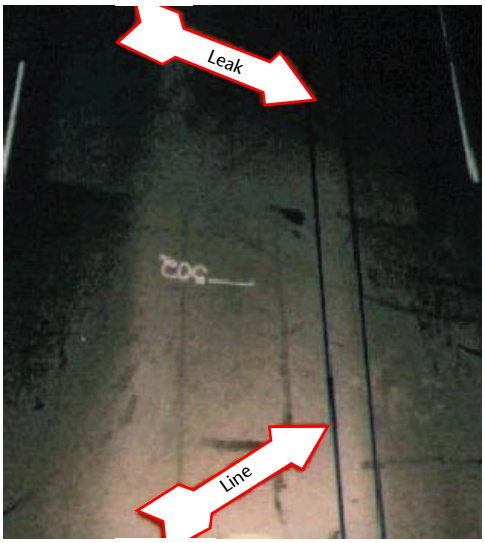
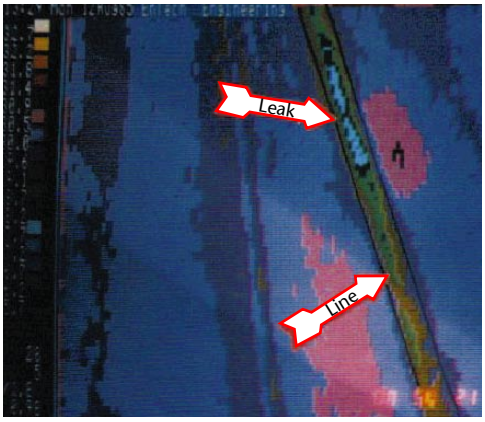


FIGURE 6. Buried natural gas pipeline and pipeline leakage in downtown Belleville, Illinois: (a) visual photograph; (b) thermogram.

(a)



(b)



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Buried Hot Water Pipeline

In 1986, the State of Utah used infrared thermography to inspect the hot water, radiant heat system used to heat steps, driveways and roads near the state capital buildings in Salt Lake City, Utah. These pavements were heated during the winter months to melt ice and snow before it could become dangerous to pedestrians and automobile traffic.

The 20 year old system normally worked properly but was beginning to show its age by higher than normal water usage, higher than normal boiler fuel bills and higher than normal quantities of boiler chemical additives used to reduce pipe fouling.

Several water leaks were detected as evidenced by expected warm spots on the thermographic images. Most leaks though, were more difficult to locate because they did not start with a hot spot and radiate in a circular pattern from the leaks. Instead, these leaks started with a smaller warm spot and spread out along the pipeline for just a short distance. It was determined that significantly smaller leaks had not cracked the pipeline concrete encasement but rather had exited the water pipe and traveled along the outside of the pipeline until they found an exfiltration point somewhere downstream in the pipe casement. The reason the heat dissipated so quickly was that the line acted as a heat sink and brought the outside water temperature to the temperature of the line very quickly (Fig. 7).

Buried Steam Pipeline

In 1981, Union Electric Company, the steam generating and distribution utility company in St. Louis, Missouri, used thermographic techniques to locate buried steam pipeline leaks. The steam distribution loop in downtown St. Louis was about 29 km (18 mi) long and 3.6 m (12 ft) below the pavement surface in most locations. It was the beginning of winter and several large industrial customers downstream of where the line crossed Seventh Street along Washington Avenue complained of a lack of capacity. Union Electric personnel were able to localize the leak to an area between two manholes 78 m (256 ft) apart (Fig. 8).

Infrared thermographic techniques were used to locate the leak without digging or halting traffic on the major downtown street. The inspection was performed from a nearby parking garage rooftop and occurred at about 5:00 p.m. It took less than 10 min to locate and mark the pavement above what turned out to be a major leak on the bottom of a 0.3 m (12 in.) insulated pipeline buried 3.6 m (12 ft) below the surface. The major signature of the thermographic images was a central hot spot and gradual cooling along the pipeline length.

Buried Oil Cooled Electric Cable

In 1989, infrared thermography was used to locate leaks in a buried 400 kV-A electric cable which carried power for 25 percent of the city of Rome, Italy. Due to the importance and high current carrying capacity of this cable, it was designed

FIGURE 7. Thermogram of buried hot water pipeline grid used to melt snow and ice on roadway pavement.

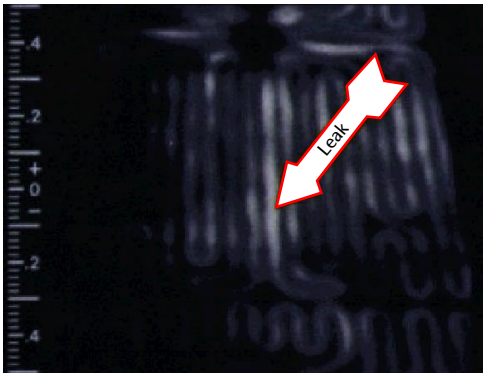
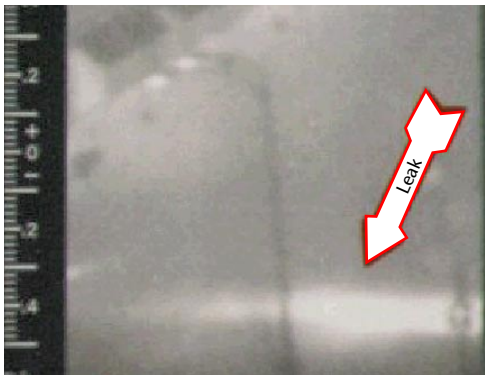


FIGURE 8. Buried steam pipeline leakage, St. Louis, Missouri: (a) visual photograph; (b) thermogram.

(a)

(b)



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with a circulating oil filled cooling system. Whenever leaks occurred in the system, controls automatically shut off electricity to the line, effectively shutting down 25 percent of the power to the city of Rome. Leaks normally took up to 48 h to locate and repair.

During the test, which was performed at night due to the high traffic volume during daylight hours, one leak was detected as evidenced by a temperature higher than the average ground cover temperature (Fig. 9). This area was brought to the attention of the authorities. It was confirmed that the area located was the site of a previous oil leak. It was certain that the images that were recorded were caused by the small pools of oil due to the leak. This site was determined to be the site of the active leak and contained about 1 L (0.25 gal) of oil.

The inspection process, including equipment setup, calibration and scanning, took about 30 min for 180 m (600 ft) of pipeline inspected.

Buried Petroleum Pipeline

In November 1990, infrared thermography was used to inspect a 7.3 km (4.5 mi) section of subsurface oil supply pipeline for a large Illinois refinery. The purpose of the investigation was to locate the cause of a drop in line pressure. The sudden drop in line pressure was believed to be caused by a leak in the subsurface oil transmission pipeline system.

Because of the rough terrain, the investigation was performed from a helicopter at an altitude of 300 m (1000 ft). With the aid of telephoto and wide angle optics, the 7.25 km (4.5 mi) section of pipeline was field inspected in less than 30 min. The results of the inspection included several small oil line leaks and one substantial pipeline leak estimated at 4.1 $L \cdot s^{-1}$ (65 $gal \cdot min^{-1}$) (Fig. 10). In addition to locating the leak precisely, the infrared thermographic techniques helped determine how much soil had been contaminated and what the rate of contamination spread was over time.

FIGURE 9. Oil leakage in buried, oil cooled electrical cable, Rome, Italy: (a) visual photograph; (b) thermogram.

(a)

(b)

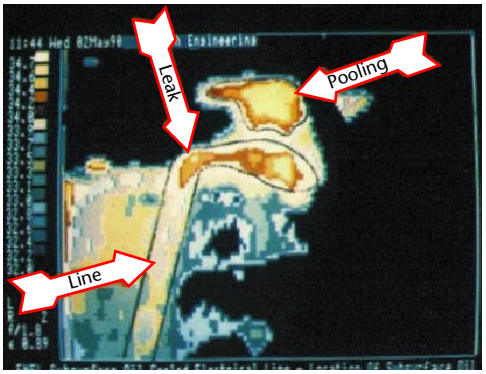
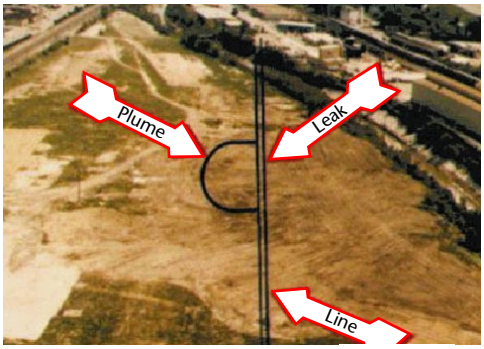


FIGURE 10. Buried oil pipeline, pipeline leakage and leakage plume: (a) visual photograph; (b) thermogram.

(a)



(b)

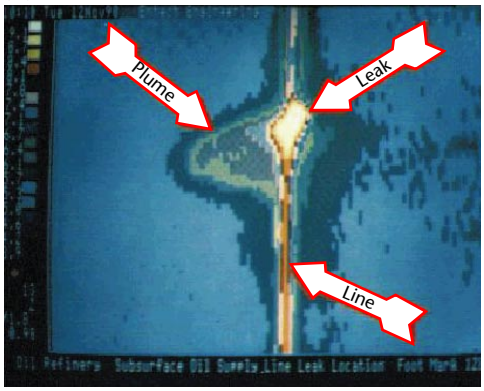


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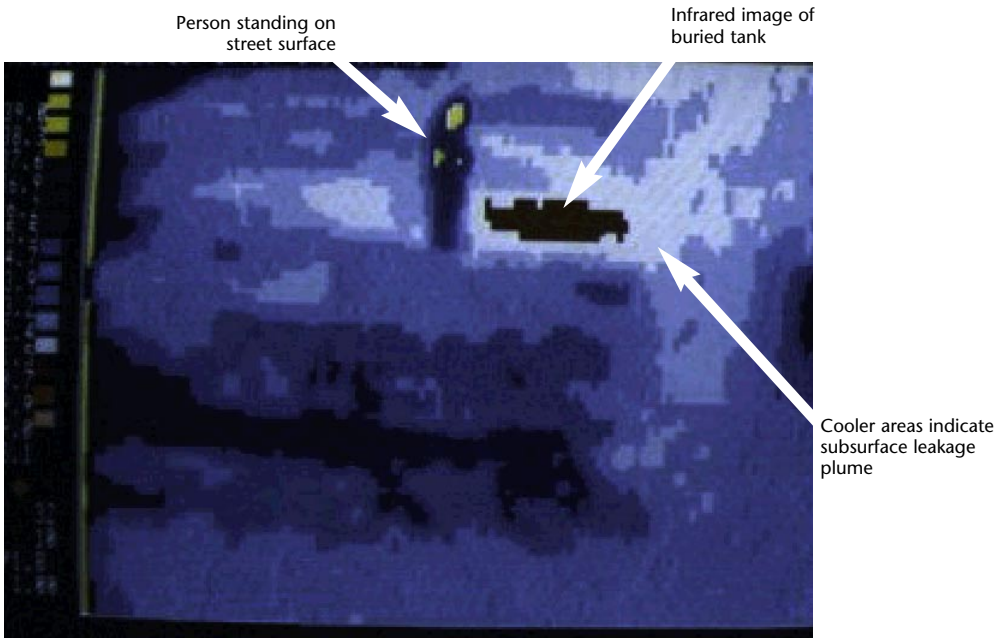
FIGURE 11. Thermogram of abandoned, buried gasoline tank, Belleville, Illinois.



Leaking Underground Storage Tank

In 1986, infrared thermographic techniques were developed to investigate 3.2 km (2 mi) of a six lane concrete pavement through the main downtown area of Belleville, Illinois. The purpose of this inspection was to locate any anomalies underneath the street that might cause future problems after the street was resurfaced.

During the investigation several anomalies were located including an abandoned and leaking gasoline tank about 3 m (10 ft) below the surface. The thermogram illustrating the tank and leak plume showed cooler areas where the chemical plume and tank were located (Fig. 11). When the tank was dug up and removed, it showed large areas of rust, a hole in one side about 350 mm (14 in.) from the bottom. It still contained about 750 L (200 gal) of petroleum materials.

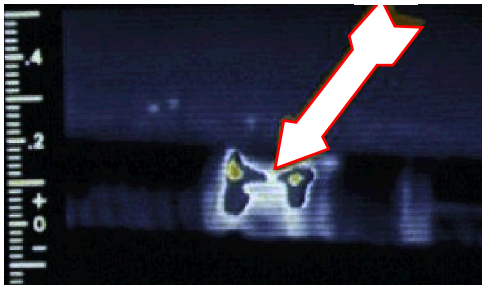
Aboveground Chemical Pipeline

In 1985, infrared thermography was used to locate small pipeline leaks and insulation problems in the world's longest, above ground pipeline used to transport liquid sulfur from a Chevron refinery in Carter Creek, Wyoming. The 34 km (21 mi) long pipeline, across the badlands of Wyoming was critical to the uninterrupted output of the refinery.

Two four wheel vehicles were used to carry engineers and equipment. One was used as a main vehicle and the other as a safety vehicle to help get the team over rough areas and out of waist deep mud holes caused by intermittent rains. The four wheel drive vehicles were used because weather conditions did not allow use of a helicopter.

During the investigation, which took about four days because of rain and rough terrain, several small leaks and insulation problems were located by their elevated temperature profiles (Fig. 12). Problems with the heat tracing equipment were located by its lack of heat in certain cables. Electrical panels supplying power to the outside heat tracing equipment were also inspected for loose connections and discontinuous components as evidenced by their elevated temperatures.

FIGURE 12. Thermogram of leakage in sulfur pipeline, Carter Creek, Wyoming.



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PART 3. Leak Testing Using Infrared Absorption[4]

Principle of Operation

The concept of using backscatter/absorption gas imaging (BAGI) was developed by the United States Department of Energy and transferred to the private sector for commercialization in the late 1980s. This technique is designed to locate leaks by making the normally invisible gas leakage visible on a standard video display of the region of interest. This image of the escaping gas lets the operator quickly identify the location of the leak. The system is not designed to determine the gas concentration values of the leakage.

The principle of operation of the technique is the production of a video image by backscattered laser radiation where the laser wavelength is strongly absorbed by the gas of interest. When achieved, the result is that the normally invisible gas becomes visible on a standard television monitor. The technique has three basic constraints: (1) there must be a topographical background against which the gas is imaged, (2) the system must operate in an atmospheric transmission window and (3) the gas of interest must absorb the laser radiation. Imaging equipment in the infrared wavelengths fulfills these needs.

FIGURE 13. Backscatter/absorption gas imaging system.

Table 1 shows a list of detectable gases, their maximum safe concentrations and their minimum detectable concentrations.[5,6]

Infrared Absorption Test Instrument

Investigation equipment consists of a tunable infrared laser coupled to an infrared imager (Fig. 13). Typically the optics of the imager and laser are optically coupled to let the units transmit the infrared laser radiation to the area of interest and to then receive the reflected laser energy. Typically an area consisting of a 14 × 18 degree field of view up to 30 m (100 ft) from the transmitter/receiver may be scanned.

The laser typically used in the gas imaging system is a tunable 5 W carbon dioxide waveguide laser. Using a low power laser is possible because the optical arrangement permits the laser beam and the instantaneous field of view of an infrared radiation detector to be scanned in synchronization across the area of interest. The instantaneous field of view produced by the typical small (0.05 × 0.05 mm) infrared radiation detector and a collimating lens is scanned in a raster like fashion across the target area by two orthogonally positioned horizontal and vertical scan mirrors. This ensures that the detector field of view and the laser beam are in perfect synchronization and that the laser need irradiate only that region of the target area viewed by the detector. This keeps the laser power requirements to a minimum and makes the system totally safe for eyes.

Application to Leak Testing of Pipelines

When the infrared thermographic investigation technique is used in the infrared absorption mode, a tunable laser must be coordinated with the infrared imager. In this mode, the laser is tuned to emit a specific frequency of diffused infrared radiation that will be absorbed by the gas being sought (see Table 1). The laser is then scanned across the area being investigated. When the laser radiation is absorbed by gas escaping from a leak, the infrared image is lost or turns black on

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TABLE 1. Infrared radiation absorption of detectable gases.

Gas	Chemical Formula	Safety Threshold[a] ($\mu L \cdot L^{-1}$)	Laser Wavelength[b] (μm)	Detector Sensitivity ($\mu L \cdot L^{-1} \cdot m$)[c]	Detector Sensitivity ($kg \cdot yr^{-1}$)[d]
Acetaldehyde	C_2H_4O	25	9.210 09	436	297
Acetonitrile	CH_3CN	40	9.293 79	1000	636
Acrolein	CH_2:CHCHO	0.1	10.288 80	148	128
Acrylonitrile	CH_2CHCN	2	10.303 47	86	71
Allyl alcohol	C_3H_6O	2[e]	9.694 83	69	62
Ammonia	NH_3	25	10.333 70	13	4
Amyl acetate	$C_7H_{14}O_2$	100	9.458 05	46	93
Arsine	AsH_3	0.05	10.513 12	79	95
Benzene	C_6H_6	10	9.639 17	208	251
Butane	C_4H_{10}	800	10.349 28	772	694
T-butanol	$(CH_3)_3COH$	100	10.741 12	108	124
Carbonyl difluoride	COF_2	2	10.233 17	76	78
Chlorobenzene	C_6H_5Cl	10	9.200 73	82	142
Chloroprene	C_4H_5Cl	10[e]	10.260 39	46	63
Cyclohexane	C_6H_{12}	300	9.621 22	1000	1302
Cyclopentane	C_5H_{10}	600	10.741 12	4380	4752
O-dichlorobenzene	$C_6H_4Cl_2$	25	9.260 53	79	179
Trans 1,2-dichloroethylene	$C_2H_2Cl_2$	200	10.764 06	160	238
Dimethylamine	$(CH_3)_2NH$	5	9.753 26	485	338
P-dioxane	$C_4H_8O_2$	25[e]	9.210 09	190	259
Ethyl acetate	$CH_3COOC_2H_5$	400	9.458 05	34	46
Ethyl acrylate	CH_5:$CHCOOCH_2CH_3$	5	9.317 25	57	91
Ethyl alcohol	C_2H_5OH	1000	9.503 94	61	43
Ethylene	C_2H_2	5500	10.532 09	15	6
Ethylene chlorohydrin	C_2H_5ClO	1[e]	9.249 95	45	56
Ethylene dichloride	C_2H_4Cl	10	10.494 49	1895	1850
Ethylene oxide	$(CH_2)_2O$	1	10.859 78	651	445
Ethyl ether	C_2H_6O	400	9.210 09	119	107
Ethyl mercaptan	C_2H_5SH	0.5	10.194 58	730	702
Formic acid	HCOOH	5	9.219 69	24	16
Furan	C_4H_4O	——	10.182 31	100	105
Germane	GeH_4	——	10.696 39	219	254
N-hexane	C_6H_{14}	50	9.341 76	2205	2939
Hydrazine	N_2H_4	0.01[e]	10.440 59	55	27
Hydrogen selenide	H_2Se	0.05	9.157 45	758	905
Isopropanol	$(CH_3)_2CHOH$	——	10.494 49	110	102
Methacrylonitrile	CH_2:$C(CH_3)CN$	——	10.785 16	31	32
Methanol	CH_3OH	200[e]	9.675 97	19	9
Methyl acetate	$C_3H_6O_2$	200	9.519 81	51	58
Methyl bromide	CH_3Br	5	10.696 39	402	586
Methyl chloride	CH_3Cl	50	9.603 57	1020	791
Methyl chloroform	CH_3CCl_3	350	9.200 73	26	53
Methylethylketone	$CH_3COC_2H_5$	200	10.591 04	343	383
Methyl methacrylate	$CH_2C(CH_3)COOH_3$	100	10.611 39	62	85
Monochloroethane	C_2H_5Cl	——	10.274 45	126	125
Monomethylamine	CH_3NH_2	——	9.219 69	174	84
Monomethylhydrazine	CH_3NNH_2	——	10.333 70	120	84
Orthodichlorobenzene	$C_6H_4Cl_2$	——	9.621 22	54	122
Ozone	O_3	0.1	9.503 95	33	25
Pentane	C_5H_{12}	600	9.675 97	4240	4732
Perchloroethylene	C_2Cl_4	25	10.741 12	85	217
Phosgene	$COCl_2$	0.1	10.233 17	318	509
Phosphine	PH_3	0.3	9.694 83	104	55

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TABLE 1. Infrared radiation absorption of detectable gases, continued.

Gas	Chemical Formula	Safety Threshold[a] ($\mu L \cdot L^{-1}$)	Laser Wavelength[b] (μm)	Detector Sensitivity ($\mu L \cdot L^{-1} \cdot m$)[c]	Detector Sensitivity ($kg \cdot yr^{-1}$)[d]
Propane	C_3H_8	——	10.811 11	2900	2000
Propylene	C_3H_6	——	10.674 59	174	113
Propylene oxide	C_3H_6O	20	10.513 20	332	175
Refrigerant-11	CCl_3F	1000	9.229 53	12	25
Refrigerant-12	CF_2Cl_2	1000	10.764 06	9	17
Refrigerant-13	$CClF_3$	1000	11.085 63[f]	336	542
Refrigerant-22	$CHClF_2$	1000	10.832 93[f]	564	752
Refrigerant-13B1	$CBrF_3$	1000	9.219 69	3	7
Refrigerant-113	$C_2Cl_3F_3$	1000	9.603 57	21	61
Refrigerant-114	$(CClF_2)_2$	1000	9.503 94	15	40
Styrene	$C_6H_5CHCH_2$	50	10.858 11	152	245
Sulfur dioxide	SO_2	2	9.219 69	3790	3759
Sulfur hexafluoride	SF_6	1000	10.551 40	0.4	1
Sulfuryl fluoride	F_2O_2S	5	9.249 95	2241	3543
Toluene	$C_6H_5CH_3$	50[e]	9.621 22	622	887
1,1,2 trichloroethane	$CH_2ClCHCl_2$	10[e]	9.239 61	34	67
Trichloroethylene	C_2HCl_3	50	10.591 04	33	66
Trimethylamine	$(CH_3)_3N$	5	9.586 23	101	92
Unsymmetrical dimethylhydrazine	$(CH_3)_2NNH_2$	0.01[e]	10.835 24	106	99
Vinyl acetate	$CH_3CO_2CH{:}CH_2$	10	9.714 00	44	75
Vinyl bromide	C_2H_3Br	5	10.611 39	102	168
Vinyl chloride	C_2H_3Cl	5	10.611 39	48	46
Vinylidene chloride	$CH_2{:}CCl_2$	5	9.210 09	31	46
Xylene	$C_6H_4(CH_3)_2$	100	9.535 97	479	787

a. Threshold limit value (TLV) expressed as a time weighted average (TWA), according to American Council of Governmental Industrial Hygienists.[5,6]
b. $^{12}CO^{16}O_2$ laser unless otherwise noted.
c. Average concentration for a 1 m (40 in.) thick cloud.
d. Minimum observable leak rate for gas at standard temperature and pressure; airspeed = 50 $mm \cdot s^{-1}$ (10 $ft \cdot min^{-1}$), range = 5 m (16.4 ft), right angle viewing and uniform background.
e. Threshold limit value for skin.
f. $^{13}C^{16}O_2$ laser.

FIGURE 14. Backscatter/absorption gas imaging system, viewing gas leakage from bank of gas storage tanks.

the image. The entire path from the leak point through the plume should be able to be imaged.

Figure 14 illustrates a backscatter/absorption gas imaging system viewing a gas leak occurring in a bank of gas storage tanks. The television monitor in the lower right corner shows the live image viewed by the operator showing the leak as a black plume. The plume is black because the laser energy has been absorbed by the gas and cannot return to the infrared thermographic imager as does the rest of the laser energy.

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PART 4. Infrared Thermographic Leak Testing Using Acoustic Excitation[7]

Infrared Photoacoustic Leak Testing

The photoacoustic effect was first observed by Alexander Graham Bell in 1880 and occurs whenever a gas absorbs radiation. The radiation energy absorbed by the gas produces local temperature and pressure disturbances, which if of sufficient magnitude, produce a pressure or acoustic, wave that may be detected by a microphone.

The magnitude of the acoustic emission is determined by the amount of laser energy absorbed by the leaking gas. The amount of absorbed radiant energy depends on the concentration within the volume of gas illuminated by the laser beam. If the leakage plume is larger than the laser beam cross section, then the appropriate gas volume is determined by the thickness of the gas and the laser beam diameter. If the leakage plume is smaller than the laser beam diameter, then its dimensions alone determine the absorption volume. The gas concentration within the irradiated volume is determined by the dispersion of the tracer gas as it leaves the leak point and mixes with the ambient air. Furthermore, if the laser radiation is reflected by the product surface in the vicinity of the leak, some of it may pass back through the leakage plume, resulting in additional energy being absorbed.

Test Equipment

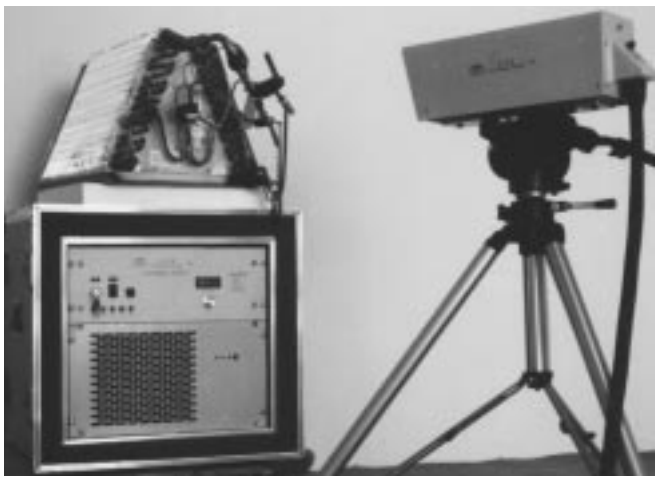
The basic components of a system used to exploit the photo acoustic effect include a carbon dioxide laser that scans a linear pattern so that a product under test is completely illuminated as it passes through the beam scan pattern. A microphone, with associated signal processing electronics, is positioned in the general area of the product as it is being illuminated.

In general, the product under test is pressurized with a gas, such as sulfur hexafluoride, which strongly absorbs the infrared radiation produced by the carbon dioxide laser. If the product has a leak, the leaking gas will absorb the laser radiation as it passes through the line scan pattern. The laser energy absorbed by the gas produces an acoustic emission which propagates away in all directions. Furthermore, the frequency of this acoustic emission corresponds to the frequency of the laser beam scan rate. This periodic acoustic emission is detected by the microphone and processed by an electronic circuit that uses synchronous detection technology. The resulting leak indication signal may be used as an alarm to automatically eject the faulty product from the assembly line.

Tests indicate that a typical photoacoustic system can detect sulfur hexafluoride leaks as small as 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}). It is quite rapid, because it is restricted only by the speed of sound and the time required to completely scan the product under test. Small products may be examined for leaks in the 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}) range in time approaching 0.2 s per product.

A combination leak alarm and pinpointing configuration technique is possible by combining the laser beam position information within the scan pattern with the signal processing unit. The probe beam position information is used to determine the exact location of the leak. Because the magnitude of the acoustic emission is directly proportional to the size of the leak, this technology offers the capability to automatically alarm if a leak is detected, to pinpoint the location of the leak and to measure the leakage rate.

FIGURE 15. Photoacoustic setup for inspection of air conditioner coils.



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Application to Manufacturing and Assembly

Figure 15 is a photograph of an air conditioning heat transfer coil manufacturing assembly test system. In this test setup, the coil is pressurized with sulfur hexafluoride gas. When the probe laser beam passes over the point of a leak, it causes the leaking tracer gas to emit a specific acoustical sound that is detected by the system's one or more microphones. With this technology, fully automated leak testing and location is possible without the need for operator input.

Summary of Infrared Thermographic Leak Testing

Infrared thermography can be used to detect buried and aboveground pipeline discontinuities such as leaks, cracks and subsurface erosion voids.

Infrared thermography can also be used to detect gas leaks in production processes.

Infrared thermographic testing techniques are considered nondestructive.

Infrared thermographic testing may be performed during day or night, depending on environmental conditions and the desired results.

Computer analysis of thermal images greatly improves the accuracy and speed of test interpretations.

Computer analysis of pipeline thermographic data can improve the ability to set repair priorities for areas in a state of change.

Aging chemical, oil, natural gas, water, steam and sewage pipeline infrastructures throughout the world are rapidly approaching the end of their design lives. This will necessitate more efficient and cost effective techniques of testing pipelines under load and in place. Infrared thermography is a nondestructive, remote sensing technique that meets these requirements.

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CHAPTER

Leak Testing of Petrochemical Storage Tanks

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PART 1. Leak Testing of Underground Storage Tanks

Aboveground and Underground Storage Tanks

Leak testing of petrochemical storage tanks is an area of growing concern to the general public, the regulatory agencies that represent the public and the owners of the tanks. Petrochemical structures consist of various types of tanks and vessels for storage and processes in petroleum refineries and petroleum related chemical plants. Failures and liquid leakage from petrochemical tanks have on occasion caused contamination of soil and both groundwater and surface water supplies.

The costs of remediation following a leak or failure are very high. In some cases the challenges of remediation exceed the existing technologies, creating environmental problems that will remain for generations to come. The near term costs associated with preventing liquid leakage or detecting it early and at small quantities are modest in comparison to remediation costs.

In addition to concerns for liquid leakage, the petrochemical tank owner must address the leak tightness of the tank system with respect to product vapors. Product vapors can be an important air pollution concern. Vapors may also be a concern from a plant safety perspective including both fire hazard and vapor toxicity issues.

In the 1990s, underground storage tanks have received more regulatory attention than aboveground storage tanks. As regulatory agencies begin to address aboveground storage tank issues, some of the techniques currently used for underground tanks may find application aboveground.

Aboveground storage tanks are typically larger than underground tanks and are also easier for the inspector to access. The floor or bottom of the aboveground tank is typically an area of concern because it may easily corrode and begin leaking unobserved.

Because aboveground storage tanks and underground storage tanks are so different in both design and nondestructive testing approach, this chapter addresses the two separately.

Underground storage tanks have been the focus of considerable regulatory attention. The technology and regulations applicable to underground storage tank leak testing are constantly changing. The owners, operators and constructors of underground storage tanks should request the latest publications from the federal, state and local regulatory agencies that may have jurisdiction for a given tank location. In the United States the primary Federal regulatory agency concerned with leak testing of underground storage tanks has been the United States Environmental Protection Agency (EPA). This agency has published extensively on the topic of underground storage tank leak testing.[1] The following discussion of underground storage tanks is excerpted from Environmental Protection Agency publications.[2,3]

Described next are several techniques for monitoring leakage from underground storage tanks:[3] (1) secondary containment with interstitial monitoring, (2) automatic tank gaging systems, (3) vapor monitoring, (4) groundwater monitoring, (5) statistical inventory reconciliation, (6) tank tightness testing, (7) inventory control, (8) manual tank gaging and (9) leak testing for underground piping. Figure 1 shows most of these as if applied to the same tank.

Secondary Containment with Interstitial Monitoring

Secondary containment provides a barrier between the tank and the environment. The barrier holds the leak between the tank and the barrier so that the leakage is detected. The barrier is shaped so that a leak will be directed toward the interstitial monitor (see Fig. 2).

Barriers

There are four kinds of barriers.

1. In *double walled* or *jacketed* tanks, an outer wall partially or completely surrounds the primary tank.
2. Concrete vaults may be used with or without lining.
3. Internally fitted liners, or *bladders,* may be used.

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4. Leakproof excavation liners partially or completely surround the tank.

Clay and other earthen materials cannot be used as barriers.

Interstitial Monitors

Monitors are used to check the area between the tank and the barrier for leakage and alert the operator if a leak is suspected.

Some monitors indicate the physical presence of the leaked product, either liquid or gaseous. Other monitors check for a change in condition that indicates a hole in the tank, such as a loss of vacuum or a change in the level of a monitoring liquid between the walls of a double walled tank.

Monitors can be as simple as a dipstick used at the lowest point of the containment to see if liquid product has leaked and pooled there. Monitors can also be sophisticated automated systems that continuously check for leaks.

Regulatory Requirements

The barrier must be immediately around or beneath the tank. A double walled system must be able to detect a release through the inner wall.

The interstitial monitor must be checked at least once every 30 days.

An excavation liner must (1) direct a leak toward the monitor, (2) not allow the specific product being stored to pass through it any faster than 10^{-7} Pa·m^{3}·s^{-1} (10^{-6} std cm^{3}·s^{-1}), (3) be compatible with the product stored in the tank, (4) not interfere with the UST's cathodic protection, (5) always be above the groundwater and the 25 year flood plain and (6) have clearly marked and secured monitoring wells, if they are used.

Other Considerations

In areas with high groundwater or a lot of rainfall, it may be necessary to select a secondary containment system that completely surrounds the tank to prevent moisture from interfering with the monitor.

This technique works effectively only if the barrier and the interstitial monitor are installed correctly. Trained and experienced installers are necessary.

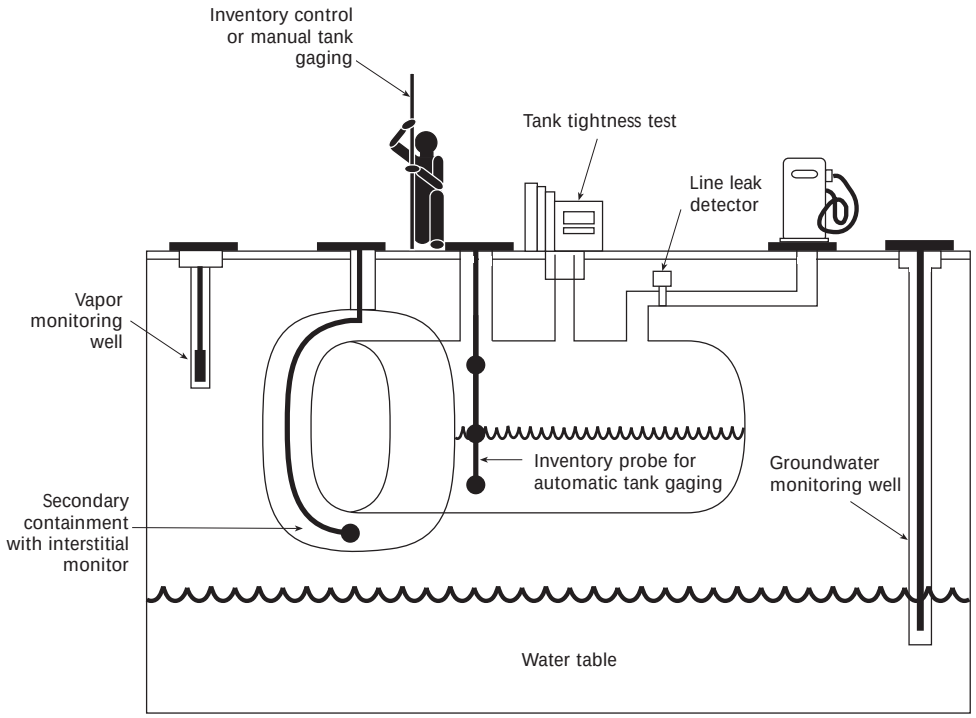
Automatic Tank Gaging Systems

Principle of Operation

The product level and temperature in a tank are measured continuously and automatically analyzed and recorded by a computer.

In the *inventory mode*, the automatic tank gaging system replaces the gage stick

FIGURE 1. Techniques for leak testing of underground storage tanks.[2]



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to measure product level and perform inventory control. This mode records the activities of an inservice tank, including deliveries.

In the *test mode,* the tank is taken out of service and the product level and temperature are measured for at least 1 h. Some systems, known as *continuous automatic tank gaging systems,* do not require the tank to be taken out of service to perform a test. This is because these systems can gather and analyze data during many short periods when no product is being added to or taken from the tank.

Regulatory Requirements

The automatic tank gaging system must be able to detect a leak no larger than 2×10^{-7} $m^3 \cdot s^{-1}$ or 0.8 $L \cdot h^{-1}$ (0.2 $gal \cdot h^{-1}$) with certain probabilities of detection and of false alarm. Some automatic tank gaging systems can also detect a leak of 1×10^{-7} $m^3 \cdot s^{-1}$ or 0.4 $L \cdot h^{-1}$ (0.1 $gal \cdot h^{-1}$) with the required probabilities.

Implementation

Automatic tank gaging systems have been used primarily on tanks containing gasoline or diesel, with a capacity of up to 57 m^3 (15 000 gal). If considering using an automatic tank gaging system for larger tanks or products other than gasoline or diesel, discuss its applicability with the manufacturer's representative.

Water around a tank may hide a leak by temporarily preventing the product from leaving the tank. To detect a leak in this situation, the automatic tank gaging system should be able to detect water in the bottom of a tank.

The automatic tank gaging system probe is permanently installed through an opening (not the fill pipe) on the top of the tank (see Fig. 3). Each tank at a site must be equipped with a separate probe.

The automatic tank gaging system probe is connected to a monitor that displays ongoing product level information and the results of the monthly test. Printers can be connected to the monitor to record this information.

Automatic tank gaging systems are often equipped with alarms for high and low product level, high water level and theft. Automatic tank gaging systems can be linked with computers at other locations, from which the system can be programmed or read.

For automatic tank gaging systems that are not continuous, no product should be delivered to the tank or withdrawn from it for at least 6 h before the monthly test or during the test (which generally takes 1 to 6 h).

It is recommended that an automatic tank gaging system be programmed to perform a test more often than once per month.

FIGURE 2. Leak testing using secondary containment with interstitial monitoring.[2]

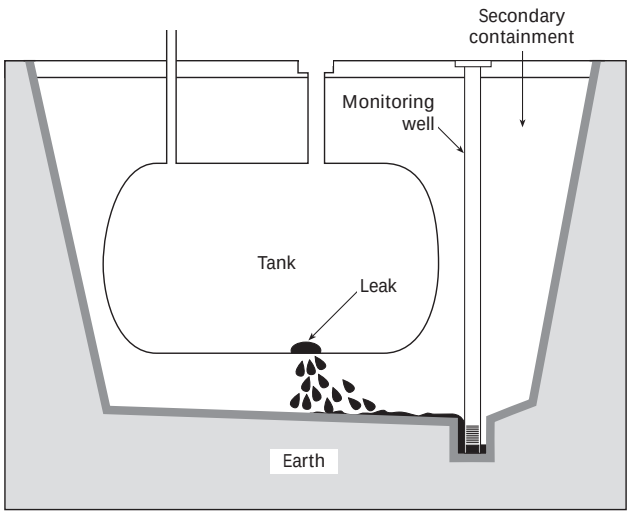
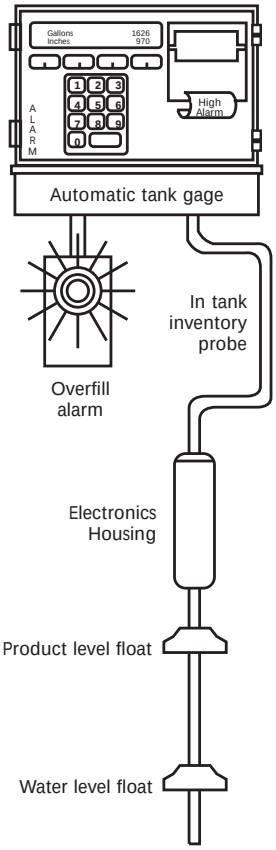


FIGURE 3. Automatic system for gaging product level in tank.[3]



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Vapor Monitoring

Principle of Operation

Vapor monitoring senses or measures fumes from leaked product in the soil around the tank to determine if the tank is leaking (see Fig. 4). Fully automated vapor monitoring systems have permanently installed equipment to continuously or periodically gather and analyze vapor samples and respond to a release with a visual or audible alarm.

Manually operated vapor monitoring systems range from equipment that immediately analyzes a gathered vapor sample to devices that gather a sample that must be sent to a laboratory for analysis. Monitoring results from manual systems are generally less accurate than those from automated systems. Manual systems must be used at least once a month to monitor a site.

All vapor monitoring devices should be periodically calibrated according to the manufacturer's instructions to ensure that they are properly responding.

Before installation, a site assessment is necessary to determine the soil type, groundwater depth and flow direction and the general geology of the site. This can only be done by a trained professional.

The number of wells and their placement is very important. Only an experienced contractor can properly design and construct an effective monitoring well system. Vapor monitoring requires the installation of monitoring wells within the tank backfill. A minimum of two wells is recommended for a single tank excavation. Three or more wells are recommended for an excavation with two or more tanks. Some state and local agencies have developed regulations for monitoring well placement.

Regulatory Requirements

The underground storage tank backfill must be sand, gravel or another material that will let the vapors easily move to the monitor.

The backfill should be clean enough that previous contamination does not interfere with the detection of a current leak.

The substance stored in the underground storage tank must vaporize easily so that the vapor monitor can detect a release.

High groundwater, excessive rain or other sources of moisture must not interfere with the operation of vapor monitoring for more than 30 consecutive days.

Monitoring wells must be secured and clearly marked.

Implementation

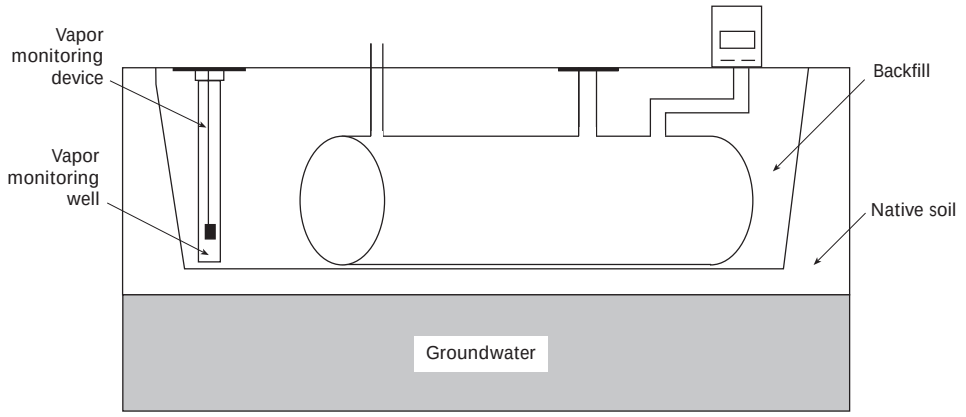
Before installing a vapor monitoring system, a site assessment must be done to determine whether vapor monitoring is appropriate at the site. A site assessment usually includes at least a determination of the groundwater level, background contamination, stored product type and soil type. This assessment can be done only by a trained professional.

Groundwater Monitoring

Principle of Operation

Groundwater monitoring involves permanent monitoring wells placed close

FIGURE 4. Underground storage tank leak testing system with vapor monitoring wells.[2]



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to the underground storage tank (see Fig. 5). The wells are checked at least monthly for the presence of product that has leaked from the underground storage tank and is floating on the groundwater surface.

The two main components of groundwater monitoring system are the monitoring device and the monitoring well, typically a well of 50 to 100 mm (2 to 4 in.) in diameter.

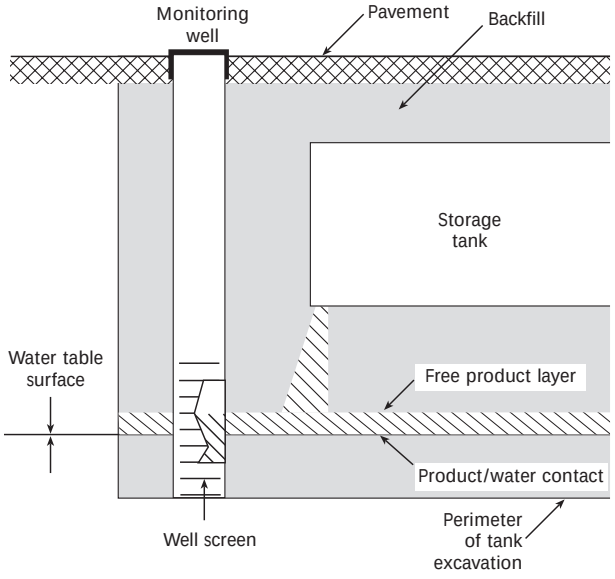
Detection devices may be permanently installed in the well for automatic, continuous measurements for leaked product.

Detection devices are also available in manual form. Manual devices range from a bailer (used to collect a liquid sample for visual inspection) to a device that can be inserted into the well to electronically indicate the presence of leaked product. Manual devices must be operated at least once a month.

Before installation, a site assessment is necessary to determine the soil type, groundwater depth and flow direction and the general geology of the site. This assessment can only be done by a trained professional.

The number of wells and their placement is very important. Only an experienced contractor can properly design and construct an effective monitoring well system. A minimum of two wells is recommended for a single tank excavation. Three or more wells are recommended for an excavation with two or more tanks. Some state and local agencies have developed regulations for monitoring well placement.

FIGURE 5. Monitoring wells installed in the excavation zone will quickly detect a release when the groundwater table is within the tank excavation.[2]



Regulatory Requirements

Groundwater monitoring can only be used if the stored substance does not easily mix with water and floats on top of water.

If groundwater monitoring is to be the sole technique of leak testing, the groundwater must not be more than 6 m (20 ft) below the surface and the soil between the well and the underground storage tank must be sand, gravel or other coarse materials.

Monitoring wells must be properly designed and sealed to keep them from becoming contaminated from outside sources. The wells must also be clearly marked and secured.

Wells should be placed in the underground storage tank backfill so that they can detect a leak as quickly as possible.

Product detection devices must be able to detect 3 mm (0.12 in.) or less of leaked product on top of the groundwater.

Implementation

In general, groundwater monitoring works best at underground storage tank sites where (1) monitoring wells are installed in the tank backfill and (2) there are no previous releases of product that would falsely indicate a current release.

A professionally conducted site assessment is critical for determining these site specific conditions.

Statistical Inventory Reconciliation

Principle of Operation

Statistical inventory analysis analyzes inventory, delivery and dispensing data collected over a period of time to determine whether or not a tank system is leaking.

Each operating day, the product level is measured using a gage stick or other tank level monitor. Complete records can be kept of all withdrawals from the underground storage tank and all deliveries to the underground storage tank. After data have been collected for the period of time required by the statistical inventory reconciliation vendor, the data are provided to the statistical inventory reconciliation vendor.

The statistical inventory reconciliation vendor uses computer software to conduct a statistical analysis of the data to determine whether or not the

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underground storage tank may be leaking. The statistical inventory reconciliation vendor provides a test report of the analysis.

Regulatory Requirements

To be allowable as monthly motoring, a statistical inventory reconciliation technique must be able to detect a leak at least as small as 1×10^{-7} $m^3 \cdot s^{-1}$ or 0.4 $L \cdot h^{-1}$ (0.2 $gal \cdot h^{-1}$) and meet the federal regulatory requirements regarding probabilities of detection and of false alarm. Data must be submitted at least monthly.

To be allowable as an equivalent to tank tightness testing, a statistical inventory reconciliation technique must be able to detect a leak at least as small as 0.4 $L \cdot h^{-1}$ (0.1 $gal \cdot h^{-1}$) and meet the federal regulatory requirements regarding probabilities of detection and of false alarm.

The individual statistical inventory reconciliation technique must have been evaluated with a test procedure to certify that it can detect leaks at the required level and with the appropriate probabilities of detection and of false alarm.

If the test report is not conclusive, the steps necessary to find out conclusively whether the tank is leaking must be taken. Because statistical inventory reconciliation requires data for multiple days, it will probably be necessary to use another technique.

Records must be kept of both the test reports and of the documentation that the statistical inventory reconciliation technique used is certified as valid for the underground storage tank system.

Implementation

Generally, few product or site restrictions apply to statistical inventory reconciliation.

Statistical inventory reconciliation has been used primarily on tanks no more than 68 m^3 (18 000 gal) in capacity. The applicability of a statistical inventory reconciliation technique for larger tanks should be discussed with the vendor.

Water around a tank may hide a hole in the tank or distort the data to be analyzed by temporarily preventing a leak. To detect leakage in this situation, a check for water must be made at least once a month.

Data, including product level measurements, dispensing data and delivery data, should all be carefully collected according to the statistical inventory reconciliation vendor's specifications. Poor data collection produces inconclusive results and noncompliance.

The statistical inventory reconciliation vendor will generally provide forms for recording data, a calibrated chart converting liquid level to volume and detailed instructions on conducting measurements.

Statistical inventory reconciliation should not be confused with other release detection techniques that also rely on periodic reconciliation of inventory, withdrawal and delivery data. Unlike manual tank gaging or inventory control, statistical inventory reconciliation uses a sophisticated statistical analysis of data to detect releases. This analysis can only be done by competent, trained practitioners.

Tank Tightness Testing

Principle of Operation

Tightness tests include a wide variety of techniques. Other terms used for these techniques include *precision, volumetric and nonvolumetric* testing.

Many tightness test techniques are volumetric techniques in which the change in product level in a tank over several hours is measured very precisely (in milliliter or thousandths of an inch).

Other techniques use acoustics or tracer chemicals to determine the presence of a hole in the tank. With such techniques, the following factors may not all apply.

For most techniques, changes in product temperature also must be measured very precisely (thousandths of a degree) at the same time as level measurements, because temperature changes cause volume changes that interfere with finding a leak.

For most techniques, a net decrease in product volume (subtracting out volume changes caused by temperature) over the time of the test indicates a leak.

The testing equipment is temporarily installed in the tank, usually through the pump line or fill pipe (see Fig. 6). The tank must be taken out of service for the test, generally for several hours, depending on the technique.

Many test techniques require that the product in the tank be a certain level before testing, which often requires adding product from another tank on site or purchasing additional product.

Some tightness test techniques require all of the measurements and calculations to be made by hand by the tester. Other tightness test techniques are highly automated. After the tester sets up the equipment, a computer controls the measurements and analysis.

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A few techniques measure properties of the product that are independent of temperature, such as the mass of the product, and so do not need to measure product temperature.

Some automatic tank gaging systems can meet the regulatory requirements for tank tightness testing and be considered as equivalent techniques.

Regulatory Requirements

The tightness test technique must be able to detect a leak at least as small as 1×10^{-7} $m^3 \cdot s^{-1}$ or 0.4 $L \cdot h^{-1}$ (0.1 $gal \cdot h^{-1}$) with certain probabilities of detection and of false alarm.

Tightness tests must be performed periodically. New tanks must be tightness tested every five years for ten years following installation. Many older tanks have been upgraded to have spill, overfill and corrosion protection as all new tanks do in the United States. Upgraded tanks must be tightness tested every five years for ten years following upgrade.

After the applicable time period noted above, a monitoring technique must be performed at least once per month.

Other Considerations

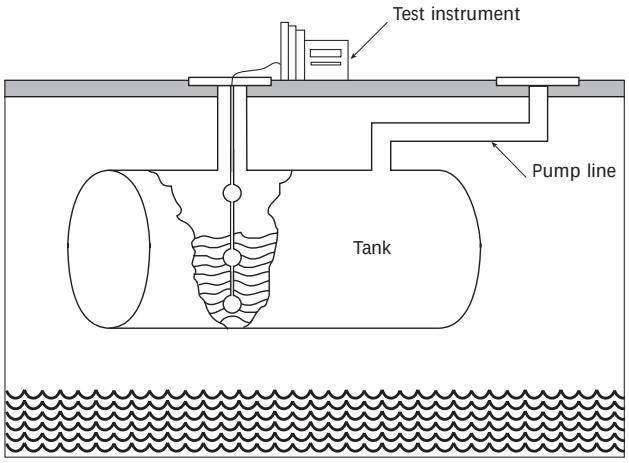
For larger tanks or products other than gasoline or diesel, a technique's applicability should be discussed with the manufacturer's representative.

Manifolded tanks generally should be disconnected and tested separately.

Depending on the technique, up to four tanks can be tested at one time. Generally, an automated system is necessary to test three or four tanks at a time.

Procedure and personnel, not equipment, are usually the most important factors in a successful tightness test. Therefore, well trained and experienced testers are very important. Some states and local authorities have tester certification programs.

FIGURE 6. In most tank tightness test techniques, sensing apparatus is temporarily installed through the fill pipe to monitor product level and temperature in the tank.[2]



Inventory Control

Principle of Operation

Inventory control requires daily measurements of tank contents and math calculations that permit comparison of the *stick inventory* (what has been measured) to the *book inventory* (what record keeping indicates should be present). This process is called *inventory reconciliation*. If the difference between *stick* and *book* inventory is too large, the tank may be leaking.

Underground storage tank inventories are determined daily by using a gage stick and the data are recorded on a form. The level on the gage stick is converted to a volume of product in the tank by using a calibration chart, which is often furnished by the underground storage tank manufacturer.

The amounts of product delivered to and withdrawn from the underground storage tank each day are also recorded. At least once each month, the gage stick data and the sales and delivery data are reconciled and the month's overage or shortage is determined. If the overage or shortage is greater than or equal to 1.0 percent of the tank's flow-through volume plus 490 L (130 gal) of product, the underground storage tank may be leaking.

Regulatory Requirements

Inventory control must be used in conjunction with periodic tank tightness tests.

The gage stick should be long enough to reach the bottom of the tank and marked so that the product level can be determined to the nearest 3 mm (0.125 in.).

A monthly measurement must be taken to identify any water at the bottom of the tank.

Deliveries must be made through a drop tube that extends to within 0.3 m (1 ft) of the tank bottom.

Product dispensers must be calibrated to the local weights and measures standards.

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Implementation

If a given tank is not level, inventory control may need to be modified. The tank owner or operator will need to get a corrected tank chart.

Inventory control is a practical, commonly used management tool that does not require closing down the tank operation for long periods.

The accuracy of tank gaging can be greatly increased by spreading product finding paste on the gage stick before taking measurements (or by using in-tank product level monitoring devices).

Manual Tank Gaging

Principle of Operation

Four measurements of the tank's contents must be taken weekly, two at the beginning and two at the end of at least a 36 h period during which nothing is added to or removed from the tank. See the table on the next page.

The average of the two consecutive ending measurements are subtracted from the average of the two beginning measurements to indicate the change in product volume.

Every week, the calculated change in tank volume is compared to the standards in Table 1. If the calculated change exceeds the weekly standard, the UST may be leaking. Also, monthly averages of the four weekly test results must be compared to the monthly standard in the same way.

Regulatory Requirements

Liquid level measurements must be taken with a gage stick marked to measure the liquid to the nearest 3 mm (0.12 in.).

Manual tank gaging may be used as the sole technique of leak testing for tanks with a capacity of 4 m^3 (1000 gal) or less for the life of the tank. Tanks between 2.1 and 4 m^3 (551 and 1000 gal) have two testing standards based on their diameter (see table). These tanks may use a combination of manual tank gaging and periodic tank tightness for the life of the tank (see Table 1).

For tanks with a capacity of 3.8 to 7.6 m^3 (1001 to 2000 gal), manual tank gaging must be combined with periodic tightness testing. New tanks must be tightness tested every five years for ten years following installation. Upgraded existing tanks must be tightness tested every five years for ten years following upgrade. (*Upgraded* tanks have spill, overfill and corrosion protection.) Existing tanks that have not been upgraded must be tightness tested every year until 1998.

Unless the tank is 3.8 m^3 (1000 gal) or less, this combined technique will meet the federal requirements only temporarily (as explained above). Another monitoring technique must eventually be implemented that can be performed at least once a month. See the other chapters of this booklet for allowable monthly monitoring options.

Tanks greater than 7.6 m^3 (2000 gal) in capacity may not use this technique of leak testing to meet these regulatory requirements.

Implementation

Manual tank gaging is inexpensive and can be an effective leak testing technique when used according to recommended procedures with tanks of the appropriate size.

Correct gaging, recording and interpretation are the most important factors for successful tank gaging. The accuracy of tank gaging can be greatly increased by spreading product finding paste on the gage stick before taking measurements.

Leak Testing for Underground Piping

When installed and operated according to the manufacturer's specifications, the leak testing techniques discussed here meet the federal regulatory requirements for the life of new and existing underground piping systems Some underground storage

TABLE 1. Test standards for manual gaging of product stored in tanks.

Tank Capacity		Minimum Duration	Once per Week		Four Times per Month		
m^3	(gal)	(h)	L	(gal)	L	(gal)	Notes
≤ 2	(≤ 550)	36	38	(10)	19	(5)	
2 to 4	(551 to 1000)	44	34	(9)	15	(4)	when tank diameter is 1.6 m (64 in.)
≤ 2 to 4	(551 to 1000)	58	45	(12)	23	(6)	when tank diameter is 1.2 m (48 in.)
2 to 4	(551 to 1000)	36	49	(13)	27	(7)	also requires periodic tank tightness testing
4 to 8	(1001 to 2000)	36	99	(26)	49	(13)	also requires periodic tank tightness testing

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tanks have suction or pressurized piping, which are discussed below.

Regulatory Requirements for Suction Piping

Typically, no leak testing is required if the suction piping has (1) enough slope so that the product in the pipe can drain back into the tank when suction is released and (2) has only one check valve, which is as close as possible beneath the pump in the dispensing unit. If a line is to be considered exempt based on these design elements, there must be some way to check that the line was actually installed according to these plans.

If a suction line does not meet all of the design criteria noted above, one of the following leak testing techniques must be used: (1) line tightness test at least every three years, (2) monthly interstitial monitoring, (3) monthly vapor monitoring, (4) monthly groundwater monitoring or (5) monthly statistical inventory reconciliation.

The line tightness test must be able to detect leakage at least as small as 1×10^{-7} $m^3 \cdot s^{-1}$ or 0.4 $L \cdot h^{-1}$ (0.1 $gal \cdot h^{-1}$) with certain probabilities of detection and of false alarm.

Interstitial monitoring, vapor monitoring, groundwater monitoring and statistical inventory reconciliation (discussed above) have the same regulatory requirements for piping as they do for tanks.

Regulatory Requirements for Pressurized Piping

Each pressurized piping run must be monitored by one of the following:
(1) automatic line leak testing,
(2) automatic flow restriction,
(3) automatic flow shutoff or
(4) continuous alarm system.

Each pressurized piping run must also have one of the following leak testing techniques: (1) monthly interstitial monitoring, (2) monthly vapor monitoring, (3) monthly groundwater monitoring, (4) monthly statistical inventory reconciliation or (5) annual tightness test.

The automatic line leak detector must be designed to detect a leak at least as small as 3×10^{-7} $m^3 \cdot s^{-1}$ or 1.2 $L \cdot h^{-1}$ (0.3 $gal \cdot h^{-1}$) at a line pressure of 70 kPa (10 $lb_f \cdot in.^{-2}$) within 1 h by shutting off the product flow, restricting the product flow or triggering an audible or visual alarm.

The line tightness test must be able to detect a leak at least as small as 1×10^{-7} $m^3 \cdot s^{-1}$ or 0.4 $L \cdot h^{-1}$ (0.1 $gal \cdot h^{-1}$) when the line pressure is 1.5 times its normal operating pressure. The test must be conducted each year. If the test is performed at pressures lower than 1.5 times operating pressure, the leak rate to be detected must be correspondingly lower.

Automatic line leak detectors and line tightness tests must also be able to meet the federal regulatory requirements regarding probabilities of detection and false alarm.

Interstitial monitoring, vapor monitoring, groundwater monitoring and statistical inventory reconciliation have the same regulatory requirements for piping as they do for tanks.

Automatic Line Leak Detectors

Flow restrictors and flow shutoffs can monitor the pressure within the line in a variety of ways: whether the pressure decreases over time, how long it takes for a line to reach operating pressure and combinations of increases and decreases in pressure.

If a suspected leak is detected, a flow restrictor keeps the product flow through the line well below the usual flow rate. If leakage is detected, a flow shutoff completely cuts off product flow in the line or shuts down the pump.

A continuous alarm system constantly monitors line conditions and immediately triggers an audible or visual alarm if a leak is suspected. Automated internal, vapor or interstitial line monitoring systems can also be set up to operate continuously and sound an alarm, flash a signal on the console or even ring a telephone in a manager's office when a leak is suspected.

Both automatic flow restrictors and shutoffs are permanently installed directly into the pipe or the pump housing.

Vapor and interstitial monitoring systems can be combined with automatic shutoff systems so that whenever the monitor detects a suspected release the piping system is shut down. This would qualify as a continuous alarm system. Such a setup would meet the monthly monitoring requirement as well as the line leak detector requirement.

Line Tightness Testing

Tracer techniques do not measure pressure or flow rates of the product. Instead they use a tracer chemical to determine if there is a hole in the line. With tracer techniques, not all of the factors below may apply.

The line is taken out of service and pressurized, usually above the normal operating pressure. A drop in pressure over time, usually an hour or more, suggests a possible leak.

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Suction lines are not pressurized very much during a tightness test — about 50 to 100 kPa (7 to 15 $lb_f \cdot in.^{-2}$).

Most line tightness tests are performed by a testing company. Storage facility operators may observe the test.

Some tank tightness test techniques can be performed, including a tightness test of the connected piping.

For most line tightness tests, no permanent equipment is installed.

In the event of trapped vapor pockets, it may not be possible to conduct a valid line tightness test. There is no way to tell definitely before the test begins if this will be a problem, but long complicated piping runs with many risers and dead ends are more likely to have vapor pockets.

Some permanently installed electronic systems of some automatic tank gaging systems can meet the requirements of a line tightness test.

Secondary Containment with Interstitial Monitoring

A barrier is placed between the piping and the environment. Double walled piping or a leakproof liner in the piping trench can be used.

A monitor is placed between the piping and the barrier to sense leakage if it occurs. Monitors range from a simple stick that can be put in a sump to see if a liquid is present, to continuous automated systems that monitor for the presence of liquid product or vapors.

Proper installation of secondary containment is the most important and the most difficult aspect of this leak testing technique. Trained and experienced installers are necessary.

Secondary containment for piping is similar to that for tanks.

Vapor or Groundwater Monitoring

Vapor monitoring detects product that leaks into the soil and evaporates. *Groundwater monitoring* checks for leaked product floating on the groundwater near the piping. A site assessment must be used to determine monitoring well placement and spacing.

Underground storage tank systems using vapor or groundwater monitoring for the tanks are well suited to use the same monitoring technique for the piping. Use of these techniques with piping is similar to that for tanks.

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PART 2. Leak Testing of Aboveground Storage Tanks[4]

The primary leak tests for tanks under construction or following alteration or repair are usually described and required by design standards and specifications. A few commonly applied standards and specifications include the following:
API 650, *Welded Steel Tanks for Oil Storage;*[5]
API 620, *Design and Construction of Large, Welded, Low-Pressure Storage Tanks;*[6]
API Standard 653, *Tank Inspection, Repair, Alteration, and Reconstruction;*[7] and
ASME B96.1, *Welded Aluminum-Alloy Storage Tanks.*[8]

The following is a discussion of aboveground storage tank components and of leak tests typically required.

Tank shell plates and welds below the maximum liquid level height are generally water fill tested. This test is a hydrostatic test for atmospheric pressure tanks or a hydropneumatic test for low pressure tanks. The advantages of a water fill test for leakage include its technical simplicity and the ability to leak test with the full design load applied to the tank shell and foundation. Disadvantages of a water fill test include the difficulties of obtaining large volumes of test water in locations where water supplies are limited and concerns with test water corrosion of the tank. One of the most common difficulties associated with a water fill test is the question of water disposal following testing. A legal and environmentally acceptable means of disposal must be available. This is more difficult in testing tanks that have previously been in service than in testing new construction.

Under special circumstances a tank shell may be leak tested by filling the tank with product without a water fill test. A tank failure on initial testing with product is a serious matter with consequences that require careful evaluation. In the unusual circumstance where a tank will be filled with product without a water test, additional nondestructive testing may be justified before filling the tank. Among the additional tests, a vacuum box bubble test of shell and corner welds with a film of leak testing solution may be considered.

Tank shell fittings with reinforcing pad plates are generally bubble tested with a film of leak testing solution. This test is accomplished by applying air pressure to the space under the pad plate and applying a film of leak test solution to the welds and inner bore of the fitting. A threaded hole through the pad plate is usually provided for this purpose. Tank shell fittings are then water fill tested during the hydrostatic or hydropneumatic test of the tank shell.

Atmospheric pressure tank shell welds above the maximum liquid level are not generally leak tested unless there is a requirement that the welds be gas tight.

For low pressure tanks and gas tight tanks, the welds above the maximum liquid level are generally bubble tested with a leak testing solution. This includes shell welds, shell-to-roof welds, roof plate welds and fittings through the roof and shell. This bubble test can be performed with a vacuum box. Alternatively, the bubble test can be performed by applying a solution film to the welds when the upper shell and roof are pressurized during the hydropneumatic test.

Specifics of technique for bubble testing, hydrostatic testing and hydropneumatic testing can be found in the standards and specifications listed above and elsewhere in this book.

Leak testing of tank bottoms is currently the area of greatest ongoing concern and technology development in aboveground storage tank leak testing. This is reasonable as most liquid product leaks from aboveground storage tanks are through tank bottoms. This is true for both new structures and for tanks in service.

Leak testing of tank bottoms may be broadly divided into two categories: leak location test techniques and quantitative (volumetric) leak test techniques.

Leak Location Test Techniques

Nine techniques for leak location are discussed below:

1. vacuum box bubble testing using soap solution, commercial leak detector solution, linseed oil or other suitable solution;
2. vacuum box liquid penetrant testing;
3. vacuum box penetrant developer testing;
4. ammonia tracer gas with ammonia sensitive paint;
5. ammonia tracer gas with ammonia sensitive tape;

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6. detector probe tracer testing using refrigerant-12 or refrigerant-22 halogen rich tracer with a halogen diode leak detector;
7. detector probe tracer testing using sulfur hexafluoride halogen rich tracer with an electron capture halogen leak detector;
8. detector probe tracer testing using helium with a helium mass spectrometer leak detector; and
9. acoustic emission leak testing.

Disadvantages of Leak Location Test Techniques

With the exception of the tracer gas tests, all of the listed leak location tests have been used for many years to one degree or another on these structures. However, no leak location test enables the test technician to determine the total leakage rate for a test system. Consequently, when a leak location test is completed, there cannot be total confidence that all unacceptable leaks were detected.

Comparison of Leak Location Test Techniques

Table 2 compares tank bottom leak location test techniques.

Quantitative Volumetric Test Techniques

Recently, owners have been specifying double bottom designs and quantitative leak test techniques.

These quantitative leak test techniques are intended to ensure that all unacceptable leaks have been detected and repaired. These quantitative leak test techniques include (1) pressure rise measurement, (2) pressure loss measurement and (3) constant pressure mass flow measurement.

Applicable Design Standards

For many years, API 650, *Welded Steel Tanks for Oil Storage,*[5] has required either an air pressure test or a 13.8 kPa (2 $lb_f \cdot in.^{-2}$ gage) pressure differential vacuum box test.

Soap film, linseed oil or other suitable leak detector solution is specified for leak testing all bottom lap or butt welds and the shell to bottom corner weld of this design of tank. Similarly, API Standard 620, *Design and Construction of Large, Welded, Low-Pressure Storage Tanks,*[6] requires a 20.7 kPa (3 $lb_f \cdot in.^{-2}$ gage) vacuum box solution film test of all joints between bottom plates of tanks of this design.

In May 1992, Appendix I on *Underground Leak Detection and Subgrade Protection* was issued as an addendum to API 650.[5] It contains cross chapters of typical arrangements for leak testing at the tank perimeter on double bottom or flexible membrane liner designs. It refers to API Recommended Practice 651, *Cathodic Protection of Aboveground Petroleum Storage Tanks,*[9] for guidelines on the use of cathodic protection techniques. It also refers to API Recommended Practice 652, *Lining of Aboveground Petroleum Storage Tank Bottoms,*[10] on the use of linings to prevent internal bottom corrosion.

API Standard 653, *Tank Inspection, Repair, Alteration, and Reconstruction,*[7] which covers tanks built to API Standard 650,[5] requires either a vacuum box solution film bubble test or a tracer gas test of all bottom weld joints. It requires a vacuum box solution film bubble test or a light diesel oil test of the shell-to-bottom corner weld joint. No pressure differential is listed in this standard for the vacuum box test. Item C.2.3.i of the "Tank Out-of-Service Inspection Checklist" simply says to "vacuum test the bottom lap welds."[7]

API Recommended Practice 575, *Inspection of Atmospheric and Low Pressure Storage Tanks,*[11] is the guideline for the aboveground storage tank Inspector Certification Program and is based on the API 653 Standard.[7]

TABLE 2. Comparison of leak location test methods for aboveground storage tank bottoms.

Test Method	Test Sensitivity		Relative Cost	Training (h)	Equipment
	$Pa \cdot m^3 \cdot s^{-1}$	(std $cm^3 \cdot s^{-1}$)			
Bubble test	10^{-3} to 10^{-4}	(10^{-2} to 10^{-3})	1.0	2	vacuum box, solution
Vaccuum box penetrant	10^{-4} to 10^{-5}	(10^{-3} to 10^{-4})	1.5	2	vacuum box, penetrant, developer
Vaccuum box developer	10^{-4}	(10^{-3})	0.5	unspecified	vacuum box, developer
Ammonia sensitive paint or tape	10^{-4} to 10^{-5}	(10^{-3} to 10^{-4})	2.0	unspecified	vacuum box, ammonia, ammonia sensitive tape or paint
Halogen diode detector probe	10^{-3} to 10^{-4}	(10^{-2} to 10^{-3})	3.0	8 to 12	tracer gas supply, detector instrument, related equipment
Electron capture	10^{-5} to 10^{-6}	(10^{-4} to 10^{-5})	4 to 5	8 to 12	tracer gas supply, detector instrument, related equipment
Helium detector probe	10^{-3} to 10^{-6}	(10^{-2} to 10^{-5})	4 to 6	28 to 40	helium mass spectrometer leak detector, detector probe, helium supply

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Leak Testing Training

The API standards applicable to aboveground storage tanks do not have any training requirements for personnel who perform leak testing of these structures.

Leak location test techniques are particularly dependent on both the test operator's ability and dedication to doing a thorough leak test at the best level of that ability. Operators being human, this does not always happen. To increase the level of reliability of these economical leak location tests requires an increase in training, qualification and certification of leak testing personnel.

The original edition of ASNT's *Recommended Practice No. SNT-TC-1A: Personnel Qualification and Certification in Nondestructive Testing* and every later revised edition of that document has included recommendations for training, qualification and certification of leak testing personnel.

Unfortunately, none of the standards for aboveground storage tanks require training of leak testing personnel. As a result, the frequent use of personnel with little or no training and experience contributes to the practice of putting aboveground storage tanks into service with intolerable or objectionable leakage.

Acoustic Emission Leak Testing

Acoustic emission testing has attracted interest for storage tank applications. Disadvantages of acoustic emission leak testing are that it does not quantify leakage and it is sensitive to interference from ambient noise and vibration. Advantages are that it provides precise information for leak location, it may provide continuous monitoring and it does not require additional structures such as displacement chambers and double bottoms. The acoustic techniques of leak testing are discussed in more detail in technical literature[12-15] and elsewhere in this book.

Double Bottom Designs

Current practice to attempt to achieve quantitative bottom leak testing results when constructing new or reconstructing existing aboveground storage tanks is to specify a design that requires the installation of two bottoms. Figures 7 to 9 are cross section examples of specified double bottom designs that may have been used in the construction of new aboveground storage tanks.

FIGURE 7. Planar and cross section views of double bottom design for construction of aboveground storage tanks with spacer plates.

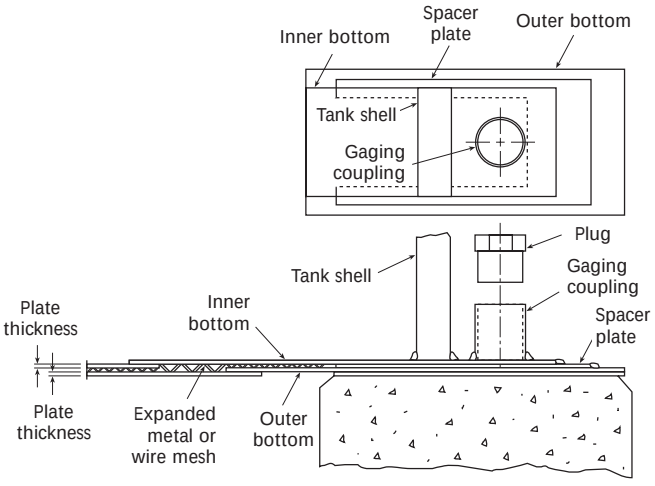


FIGURE 8. Cross section of double bottom design for construction of aboveground storage tanks.

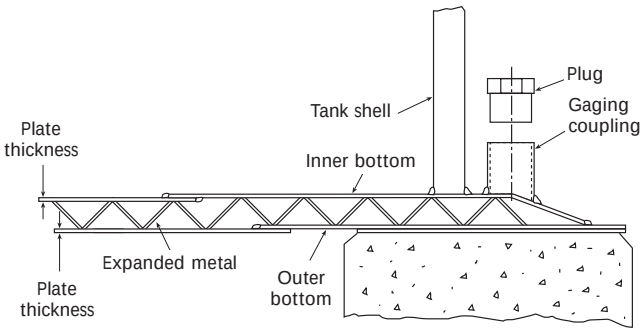
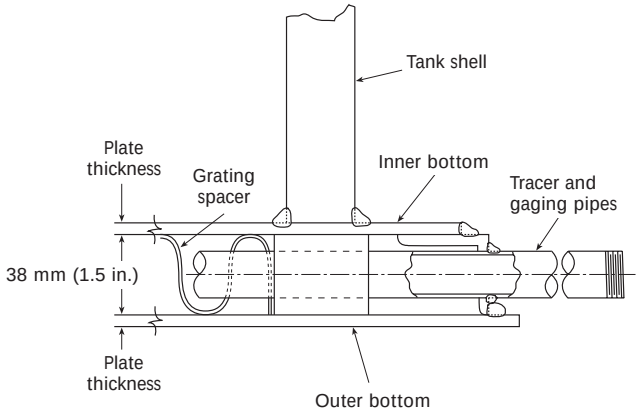


FIGURE 9. Cross section of double bottom design for construction of aboveground storage tanks in which tank shell rests on outer bottom.



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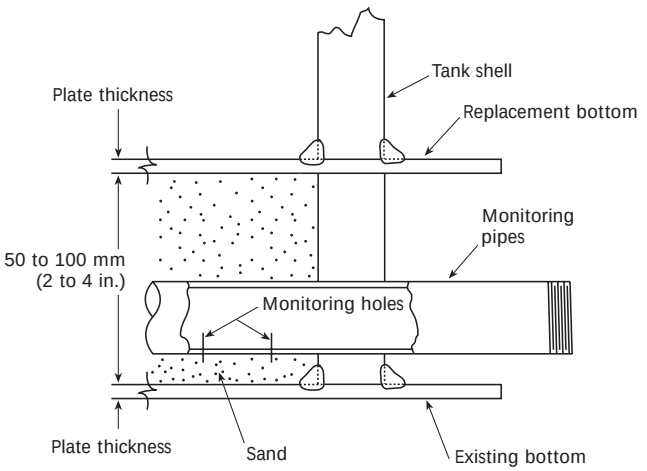
The function of the inner bottom is to contain the stored product with no unacceptable or objectionable leakage. A function of the outer bottom would be to provide a closed test system that could either be (1) pressurized with a tracer gas to a very low pressure for a semiquantitative detector probe test, (2) pressurized to a very low pressure for a quantitative pressure loss measurement test, (3) partially evacuated for a quantitative pressure rise measurement test or (4) pressurized to a very low specific pressure and held at that pressure for the purpose of a mass inflow quantitative measurement leak test.

Another function of the outer bottom could be to use it as a catch basin to monitor for inservice leakage from the inner bottom. This function may be in addition to its use for a quantitative leak test or it may be its primary function.

Figure 10 is a cross section example of a second bottom installed on top of an existing tank bottom with radial monitoring pipes leading to the tank perimeter. The inner bottom is supported on sand, which surrounds the pipes containing holes on the underside. The outer bottom in such situations is usually used only for inservice leakage monitoring. Because of the sand it is a poor design for quantitative testing of the inner bottom.

FIGURE 10. Cross section of second bottom installed on top of existing tank bottom with radial monitoring pipes leading to tank perimeter.



Comparative Test Sensitivities of Leak Location Techniques

Table 2 compares leak location test techniques for aboveground storage tank bottoms.

Vacuum Box Bubble Testing

Vacuum box bubble testing under field conditions can produce a test sensitivity of 10^{-3} to 10^{-4} Pa·m^3·s^{-1} (10^{-2} to 10^{-3} std cm^3·s^{-1}) at a reasonable cost. With extra care 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) range leakage size can be detected under field conditions, but to detect this much less commonly occurring, smaller size of leakage requires the expenditure of additional time and money.

This is not a highly technical test technique and requires a minimal amount of operator training. This technique can be performed progressively during construction of the tank bottom, saving time on the schedule because it does not require a closed test system to be pressurized. For these reasons this is the test technique that has been most commonly used by owners and tank contractors. Because this technique has been the industry standard for many years, it is the test technique against which all others listed in this chapter are compared.

Vacuum Box Liquid Penetrant Testing

Vacuum box liquid penetrant testing of bottom lap or butt welds is performed by applying liquid penetrant to the test surface, removing the excess after the penetration time has elapsed, applying the developer and then applying a differential pressure with the vacuum box. This is a variation of vacuum box bubble testing that is normally only used in situations where very small leakage is known to exist but has escaped detection by other techniques.

Under field conditions the achievable sensitivity of this test technique is in the range of 10^{-4} to 10^{-5} Pa·m^3·s^{-1} (10^{-3} to 10^{-4} std cm^3·s^{-1}). However, compared to vacuum box bubble testing, it costs considerably more and requires more background and experience to determine when the situation warrants this approach.

Vacuum Box Penetrant Developer Testing

Vacuum box penetrant developer testing is a special leak test technique, normally only used for lap and butt welds in single

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bottoms. It is applied when leakage has been detected during the tank hydrostatic test and it is suspected that a normal vacuum box test would be ineffective due to the possibility of water lying against the underside of the tank bottom in the area of the leak. For this test technique the developer is applied to the suspected area or areas and allowed to dry. It is then visually inspected after a number of hours have elapsed (maybe overnight) for signs of moisture bleed out into the developer indicating the area of the leak.

This test technique is normally used to detect gross leakage but has the capability under production conditions of enabling an operator to detect leakage as small as the 10^{-4} Pa·m^3·s^{-1} (10^{-3} std cm^3·s^{-1}) range. This too requires more experience in order to determine the best course of action for the various situations that develop.

Ammonia Sensitive Paint or Tape

Ammonia sensitive paint testing or ammonia sensitive tape testing with an ammonia gas mixture under the bottom can result in a test sensitivity as small as the 10^{-4} to 10^{-5} Pa·m^3·s^{-1} (10^{-3} to 10^{-4} std cm^3·s^{-1}) range. However, it is rarely used because of the hazards to human life that ammonia presents to those doing the testing. It also costs considerably more to perform than the vacuum box bubble test technique. Furthermore, leakage in the 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) range is not very common and for that reason does not justify the additional hazards and cost to detect. This technique is more technical in nature than vacuum box bubble testing and requires more extensive safety equipment, training and testing experience.

Halogen Diode Detector Probe Testing

Halogen diode detector probe testing with refrigerant-12 and refrigerant-22 as the tracer gas was used during the 1960s on a trial basis to test the bottom lap welds in several liquid natural gas tanks per API 620 Appendix Q.[6] For these experimental leak tests, nylon reinforced rubber blankets were installed under the tank bottoms during construction. These blankets were epoxied to the shell in an attempt to achieve a more uniform higher pressure tracer gas mixture under the bottoms while having a minimum of tracer gas background around the tank perimeters during a test. This leak location approach produced no marked increase in the pressure attainable under the bottom or in the test sensitivity over that attainable by vacuum box testing. The reasons for the lack of sensitivity increase are given in the following paragraphs.

If all test parameters, such as differential pressure, were equal, any of the tracer gas leak location techniques would be able to detect smaller leakage than the other less technical test techniques such as vacuum box testing, ammonia sensitive tape or paint etc. However, test parameters are not the same for each technique. All tracer gas leak test techniques depend on the instrument sensitivity, the differential test pressure, the percentage by volume mixture of tracer gas, the uniformity of that tracer gas mixture throughout the test system. Techniques using a detector probe depend on the scanning speed and the distance the detector probe is held from the test surface during scanning (sniffing). Techniques using accumulation will depend on the accumulation time and the leak tightness of the accumulation box.

When performing a nonquantitative (semiquantitative at best) detector probe test of the flat bottom of a tank, the amount of pressure that can be applied (either single or double bottom) is limited to slightly higher than the weight of the bottom being pressurized. This limitation is due to ballooning of the bottom when the pressure exceeds the weight of the bottom.

For example, 6.4 mm (0.25 in.) thick steel weighs about 50 kg·m^{-2} (10.2 lb$_f$·ft^{-2}). Thus, for a 6.4 mm (0.25 in.) thick steel bottom, the bottom will start to balloon when the pressure reaches 10.2/144 = 490 Pa (7.08×10^{-2} lb$_f$·in.$^{-2}$) = 51 mm H_2O (1.93 in. H_2O) column pressure.

Allowing an additional 13 mm H_2O (0.5 in. H_2O) pressure for some amount of bottom ballooning, the maximum test pressure of 64 mm H_2O (2.5 in. H_2O) equals 2.5/27.7 ≅ 690 Pa (0.1 lb$_f$·in.$^{-2}$).

The reduction in differential pressure from 101 kPa (14.7 lb$_f$·in.$^{-2}$ or 1 atm) attainable with a vacuum box to only 64 mm H_2O (2.5 in. H_2O) pressure attainable for tracer gas testing reduces the attainable test sensitivity of viscous or transitional flow by an approximate factor of 220.

Dilution of leakage tracer gas by surrounding air at a leak further reduces test sensitivity by an additional factor of at least ten. The test sensitivity attainable would be further reduced by at least another factor of ten based on a tracer gas mixture of ten percent by volume. For outer bottoms, this mixture is achieved by flowing the tracer under the bottom for a period of time or injecting it through coupling at various points in the bottom. The shortcoming is that the uniformity of the tracer gas mixture is not known. For inner bottoms, this mixture is obtained uniformly between the bottoms by

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evacuating the space between the bottoms to a pressure of 0.10 (14.7 + 0.1) – 0.1 = 1.48 – 0.1 = 1.38 (9.6 kPa [1.4 $lb_f \cdot in.^{-2}$] in round numbers) below atmosphere before backfilling and pressurizing with the tracer gas to 64 mm H_2O (2.5 in. H_2O) pressure.

If the space is not evacuated to 9.6 kPa (1.4 $lb_f \cdot in.^{-2}$) below atmosphere before pressurizing, then the uniformity of the tracer gas would not be known and the mixture would only be 0.10(100)/14.8 = 0.68 percent by volume. The test sensitivity attainable would then be reduced by a factor of 100/0.68 = 147 instead of a factor of ten. For this discussion, a ten percent mixture is assumed.

Thus, for this leak location test technique performed under the conditions described, the total reduction in test sensitivity from the maximum realistic attainable test sensitivity would be by a factor of about 220 (10) (10) = 22 000 or 2.2×10^4.

The maximum realistic test sensitivity attainable under field conditions for either a halogen diode or electron capture type leak detector probe test performed using a 100 percent tracer gas mixture at a differential pressure of about 100 kPa (15 $lb_f \cdot in.^{-2}$) gage with a scanning speed of 13 $mm \cdot s^{-1}$ (30 $in. \cdot min^{-1}$) and a detector probe-to-surface distance of 3 mm (0.125 in.) is on the order of 5×10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-7} std $cm^3 \cdot s^{-1}$).

Based on these values, the estimated test sensitivity for this test technique when performed on the bottom welds of an aboveground storage tank would be about $(5 \times 10^{-7}) \times (2.2 \times 10^4) = 1 \times 10^{-3}$ $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-2} std $cm^3 \cdot s^{-1}$).

This is about the same test sensitivity as the vacuum box bubble test technique but costs considerably more to perform. It also requires much more technical training and experience, particularly if those performing or witnessing this technique of testing are to understand the actual test sensitivity that is being obtained.

The test sensitivity for this test technique can be increased by increasing the percent of the tracer gas mixture and by attaching the detector probe to a pod or box placed over a section of test area (as shown in Fig. 11) and waiting for tracer gas leakage from potential leaks to accumulate. The test sensitivity increase is greater for smaller boxes and/or longer accumulation times, but both of these factors rapidly increase test costs. Test sensitivities in the range of 10^{-5} to 10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-4} to 10^{-5} std $cm^3 \cdot s^{-1}$) can be achieved but at a considerable cost increase.

Electron Capture Detector Probe Testing

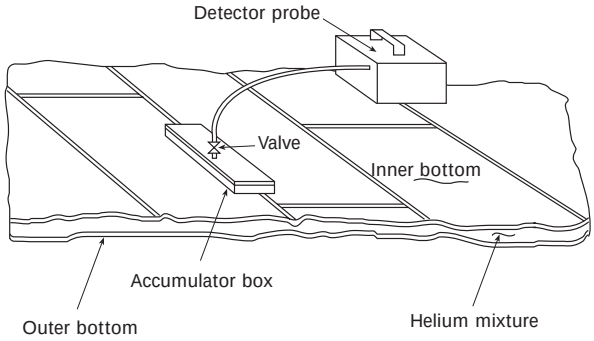
Electron capture detector probe leak testing (ECLT) using sulfur hexafluoride (SF_6) as the tracer gas has come into greater usage with the environmental banning of hydrofluorocarbons and chlorofluorocarbons prevalent in refrigerant-12 and refrigerant-22. This technique has about the same limitations for instrument sensitivity, test pressure under flat bottoms, scanning speed and probe-to-surface distance as the halogen diode detector probe test technique discussed earlier. Thus, the estimated achievable test sensitivity is in the range of 10^{-3} to 10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-2} to 10^{-3} std $cm^3 \cdot s^{-1}$) when detector probe leak testing the bottoms of tanks by this technique. Again, test sensitivities can be increased to the range of 10^{-5} to 10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-4} or 10^{-3} std $cm^3 \cdot s^{-1}$) by the accumulation technique but at a considerable increase in cost.

Mass Spectrometer Detector Probe Testing

As with the halogen diode detector probe or the electron capture detector probe, when using the helium mass spectrometer in the detector probe test mode, the attainable test sensitivity when testing a tank bottom is in the range of 10^{-3} to 10^{-4} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-2} to 10^{-3} std $cm^3 \cdot s^{-1}$). Because a helium mass spectrometer leak detector is a high vacuum instrument, the detector probe pressure test is the test technique for which it is least suited and has the poorest sensitivity.

One advantage of using a helium mass spectrometer leak detector with a pumped detector probe connected to a permeation

FIGURE 11. Test sensitivity is increased by increasing percent of tracer gas mixture and by attaching the detector probe (halogen diode, electron capture or helium mass spectrometer) to pod or box placed over a section of test area and waiting for tracer gas leakage from potential leaks to accumulate.



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membrane accumulation chamber is the very long probe hose (up to 60 m or 200 ft) that may be used with little or no loss in response time and no loss in test sensitivity. This allows the operator to place the helium mass spectrometer at one location near an entry hole and scan the welds of the entire bottom from that one location. The probe hose is so small as to be virtually weightless, whereas a halogen diode or electron capture instrument that weighs 5 to 10 kg (several pounds) must be carried with the probe.

A disadvantage is the added technical training and experience needed to perform helium mass spectrometer detector probe leak testing versus what is needed to perform halogen diode or electron capture detector probe leak testing.

Another disadvantage of the helium mass spectrometer is the greater cost. Depending on the degree of sophistication of the model purchased, the cost of the helium mass spectrometer and associated equipment may cost anywhere from three to six times more than the best halogen diode or electron capture instruments.

If a quantitative leak test of a double bottom is required, then the welds outside the tank shell between the inner and outer bottoms on double bottom designs must be leak location tested before the quantitative test. The fastest and most economical test technique is usually a bubble pressure test.

Comparison of Quantitative Leak Testing Techniques

Pressure rise measurement is one of the quantitative leak test techniques that has been appearing quite frequently in many aboveground storage tank double bottom design specifications. After completion of required preliminary leak testing, the specifications normally require that the space between the double bottoms be partially evacuated to some pressure below atmosphere and held at that pressure for a defined period of time without any increase in the pressure (loss of vacuum). A typical requirement is to evacuate to a negative pressure (vacuum) of 98 kPa (14.2 $lb_f \cdot in.^{-2}$ or 735 torr) below atmosphere and hold for 8 h without any loss (degradation) of the vacuum. Another typical requirement is to evacuate to 68 kPa (9.8 $lb_f \cdot in.^{-2}$ or 508 torr) below atmosphere and hold for 24 h without any loss of the vacuum.

The basic pressure rise relationship is:

$$\textbf{(1)} \quad Q = \frac{\Delta PV}{\Delta t}$$

If Q = leakage rate in mass flow units ($Pa \cdot m^3 \cdot s^{-1}$ or std $cm^3 \cdot s^{-1}$), V = volume of the test system (cubic foot), ΔP = change in pressure (in. Hg) and Δt = change in time (hour), then 3.8 = conversion factor and $Q = \Delta PV/3.8\Delta t$.

As an example, if a tank were 30.5 m (100 ft) in diameter and the grating space between the inner and outer bottoms were 13 mm (0.5 in.) and the grating occupied 20 percent of that space, the volume of that space would be about $V = (50)^2\pi\,(0.4)/12 \cong 7.4\ m^3$ (262 ft^3).

If this volume were evacuated to a negative gage pressure of 98 kPa (736 torr), then held for 8 h and the vacuum gage reading had increased 333 Pa (2.5 torr) in that time, then *one* of the following would be indicated.

1. Real leakage $Q = (0.1)(262)/(3.38)(8) = 0.86$ std $cm^3 \cdot s^{-1}$.
2. Or system volume changes normally because of the tendency of large flat membranes to change shape with very slight temperature changes.
3. Or nothing significant is indicated because the pressure change is within the gage's listed accuracy of 0.33 percent of full scale. This is about the listed accuracy for such gages.

This is the first element of uncertainty when performing a quantitative pressure rise measurement test of an aboveground storage tank with a double bottom. That is, does a small pressure increase during such a test reveal actual leakage or a false indication of leakage? When this occurs, the test can be repeated or continued for a longer time period in order to average down any errors. This may or may not produce a more conclusive result, but in any event it will cost more time and money.

If the conclusion is that this is real leakage, the second element of uncertainty becomes apparent — namely, where is the leakage? Is it in the outer bottom, in the inner bottom or in the perimeter welds between the bottoms outside the tank shell?

To reach a conclusion, the only option is to retest the inner bottom and the perimeter welds between the bottoms outside the tank shell by a leak location technique. If leaks are detected in one or both of these areas, the leaks can be repaired, the hold test can be rerun and the results will most likely be satisfactory. If no leaks are detected in either of these areas, it would have to be assumed that the leakage was from the outer bottom.

Pressure loss measurement is a quantitative leak test technique that can be specified for a double bottom system.

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This technique should not be performed with the tank empty. This is because of the very small positive pressure differential that a large flat membrane bottom can tolerate before it balloons; i.e., 600 to 750 Pa (63 to 76 mm H_2O or 2.5 to 3 in. H_2O) water pressure. Even a slight variation in temperature will change the pressure and make a sizable change in the volume due to the movement of the inner bottom. This will produce test results impossible to interpret.

When this technique is specified, it should include the requirement that a head of water must be in the tank during the test. If a tank contains 3.7 m (12 ft) of water, assuming an adequate foundation under the outer bottom, the space between the bottoms could then be pressurized to 34 kPa (5 $lb_f \cdot in.^{-2}$), without any ballooning of the inner bottom. This would also virtually eliminate temperature as a variable for that space because the water would be a large heat sink that would keep the temperature of that space stable. On completion of the test, the drain or sampling pipes between the bottoms can be checked for signs of moisture. If the hold test fails, an indication or lack of an indication of moisture between bottoms would indicate the source of the leakage.

Mass flow measurement is a quantitative test technique that is not usually specified by owners but that has several decided advantages. First, it can be performed at a pressure either above or below atmospheric pressure; second, it can be performed in a minute or two. Limiting the time duration of the measurement can eliminate temperature as a test variable. If conducting this test technique at a pressure above atmospheric pressure, it is suggested that it be done with water in the tank so that a pressure of about 20 kPa (a few pounds per square inch) can be used rather than only about 1 kPa (3 or 4 in. H_2O). A disadvantage of this test technique is that the mass flow meter must be purchased for the specific pressure level and anticipated potential leakage rate range that would have to be measured.

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PART 3. Determining Leakage Rate in Petrochemical Structures

Sealed Volume Flow Meter Leak Testing of Large Pipelines and Systems

The sealed volume flow meter leak testing technique can be used for leak testing of pipelines and other large volume systems. The pipeline segment to be leak tested is plugged off at its ends. The isolated section is connected to a source of pressurizing gas or air and equipped with a pressure regulator and a *bubbler*. Pressurized gas or air is injected through the bubble to fill the pipeline segment or large system under test. If the line or vessel is leak tight, the bubbling will eventually cease when the pressure within the system is equal to the pressure applied externally. If leakage exists, there will be no cessation of bubbling while the pressurizing source continues to inject gas or air to replace that lost by leakage in the system under test.

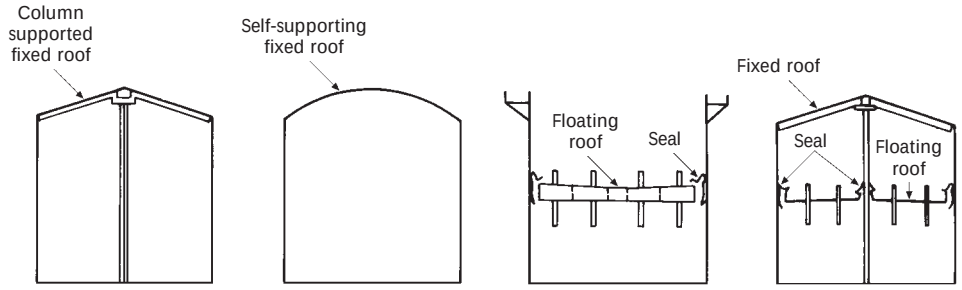
Flow Meter Leakage Testing of Petrochemical Structures

The flow measurement leak test technique finds applications in leak testing of large petrochemical structures. Flat bottomed storage tanks are designed with column supported or self-supporting fixed roofs, various types of floating roofs and combinations of fixed and floating roofs, as sketched in Fig. 12. These structures normally operate with atmospheric pressure or with gage pressures of about 300 Pa (1.25 in. water) in air or gas above the liquid petroleum product contained in the structure. These storage tanks are normally built and tested in accordance with API 650 standards.[5,16-18]

Tanks such as those sketched in Fig. 12 are used to store crude oil, various grades of refined oils, jet engine fuels, kerosene, gasolines and other petroleum products. When constructed of corrosion resistant materials, they are also used to store acidic and caustic products. As single wall low pressure structures with external insulation, or as double wall structures with the same configurations as cryogenic structures, they are used to store liquid propane, liquid butane, anhydrous ammonia and other low temperature liquid products. They are normally built and tested in accordance with API 620, *Design and Construction of Large, Welded, Low·Pressure Storage Tanks*.[6]

To reduce vapor loss from fixed roof tanks containing volatile products such as gasoline, several of these tanks containing the same product are sometimes manifolded to a variable volume structure such as a lifter roof tank, vapor tank or vapor sphere such as those sketched in Fig. 13. When properly sized, these constant gage pressure structures maintain the pressure in the manifolded fixed roof tanks at levels below the settings of the pressure relief vents and above the settings of the vacuum relief vents. Thus, they not only reduce loss of product vapor but also prevent the intake of oxygen laden atmospheric air, which can create explosive atmospheres above flammable products.

FIGURE 12. Petrochemical storage vessels with various roof designs.



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Leakage Requirements for Petrochemical Structures

Functional and code leakage requirements for petrochemical structures such as those sketched in Fig. 12 and 13 are intended to ensure that finished product tanks have no liquid product leakage. For tanks that are to store volatile products that are flammable, toxic, acidic or caustic, no vapor leakage is desirable. But for tanks such as those with floating roofs with moving flexible seals, this is not a realistic functional requirement. For these types of tanks the product vapor leakage must be sufficiently limited so that it does not create a toxic or flammable hazard in the area around the structure and so that it minimizes product evaporation losses. These same requirements apply also to variable volume vapor storage tanks. The leak testing requirements of the API code are normally adequate for these types of structures. Any additional leak testing performed is normally done because of customer specification or manufacturer requirements.

Selection of Leak Testing Technique for Petrochemical Structures

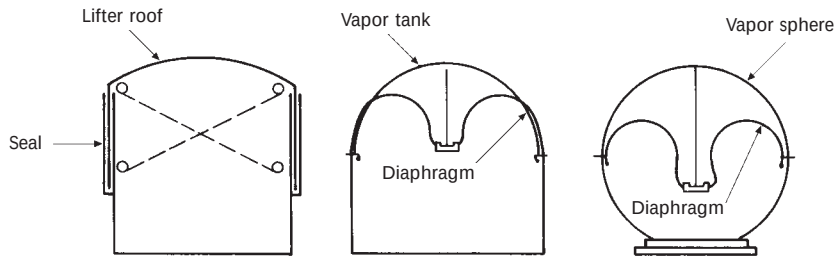
The leak testing performed on petrochemical structures such as those sketched in Fig. 12 and 13 is normally limited to the code required bubble testing or solution film testing technique and the through penetrant leak testing technique. Past experience has shown that the sensitivity of these two techniques, when they are performed properly, is adequate. When the manufacturer of a floating roof storage tank desires new or additional performance data on a seal design or when the specifications for a floating roof tank require that the roof-to-shell seal leakage rate be determined, it is usually accomplished by the flow measurement leakage test technique. When a customer specification requires the determination of the total leakage rate of a variable volume structure with a movable diaphragm, the leak test is performed by a volume change leak testing technique.

Techniques Used in Leak Testing of Typical Petrochemical Structures

Preliminary leak testing of small critical areas of weldments or assemblies in petrochemical structures uses bubble tests performed by direct pressurization or vacuum box techniques. As an alternative to bubble testing, leak testing with liquid tracers is performed either directly (using capillarity of penetrants rather than pressure differentials) or with a pressure differential obtained with a vacuum box.

A pressure drop orifice flow measurement test technique is adequate for the accuracy required to determine the leakage rate of a floating roof-to-shell seal. The volume change technique, auxiliary chamber displacement technique or auxiliary chamber accumulation flow meter technique may be used to determine the total leakage rate of a variable volume petrochemical structure with a movable diaphragm. These techniques are described next.

FIGURE 13. Various designs of variable volume petrochemical storage vessels, including some with diaphragms.



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Pressure Drop Orifice Test for Leakage Rate of Floating Roof Seal

Figure 14 shows schematically the technique for connecting a water manometer to the seal space between the tank shell and the outer rim plate of a floating roof of a petrochemical storage structure. Then the leakage rate of the floating roof seal can be determined by the pressure drop orifice leak test technique, after installing an orifice with a pressure regulator in a line to the seal space. The pressure regulator is then adjusted until the orifice flow maintains the seal space at the test pressure specified.

The flow rate for the system orifice for the pressure shown on the pressure regulator is found by reference to the flow chart relating flow rate to pressure drop for the specific orifice being used. (The downstream pressure at the outlet of the orifice can be ignored because it is insignificant compared to the orifice inlet pressure.) The flow rate that is through the orifice and that just maintains the seal space pressure constant is a direct measure of the total leakage rate of the seal. The preceding steps can be repeated using different sizes of orifice (to vary the air flow rate), if needed to increase the accuracy of the leakage rate test measurements.

Other leak test techniques could be used to determine the total leakage rate of the seal of the floating roof in this example. These include use of a combination flow meter and orifice or use of a low pressure flow meter and regulator to control inflow to match the leakage of the roof seal.

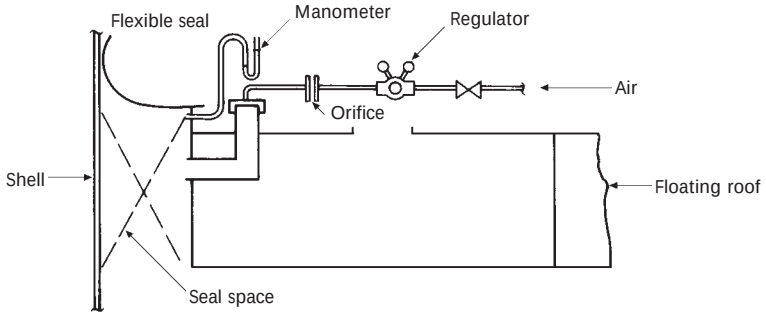
Leakage Rate Test of Variable Volume Vessel with Moving Diaphragm

Figure 15 shows schematically the connections of a water manometer and a pressurizing line to a tank in preparation for a leakage rate test for determining the total leakage rate of a variable volume structure with a movable diaphragm. Vessels of this type operate at a constant gage pressure except at the upper and lower limits of diaphragm displacement. However, due to the very low operating pressure of the diaphragm system, changes in barometric pressure cannot be ignored during the leakage test. Surface thermometers are placed at various locations on the shell and the barometer is placed in a sheltered area near the tank.

With a roof fitting open to the atmosphere, air is injected into the tank below the diaphragm until the diaphragm has been raised to a level near the middle of its travel range. The water manometer measures this air pressure, relative to the atmosphere (249 Pa = 1 in. H_2O; 9.8 Pa = 1 mm H_2O). However, it is necessary to obtain forecasted trends in ambient temperature for the test duration. If the temperature trend is upward, start the leak test with the diaphragm in the lower half of its travel range. If the temperature is expected to fall, start the test with the diaphragm above the middle of its range.

At the start of the leak testing period, record the average, surface temperature T_1, the barometric pressure P_{b1}, and the vessel gage pressure P_{v1}. Also, measure the height of the diaphragm at its center (see Fig. 15). Determine the initial measured volume V_{m1}. From these data, determine the quantity P_1V_1 of gas (mass at standard temperature conditions of 20 °C or 70 °F) from the relation:

FIGURE 14. Arrangement of manometer and pressure drop orifice connections for leakage rate test of the seal of a floating roof petrochemical storage structure.



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$$\textbf{(2)} \quad PV = V_m \left(P_v + P_b\right) \frac{293}{T + 273}$$

where PV is quantity of gas (Pa·m^3); V_m is measured volume estimated from tank capacity chart (cubic meter); P_v is pressure within vessel (pascal); P_b is measured barometric pressure (pascal); and T is average temperature (degree celsius).

At the end of the required leak testing period, record the same parameters as P_{v2}, P_{b2}, T_2 and V_{m2} and again determine the final mass P_2V_2 of gas, corrected to the standard temperature (see Eg. 2). The leakage rate in SI units is now given by:

$$\textbf{(3)} \quad Q = \frac{P_1V_1 - P_2V_2}{t_2 - t_1}$$

where t is time (second).

In applications where English units and fahrenheit temperatures are used and time is measured in hours, the quantity of gas at standard conditions is computed as:

$$\textbf{(4)} \quad PV = V_m \frac{P_v + P_b}{14.7} \frac{530}{T + 460}$$

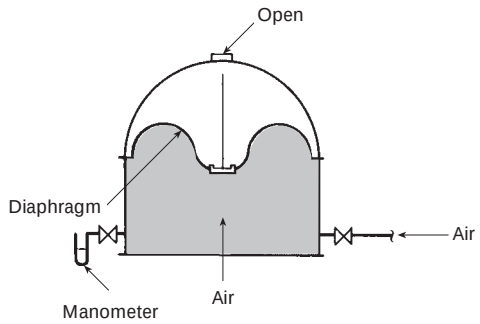
where PV is quantity of gas (standard cubic foot), V_m is measured volume estimated from tank capacity chart (cubic foot), P_v is pressure within vessel (lb_f·in.$^{-2}$ gage), P_b is barometric pressure (lb_f·in.$^{-2}$ absolute), T is average temperature (degree fahrenheit) and t is time (hour).

Again, it is necessary to determine the initial quantity of gas V_1 at the beginning of the leak test and the final quantity P_2V_2 of gas, at the end of the test by use of Eq. 4. The leakage rate in mixed English units is then given by:

$$\textbf{(5)} \quad Q = \frac{P_1V_1 - P_2V_2}{t_2 - t_1}$$

where t is time (hour).

FIGURE 15. Arrangement for air injection and water manometer connections for leakage rate test of variable volume petrochemical vessel with moving diaphragm.



Auxiliary Chamber Leakage Rate Test of Movable Diaphragm in Variable Volume Petrochemical Structure

Figure 16 and 17 show schematically the leak testing equipment arrangement used for measuring leakage rates of a movable diaphragm in a variable volume storage vessel, such as might be used in a petrochemical industrial facility. In preparation for this leak test, first remove the pressure vacuum vent and blank this fitting (shown at top of sphere in Fig. 16). Then an automatic or manual relieving mechanism is connected to the tank, as shown at the lower right of Fig. 16. A water manometer and a pressurizing line are next connected to the tank (as shown at bottom left and bottom right of Fig. 16). Then, open a roof fitting to the atmosphere to let air above the movable diaphragm escape as pressure is applied beneath the diaphragm.

Pressurization and Preliminary Leak Tests of Vessel Test Connections

The tank volume below the movable diaphragm is then pressurized as follows. With the roof fitting still open to the atmosphere, add air to the tank until the movable diaphragm rises to the top. Continue to add air until the tank is at the required test pressure. Add air to the tank or release air from the tank as necessary to maintain this pressure at a fixed level during the leakage rate test.

After pressurization is completed, fill the previously tested displacement chamber with water through the inlet valve at the top of this chamber (see Fig. 17). Then close this inlet valve and install hose or tubing from this valve on the displacement chamber to a roof connection on the large tank. Next, pressurize the space above the tank diaphragm through a valve connection on the topmost access hole cover to provide a slight air pressure. Use bubble testing solution to leak test the connections from the tank roof through the hose or tubing connecting the roof to the displacement chamber, to the blanked vent and to the access hole valve. Any leaks indicated must be repaired and the connections retested before proceeding further. Finally, open the access hole valve to release the pressure above the diaphragm, then close the access valve and install a plug in it.

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FIGURE 16. Arrangement for auxiliary chamber displacement leakage rate test of the movable diaphragm in a variable volume petrochemical storage tank. Overall view of spherical tank and leak test equipment.

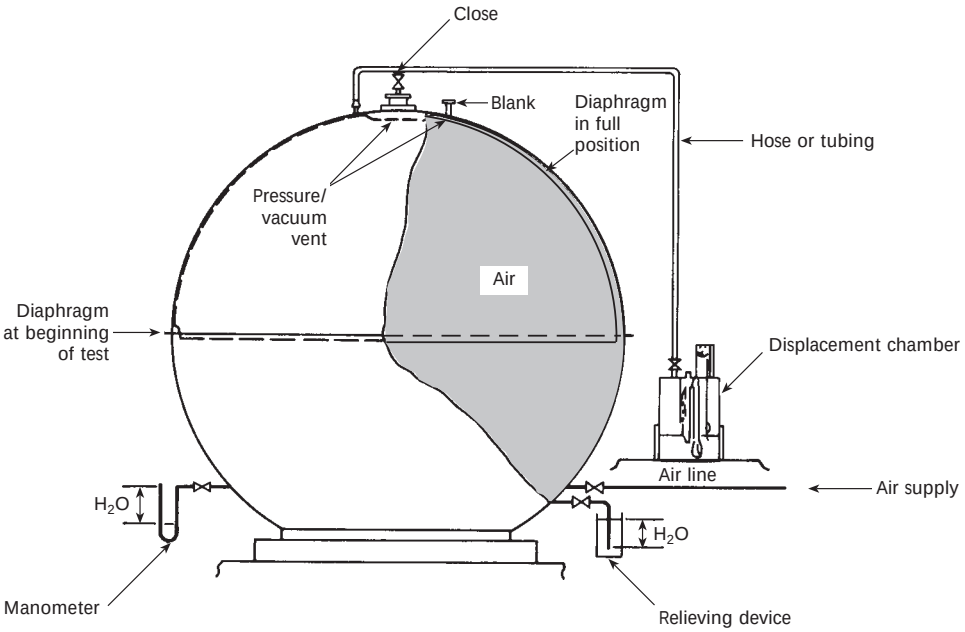
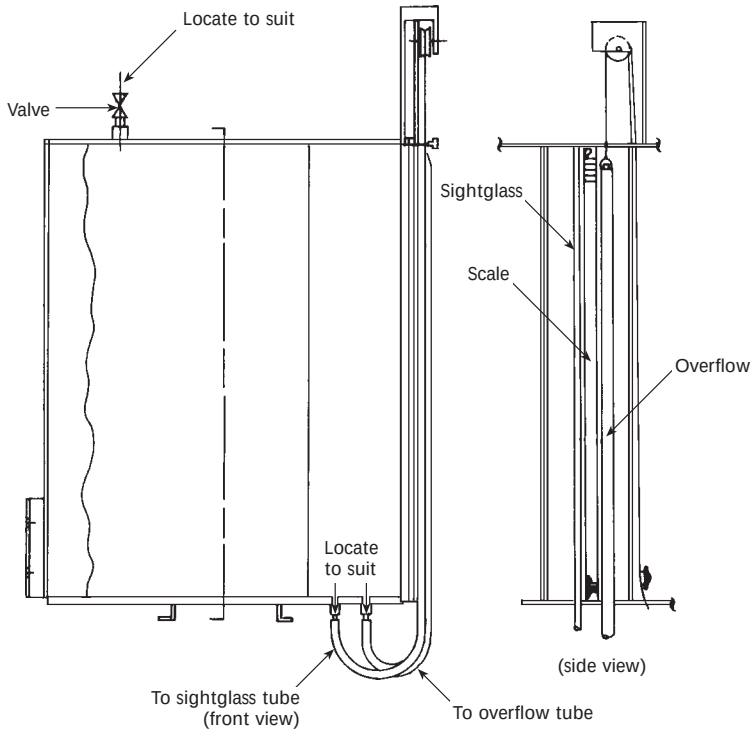


FIGURE 17. Detailed drawing of design of displacement chamber shown at lower right in Fig. 16.



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Procedure for Auxiliary Chamber Displacement Leakage Rate Test of Tank

Following these preparations, the leakage rate test for the tank system of Fig. 17 may be started. A surface thermometer is installed on top of the displacement chamber and this chamber is shaded from direct sunlight for the duration of the leakage test. A barometer is placed near the displacement chamber to measure changes in atmospheric pressure. The top end of the overflow tube is then adjusted to the same height as the water in the displacement chamber sight glass. Then the valve on the top of the displacement chamber is opened. Start the leakage rate test just as the water begins to move.

At the start of the test, record time t_1, surface temperature T_1 of the displacement chamber, barometric pressure P_b and displacement chamber sight glass water height H_1. During the leakage rate test, maintain the top end of the overflow tube at the same height as the height of the water by using the pulley cable attached to the overflow tube. This is done in the displacement chamber sight glass to prevent any pressure buildup in the displacement chamber. At the end of the required leak testing time, record the time t_2, the surface temperature T_2 of the displacement chamber, the barometric pressure P_{b2} and the displacement chamber sight glass water height H_2. Note the elapsed time $t_2 - t_1$ of test duration.

Calculating Total Leakage Rate for Leak Test Using Auxiliary Chamber

For the auxiliary chamber leakage rate test of the variable volume tank of Fig. 17, the following relations are used:

$$\textbf{(6)} \quad Q = \left(KH_2 P_{b2} \frac{293}{T_2 + 273} - KH_1 P_{b1} \frac{293}{T_1 + 273} \right) \div \left(t_2 - t_1 \right)$$

In Eq. 6, the SI units are those listed previously under Eqs. 2 and 3 with the addition of the height H of the water and K equal to the volume (cubic meter) per unit of height of water in the sight glass of the displacement chamber.

If the initial water height H_1 is equal to zero, the last term of Eq. 6 disappears and the leakage rate is given by:

$$\textbf{(7)} \quad Q = \frac{KH_2 P_{b2} \dfrac{293}{T_2 + 273}}{t_2 - t_1}$$

where Q is leakage (Pa·m^3·s^{-1} at 20 °C), P_b and P_m are pressure (pascal), V is volume (cubic meter), T is temperature (degree celsius) and t is time (second).

If mixed English units are used, the leakage rate Q is given at a standard pressure of 101 kPa (14.7 lb$_f$·in.$^{-2}$), a standard temperature of 20 °C (70 °F) and volume K_1 (cubic centimeter) per unit of height H of water in the sight glass of the displacement chamber:

$$\textbf{(8)} \quad Q = K_1 \left(H_2 \frac{P_{b2}}{14.7} \frac{530}{T_2 + 460} - H_1 \frac{P_{b1}}{14.7} \frac{530}{T_1 + 460} \right) \div \left(t_2 - t_1 \right)$$

If the initial water height H_1 is equal to zero, this reduces to the form of:

$$\textbf{(9)} \quad Q = \frac{K_1 H_2 \dfrac{P_{b2}}{14.7} \dfrac{530}{T_2 + 460}}{t_2 - t_1}$$

Auxiliary Chamber Accumulation Flow Meter Leak Test of Variable Volume Structure with Movable Diaphragm

Figure 18 shows schematically the leak testing equipment arrangement used for measuring leakage rates of a variable volume petrochemical storage vessel with a movable diaphragm, using the auxiliary chamber accumulation flow meter technique. Preliminary steps include removing the pressure vacuum vent and connecting a water manometer to this top fitting, connecting an automatic or manual pressure relieving mechanism to the tank and connecting a water manometer and a pressurizing line, all as shown in Fig. 18. Then, with a roof fitting open to the atmosphere, air is added to the tank until the movable diaphragm is at the top. Additional air is injected into the tank until its pressure is equal to the required test pressure. Add air to the tank or release air from the tank as necessary to maintain this pressure throughout the leakage rate test.

Flow Meter Accumulation Leakage Test Instrumentation

Instrumentation required for the auxiliary chamber flow meter technique of leakage testing of the system shown in Fig. 18

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includes a cube shaped bladder, a very low pressure differential integrating flow meter, a manometer, a barometer and a thermometer. After tank pressurization is complete, the previously tested cube shaped bladder (shown to the right of the tank in Fig. 18) is connected to the roof fitting at the top of the tank. The differential integrating flow meter is installed with a valved connection to this bladder. Both the manometer and the thermometer are included in the line to the flow meter. The barometer is placed near the bladder container.

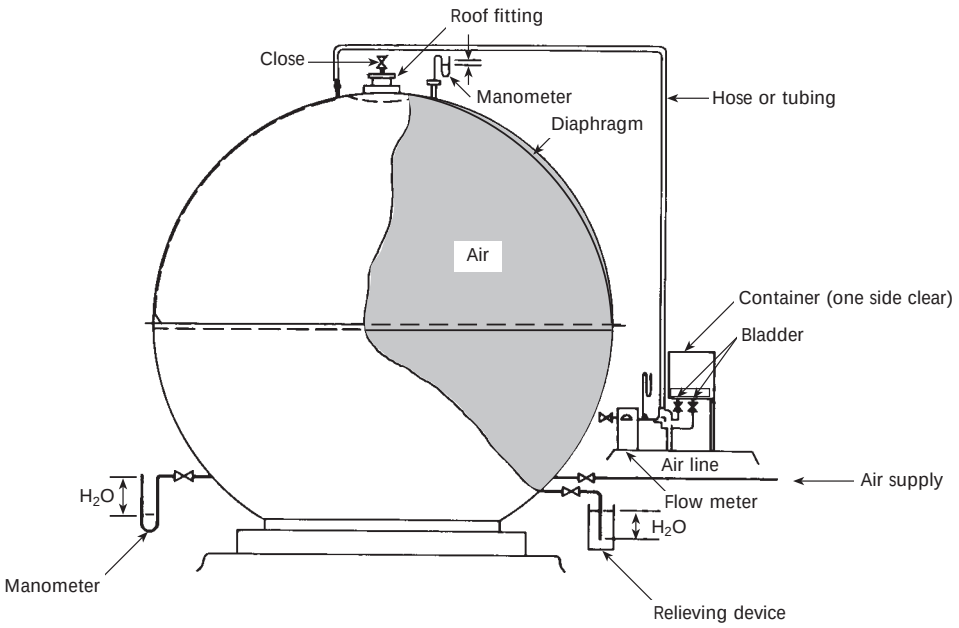
For preliminary leak testing of the instrumentation and its connections, the flow meter outlet valve is closed. The valve in the line from the roof connection to the bladder is opened, as is the valve between the bladder and the flow meter. Then, through a valved connection on the tank top access hole cover, the volume above the tank diaphragm is pressurized to a slight gage pressure sufficient to inflate the bladder. This pressure is shown by the manometer on top of the storage tank. Then bubble leak tests are made by applying solution film to a bladder and flow meter connections, the top manometer connections and the access hole valve and its connections. Any leaks indicated by the bubble test solution must be repaired and retested to show no leakage indications.

Leakage Rate Testing of Tank by Auxiliary Chamber Accumulation Flow Meter Technique

After completion of preliminary instrumentation system tests, the access hole valve is opened and the cube shaped bladder completely deflated. Then the access hole valve is closed and a plug installed in the valve outlet. The valve between the bladder and the flow meter is closed. The leakage rate test is started and the starting time t_1 is recorded. At the end of the required leak testing period, the time t_2 is recorded and the valve is closed between the bladder and the tank. At this time, the bladder must be at least just slightly less than completely inflated or the leak test will be invalid.

The bladder container (a cube shaped box with one side transparent) is now pressurized to the very low differential pressure specified for the flow meter. To measure the leakage accumulated within the flexible cube shaped bladder, the valve between the bladder and the flow meter is opened, as is the outlet valve from the flow meter. The pressure in the box containing the bladder is adjusted as necessary to maintain a specified water column height in the manometer connected into the line between the bladder and the flow meter. Record the manometer pressure m and the temperature T in the line to the flow meter. Also, record the barometric

FIGURE 18. Arrangement for auxiliary bladder accumulation flow meter technique for leakage rate test of variable volume petrochemical storage vessel with movable diaphragm within the tank.



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pressure P_b. As soon as the bladder is fully deflated, close both the outlet and inlet valves to the flow meter. Record the accumulated flow meter reading as the volume *V*.

Calculation of Leakage Rate for Accumulation Flow Meter Leak Test

The total leakage rate *Q* for the vessel of Fig. 18 is then calculated in SI units by use of the relation:

$$\textbf{(10)} \quad Q = \frac{V\left(P_b + P_m\right)\dfrac{293}{T + 273}}{t_2 - t_1}$$

where *Q* is leakage rate ($Pa \cdot m^3 \cdot s^{-1}$ at 20 °C), P_b and P_m are pressure (pascal), *V* is volume (cubic meter), *T* is temperature (degree celsius) and *t* is time (second).

In mixed English units, the leakage rate is given by:

$$\textbf{(11)} \quad Q = \frac{V\dfrac{P_b + P_m}{14.7}\dfrac{530}{T + 460}}{t_2 - t_1}$$

In Eq. 11, *Q* is leakage rate (std $ft^3 \cdot h^{-1}$ at 70 °F), *T* is temperature (degree fahrenheit), P_b is barometric pressure ($lb_f \cdot in.^{-2}$ absolute), P_m is manometer pressure ($lb_f \cdot in.^{-2}$ gage, converted from in. H_2O) and *t* is time (hour).

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CHAPTER 14

Leak Testing of Hermetic Seals

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PART 1. Characteristics of Gasketed Mechanical Hermetic Seals

Functions and Limitations of Hermetic Seals

By strict definition, a *hermetically sealed* device or system is one in which the gas or gases contained in the internal free volume of the sealed system cannot escape or be exchanged with any gas, vapor or liquid contained in the environment external to the sealed system.

In reality, such hermetic seals do not exist: given enough time, any gas should be able to permeate or diffuse through any known material. For this reason, either maximum rates of leakage or degrees of hermeticity are usually specified for particular operating environments and applications. If a device passes the specified leak testing requirements for the hermetic seal, this device can then be stated as being *hermetically sealed* to the degree specified. This does not imply, however, that the same part or device could not leak later because of its deterioration, mishandling, more stringent service, environment or other causes.

Typical Pressure Differentials Applied to Hermetic Seals during Leak Testing

The purpose of a hermetic seal is to prevent any transfer of gases or vapors from one area to another. It is commonly thought that a hermetically sealed device is used to preserve the contents of a container in some steady state. In the strict definition of the word *hermetic*, a pressure differential across the seal is not a requirement. However, many types of hermetic seals are used in service under a pressure differential of only 100 kPa (1 atm or 760 torr) or less. Thus, many leak testing specifications call for application of a testing condition that uses 100 kPa (1 atm) differential pressure across the seal for purposes of testing only the leakage rate of the seal.

The easiest way to provide a 100 kPa (1 atm) pressure differential across the pressure boundary of a hermetically sealed device or system is to draw a vacuum on one side of the seal while the other side of the seal is subject to atmospheric pressure. However, if it is decided to draw a vacuum on one side of a seal for test purposes, the leakage rate test data would obviously also be applicable to hermetic seals used for vacuum sealing as well. The alternative to drawing a vacuum on one side of the seal would be to pressurize one side of the seal to 200 kPa (2 atm) and allow leakage to occur through the pressure boundary of the sealed device to air at 100 kPa (1 atm).

Classification of Levels of Molecular Sealing by Vacuums

Leakage from atmospheric pressure to vacuum is typically molecular leakage because different gases leak at different rates in a given seal. In addition, liquid solutions will be selectively separated as they go through a seal having a leakage rate of the order of magnitude of 1×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-6} std $cm^3 \cdot s^{-1}$) or less. Selective permeability and diffusion of gases and liquids through solid sealing materials also depend on molecular structure. Thus, molecular sealing is needed for prevention of leakage into vacuum. Three commonly defined levels of molecular sealing are (1) commercial vacuum sealing, (2) hermetic sealing and (3) hard vacuum sealing. Vacuum levels are defined by the American Vacuum Society (see Table 1).[1]

Requirements for Sealing of Commercial Vacuum

Commercial vacuum systems typically operate at pressure levels between 10 and

TABLE 1. American Vacuum Society levels of vacuum.[1]

Vacuum Level	Pressure	
	SI Unit	(torr)
Low	1.01×10^5 to 3.3×10^3	(7.6×10^2 to 25)
Medium	3.3×10^3 to 1.33×10^{-1}	(25 to 1×10^{-3})
High	1×10^{-1} to 1.33×10^{-4}	(1×10^{-3} to 1×10^{-6})
Very high	1×10^{-4} to 1.33×10^{-7}	(1×10^{-6} to 1×10^{-9})
Ultra high	$\leq 1 \times 10^{-7}$	($\leq 1 \times 10^{-9}$)

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0.01 Pa (100 and 0.1 mtorr). Very seldom will these vacuum pressures be lower than 10 mPa (0.1 mtorr) — that is, of the order of 7 mPa (1×10^{-6} $lb_f \cdot in.^{-2}$). They are simply gross vacuums used in vacuum distillation of chemicals, vacuum melting furnaces and vacuum processes used to reduce the amount of water in food products, for example. However, effective vacuum seals are required for such systems to decrease the energy needed to evacuate the system to working pressures and to maintain the system's vacuum when the pumps are shut off for one reason or another. It should be emphasized that, at this vacuum level, the main concern is to minimize leakage across the seal.

Requirements for Sealing against Outer Space and Hard Vacuums

Hard vacuums are those with pressure below the level of 100 µPa (1 µtorr). These low pressures cause problems in sealing not encountered when sealing for higher vacuum pressures. The low pressures of hard vacuum cause volatilization of materials such as *plasticizers* in elastomer sealing materials. There can also be outgassing or sublimation of rubber materials into the vacuum. It has been suggested that most elastomers have vapor pressures between 10 mPa and 100 nPa (100 and 0.001 µtorr), so that they will vaporize selectively as pressures are reduced in a hard vacuum system.

Alternatively, it may be possible that vaporization of such polymers is preceded by depolymerization, whose products are subject to sublimation. The loss due to vaporization also depends on seal or device geometry, for only those monomers at the surface exposed to vacuum are removed. Certain rubbers do sublime at a rather rapid rate in the pressure range from 10 to 1 µPa (100 to 10 ntorr). This sublimation is a surface phenomenon and can erode exposed surfaces of sealing materials and gaskets. However, other polymers may be virtually unaffected at vacuum pressures as low as 100 nPa (1 ntorr) at room temperatures.

The most critical uses of seals for hard vacuums have been in the aerospace industry. Hard vacuum pressure conditions are attained at very high altitudes in the earth's atmosphere and in outer space. In these applications, it is essential that space capsules for human occupancy not lose the air needed to support life from their pressurized interior to the near vacuum of outer space. In addition, sealed devices that contain air to conduct heat to their external surfaces could lose their heat dissipation capability if this air were lost by leakage to the vacuum of outer space, with resultant damage to components and their performance.

Permeability Requirements for Hermetic Seals Having Very Small Leakage Rates

For hermetic seals to have very low leakage rates, it is essential that the seals and envelopes be fabricated from materials having very low permeabilities to passage of vapors, gases and liquids. Fluids travel directly through permeable materials by a process somewhat like osmosis. The main difference is that permeability refers to transmission of gases, whereas osmosis refers to transmission of liquids. Like solvents, different gases have different rates of travel through any specific permeable material. Conversely, a single type of gas travels through different permeable materials at different rates. For example, Table 2 indicates the air permeabilities of different types of rubber materials.[2] These permeabilities are expressed in terms of gas at a specific temperature passing per unit time through a specified area of a material with a certain thickness (1 m^2 of area with 1 m thickness), under a differential pressure of 100 kPa (1 atm). The SI unit of permeability is taken as $(Pa \cdot m^3 \cdot s^{-1}) \cdot (m^2 \cdot m^{-1})^{-1}$ or

TABLE 2. Air permeabilities of elastomers. Permeability is expressed in $Pa \cdot m^3 \cdot s^{-1} \cdot (m^2 \cdot m^{-1})^{-1}$ at 22 °C (72 °F), which would permeate through 1 m^2 of elastomer 1 m (40 in.) thick at a differential pressure of 100 kPa (1 atm) at a temperature of 80 °C (176 °F). The permeabilities given are for typical reinforced compounds: special compounding techniques can substantially change these rates.[2]

Elastomer	Permeability 10^{-12} $Pa \cdot m^3 \cdot s^{-1} \cdot (m^2 \cdot m^{-1})^{-1}$ or 10^{-7} std $cm^3 \cdot s^{-1} \cdot (cm^2 \cdot cm^{-1})^{-1}$
Butyl	0.32
Thiokol™	0.37
High acrilo nitrile	0.41
Hypalon S-2™	0.7
Kel-F™	0.80
Low acrilo nitrile	0.8
Viton A™	0.88
Polyurethane	0.97
Chloroprene	0.98
Acrylon EA-5™	1.5
Hycar 4021™	1.8
GR-S™	2.9
Natural rubber	4.4
Fluoro-rubber 1F4	9.6
Fluoro-silicone	12.8
Silicone	45.0

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(1 std $cm^3 \cdot s^{-1}) \cdot (cm^2 \cdot cm^{-1})^{-1}$ at 80 °C (176 °F) and at 100 kPa (1 atm) differential pressure. Because the quantities are very small, it is expressed as $(\mu Pa \cdot m^3 \cdot s^{-1}) \cdot (m^2 \cdot m^{-1})^{-1}$. This unit reduces to $\mu Pa \cdot m^2 \cdot s^{-1}$ but is unrecognizable in this form. In Table 2, butyl has a permeability of 3.2×10^{-13} $(Pa \cdot m^3 \cdot s^{-1}) \cdot (m^2 \cdot m^{-1})^{-1}$ whereas silicone has a permeability of 4.5×10^{-11} $(Pa \cdot m^3 \cdot s^{-1}) \cdot (m^2 \cdot m^{-1})^{-1}$ or more than 100 times greater.

Requirements for Seals to Maintain Internal Atmospheres in Near Space Systems

Hermetic seals are used to contain breathable air atmospheres in aircraft that fly in near space vacuum conditions in the earth's atmosphere at altitudes up to 30 km (100 000 ft). Leak testing for this type of vacuum imposes one more condition in addition to those required for hermetic sealing of devices whose seals are not subject to vacuum pressure differentials across their pressure boundaries. This unique condition is the slight movement of the seal gasket in response to the 100 kPa (14.7 $lb_f \cdot in.^{-2}$) pressure differential repeatedly impressed across it. Such movement is aided by a small shrinkage of rubbery materials used for flexible seals. In aircraft, movement of seals used in access doors and wing panels was overcome by seals that were molded in place. Many vacuum seals use the same closely controlled sealing principles to attain completely reliable and reusable performance.

The movement of a gasket or seal caused by low pressure and shrinkage of the rubbery element of mechanical seals in a vacuum environment acts to displace the flexible sealing element just enough to break the original line of sealing contact. Not enough force is generated by the system pressure to reestablish a seal on the interfaces of the sealing surfaces if contact is once broken (see Fig. 1). In some seal designs, the ratio of rubber to void space within the seal is controlled to eliminate this problem of low pressure rollout. This prevents slight movements of seal. It also creates high contact loading at the sealing line that increases the flow of the flexible gasket material into the microirregularities of the sealing interface. This reduces or eliminates the problem of shrinkage of the rubbery sealing material when seals are subjected to near space vacuum levels of 100 to 1 µPa (1.0 to 0.01 µtorr).

Requirements for Seals to Maintain Internal Atmospheres in Outer Space Systems

There are two critical requirements for seals and gaskets that are of importance in the hard vacuum and other operating conditions of outer space environments (see Table 3).[3] One is the effect of hard vacuum on the physical characteristics of flexible sealing materials; the second is the effectiveness of all seals in maintaining the earth's atmosphere within vehicles or individual components. Many space vehicle components (as well as astronauts) can function only in a preserved earth's surface environment. Consequently, seals that can cope with molecular leakage are required to maintain pressure in these components for long time periods. Surveillance and communication satellites, as well as deep

TABLE 3. Characteristics of outer space environment.

Low pressure
10^{-4} to 10^{-12} Pa (10^{-6} to 10^{-14} torr) dependent on altitude
Low density
10^{-9} to 10^{-17} $kg \cdot m^{-3}$ (1.7×10^{-9} to 1.7×10^{-17} $lb_m \cdot yd^{-3}$)
Chemical composition
Dissociated molecules
Ions
Thermal radiation, influencing vehicle temperature
Infrared solar radiation
Earth's albedo
Infinity radiation sink (0 K)
Early space probes indicate thermal equilibrium at 70 °C (160 °F)
Other solar radiation
Visible radiation
Ultraviolet radiation
X-radiation
Cosmic radiation
Electromagnetic (gamma rays, X-rays)
Primary particles (protons, atomic nuclei)
Secondary particles (electrons, positrons, mesons, neutrons)
Van Allen belt radiation (protons or electrons)
Meteoric particles
Penetration
Abrasion
Force fields
Electromagnetic
Gravitational
Conditions of Human Origin
Propulsion products
Vehicle outgassing
Acceleration
Vibration
Space debris
Hostile action

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FIGURE 1. Rollover of loose gasket that breaks hermetic seal at original contact surface, and of molded-in-place seal that resists loss of sealing contact as the pressure across seal varies during service: (a) O-ring in improper gland for low pressure sealing; (b) molded-in-place seal maintains same configuration regardless of pressure level.

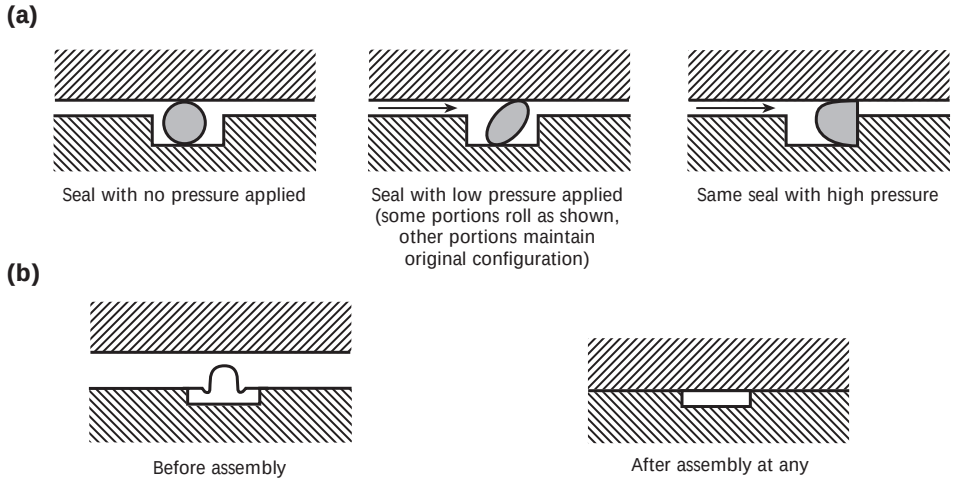

TABLE 4. Leakage rates for elastomer at vacuum conditions.[3]

Test Pressure		Leak Testing	Air Leakage Rate	
Pa	(torr)	Method	μPa·m³ per mm of Seal per Annum	(std cm³ per inch of Seal per Year)
1×10^{5}	(7.6×10^{2})	Radioactive gas	3	(0.0008)
6.7×10^{-4}	(5×10^{-6})	Helium leak detector	600	(0.1500)
2.9×10^{-5}	(2.2×10^{-7})	Pressure decay (ion pump)	4.4	(0.0011)
2.9×10^{-6}	(2.2×10^{-8})	Pressure decay (ion pump)	4900	(1.2200)

space probes, that are designed to operate in deep space for many years are in special need of adequate molecular sealing to prevent loss of internal atmospheres.

One way to provide long term sealing against outer space vacuum environments is to select envelope and sealing materials that have low vapor pressures. For example, hard vacuums are known to erode metals such as cadmium and magnesium. Elastomeric materials that have low vapor pressures (see Table 2) must be used for flexible sealing elements. A seal made of silicone rubber would allow the pressurized air within an outer space vehicle component to leak into the void of space. The effect of progressively harder vacuum levels rapidly increases the out-leakage in outer space. For example, the leakage rates measured at 3 μPa (22 ntorr) were $\times 10^{3}$ greater with the same material than when measured at 30 μPa (220 ntorr).

A weight loss test of this material made by exposing a microtome of material to a vacuum of 3 μPa (20 ntorr) showed a weight loss of about 31 percent whereas at a pressure of 13 μPa (0.1 μtorr) the weight loss was only 2.0 percent. The typical leakage rate for this material at various pressures (by using appropriate leak testing techniques for each pressure range) were reported (Table 4). The leakage rate obtained when using the pressure decay test technique and air as the medium were lower than those found by adjusting the helium leakage rates for molecular weight ratios. This suggests that the viscous flow rate assumption may not apply if the flow of air through a leak path is restricted by forces of molecular attraction in a rubber polymer, for example.

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PART 2. Characteristics of Hermetically Sealed Packages

Some Functional Requirements for Hermetically Sealed Packages

In general, packages for hermetic devices are designed to meet numerous specific requirements based on anticipated service and environmental conditions. Some common requirements include the following.

1. The package must provide a mechanically stable device enclosure so that reliability screening tests do not affect the operational characteristics of the device. Typical screening tests for high reliability service applications involve subjecting the devices to vibration, drop shock, centrifuge, thermal shock test conditions and leak testing.
2. The package must provide electrical feed-through connections from the internal electronic device to the package externals electrically insulated from the package that supplies power to the device.
3. Some packages must provide means for heat transfer away from an electronic device within the package to avoid damage by overheating. Operating parameters can change significantly with variations in temperature.
4. The package must be resistant to corrosion from external environments.
5. The package must be hermetic (sealed against air or gas flow in or out) to prevent exposure of the internal device to ambient environments, particularly those with high relative humidity (high moisture content). The electrical parameters of discrete elements comprising an integrated circuit can change values or be degraded when exposed to a moist environment. The proper function of relay contacts can be degraded by contaminates entering the device. Or, the reliable performance of an explosive (such as an air bag firing system) can be impaired by moisture.
6. The package must provide a shield against light from external sources because semiconductor junction device parameters are light sensitive. (Some electrooptical devices are controlled or switched on or off by light signal inputs.)
7. The package must isolate the internal electronic package from external electromagnetic radiation. Also, in some instances, the package must act as an electromagnetic shield to prevent internal circuit radiation from permeating the enclosure and adversely affecting the operation of other external circuitry.
8. The package must provide a physical shield for mechanical contacts and moving parts in relays and similar devices. It must physically protect explosive powders and charges in ordnance devices.

Conditions Causing Leaks in Glass-to-Metal and Glass-to-Ceramic Seals.

The reliability of many devices depends on the degree of package hermeticity. For many military or high quality devices, the critical manufacturing process is the formation of the glass-to-metal or glass-to-ceramic seals. The quality of the final seal determines the degree of package hermeticity.

Generally, this manufacturing process is well controlled. However, much more critical processes are required to provide hermetic seals around a large number of electrical lead wires when maintaining electrical insulation between the various parts of the package. Manufacturing conditions that can affect both the quality of initial seals and the degradation of glass-to-metal or glass-to-ceramic seals when they are exposed to severe environmental or operating conditions such as high temperatures, thermal shock and mechanical stressing include (1) differences in coefficients of thermal expansion of metal and glass or ceramic materials forming the seals, (2) uniformity of oxide layers grown on the metal surfaces to aid bonding to glass or ceramics, (3) cleanliness of the metal surfaces before growth of these oxide layers and (4) quality or volume of glass surrounding the lead wires and metal or ceramic surfaces.

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Functions of Oxide Layers in Glass-to-Metal and Glass-to-Ceramic Seals

A slight mismatch in the thermal coefficients of expansion of metal and glass, for example, is sufficient to cause a separation of the metal from the glass at some temperature, thus permitting possible leakage. To prevent such separation between metal and glass in hermetic seals, an oxide layer is grown on the metal surfaces before sealing them to the glass. The fusing then results in a continuous transition from (1) the metal to (2) metal oxides to (3) metal oxides in glass and finally to (4) the glass. The ceramics most commonly used for hermetic seals are themselves metal oxides, so their sealing operation involves formation of transitions (1) from ceramic (2) to ceramic in glass and finally (3) to glass.

It is essential to remove oxides, oil or particle contaminations from metal surfaces before processing them to grow the oxide transition layers used in glass-to-metal seals. The cleanliness of these metal surfaces before oxide growth and careful control of the oxidation process that forms the oxide layer are critical factors in control of the thickness of the oxide layer. In general, any surface contamination of the materials used during the sealing process will produce a nonuniform seal. Uniformity of oxide thickness is required for high quality hermetic seals.

Process of Manufacturing of Solder Glass Dual Inline Circuit Packages

Dual inline packages (DIPs) are presently used for more than 50 percent of all hermetic integrated circuits, of which tens of millions or more are manufactured each year. In fabricating dual inline packages, the glass used to seal the metal lead frame (for external connections) with the ceramic base and cap is introduced as a slurry mixture of vitreous glass beads suspended in an organic binder. In general, the sealing process involves the heating of this mixture in such a way as to allow the organic binder to evaporate while the glass is transformed from its initial vitreous or liquid state to a devitrified or crystalline state that forms the seal.

During manufacture in a particular process, the devitrification process begins with an initial heating in a belt furnace of an assembly consisting of (1) the ceramic base with (2) the lead frame resting on (3) a slurry layer of glass beads and organic binder. During the heating, the glass melts, the binder begins to burn off and the frame sinks into the glass and become partially bonded to the base. Devitrification of the glass begins during this process and continues during the subsequent semiconductor die attachment. The process for attaching the semiconductor die involves a glass-to-metal eutectic bond made at temperatures as high as 425 ºC (800 ºF).

After wire bonding and visual inspection, the ceramic cap is put in place and the final sealing is performed at a temperature in the range from 450 to 525 ºC (850 to 975 ºF). If all elements of the manufacturing process are completed successfully, the resultant dual inline packages may be *hermetically sealed.* If the manufacturing process uses improper materials or is not precisely controlled, the resultant dual inline packages may become *leakers.*

Causes of Leaks in Dual Inline Circuit Packages

Manufacturing Conditions

Seal failures in solder-and-glass dual inline packages may be caused by inadequate control over the manufacturing processes (described just previously) or from improper handling. Inadequate control of the heating process and the quality and mixture ratio of the glass and binder can result in a leaking seal. If the glass is exposed to excessively high temperature during sealing, the organic binders will not burn off before the glass devitrifies (is made hard and opaque, as by prolonged heating). This can result in gases frozen into the seal in small, bubblelike cavities. Structurally weak seals, which may be porous, can also result. (Such excessive temperatures could occur if, for example, a furnace belt were stopped by a power failure, if a package base were left too long on a heater block in preparation for die attachment or if the die attachment process were too slow.)

Cavities and porous seals can also occur if the temperature at which the frame is attached to the base is too low. In this case, the glass under the frame does not outgas sufficiently. Blow holes can then result during the final sealing operation due to the buildup of gas pressure. (Such insufficiently high temperatures might be caused, for example, by increasing the speed of the furnace belt to shorten the processing time.)

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Some electro tin plating processes used to provide a bright finish on the external lead frames of the sealed packages can also cause leaks. Acids used in this plating process can deplete the glass, particularly adjacent to the metallic lead frame. Such leaks are most prevalent in parts that have been exposed to plating solutions for extended periods of time. These leaks could also result in devices that have been replated, as in attempts to strip and replace tin plated leads that oxidized during burn-in test.

Sealants

Care must be exercised in controlling the seal materials used in manufacture of dual inline integrated circuit packages. Too high a concentration of the binder (in the glass bead mixture) can result in failure to burn off all of the binder during the sealing process. The glass mixture used for sealing must also be evaluated to ensure that it does not exhibit any abnormal devitrification behavior. For example, some lots of glasses have exhibited a partial devitrification at an abnormally low temperature that has prevented the complete removal of the binder during the sealing process. Another potential problem with the incoming glass is that additives designed to adjust the thermal coefficient of the expansion may not be in the proper balance. Integrated circuit packages made with such glasses may fail in later temperature cycling tests. Such undesirable types of glasses can be eliminated in incoming inspection by making thermal expansion tests on each lot of glass materials received.

Improper Handling

Inadequate control of the mechanical handling of integrated circuit (IC) packages can result in the degradation of well made package seals. The application of excessive physical stress on the package leads can be transmitted to the brittle glass seals, causing fracture and cracking that permit leakage to occur. Examples of handling procedures that could apply excessive stress to dual inline integrated circuit packages include (1) clipping excess length from leads, (2) excessive or repeated flexing of leads, (3) dropping of integrated circuit packages, possibly in automatic handling and loading operations, and (4) excessive heat when soldering integrated circuits to printed circuit boards.

Leaks Caused by Improper Handling of Other Hermetic Packages

The improper handling of mechanical seals in the manufacturing process, the testing process and the customer application process are major causes of damage to hermetic seals. Many glass-to-metal feed-throughs frequently depend on a meniscus seal at the surface of the pin-to-glass interface. The seal is easily damaged or broken by handling. Such devices as relays use up to a dozen of these feed-throughs, all of which are vulnerable to this type of damage. A commonly applied technique for this is an *adjunct sealant* in the form of an epoxy film applied over the glass-to-metal seals. It is allowed and defined in the military standards for relays. Adjunct sealants are known to be only temporary under most conditions. Relay socket insertion is endless and causes damage to pins due to misalignment in and out of the sockets, often the fault of the technician.

Hybrid electronic devices, radio frequency devices and many of the very large electronic, electromechanical devices are subjected to extreme thermal cycling that induces severe stresses on hermetic seals and makes them additionally vulnerable to subsequent handling.

The ordnance devices are frequently installed in physically abusive environments. Their handling is often damaging to seals due to lead wire soldering or welding and potting.

Manufacturing Conditions Causing False Indications of Leakage in Packages

So-called false indications of leakage can result from causes such as surface porosity of the seal material used in manufacture of dual inline packages for integrated circuits or electronic devices. Surface porosity could result from inadequate control of the seal materials and the sealing processes. Such porosity can trap the leak testing tracer gas or liquid so as to produce an erroneous indication of a hermeticity failure. *False cavities* with volumes nearly as large as 10 mm^3 can sometimes be found in the seal materials near the end of the integrated circuit package, totally separated from the internal cavity of the package.

Perhaps the amount of seal material has been selected only on the basis of providing a desired thickness of seal in the area of the lead frame. If too little seal material is used, it may happen that the surface tension of the liquid glass will pull

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the seal material toward the frame to leave a *false cavity* at either end of the integrated circuit package during the sealing process.

The glass in this area may also be weaker than that near the frame and package cavity. In this case, cracks or a porous seal may develop to provide a leak to the outside of the package. Such a false cavity would give a leakage indication that could not be distinguished from a true leak to the device cavity by using either the helium or radioactive tracer gas leak testing techniques. In some cases, a false leak can be verified by using a liquid dye penetrant test. In all cases it should be noted that specifications state that the package shall not leak. Therefore, it is valid to reject these leakers.

Handling Conditions Causing False Indications of Leakage from Hermetic Packages

False indications of leakage, usually seen only during helium leak testing, can be caused by careless handling that leaves fingerprints, pencil marks or dirt on the surfaces of hermetically sealed packages. A single fingerprint has been shown experimentally to increase an initial leakage rate reading of 10^{-8} Pa·m^3·s^{-1} (10^{-7} std cm^3·s^{-1}) to an erroneously high indication level of 5×10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}). This would correspond to an increase of 50 times the true leakage rate before the fingerprint was made. Treating electronic packages with a solvent rinse, followed by a high pressure air blast, after helium pressurization, can reduce this problem significantly. Some users have found that a 10 min bake at 100 °C (210 °F), following the helium pressurization, improves the repeatability of the helium leak testing operation. Surface gas rejections are almost nonexistent with the radioactive gas test technique.

Elevated Temperature Testing Conditions Causing Leaks in Hermetic Seals

In addition to standard hermeticity tests performed at room temperatures, it has often been very useful to perform similar tests at elevated temperatures in the range from 55 to 85 °C (131 to 185 °F). This range includes the typical operating temperatures of many sealed devices. It has been found that some devices that pass fine leak tests performed at room temperature will fail when tested at higher temperatures. Such leaks are apparently the result of weak seals that fail under the stress resulting from differences in the thermal coefficients of expansion of the constituent parts of the seal. This behavior is not restricted to one type of package but rather has been found in a wide variety of integrated circuit packages.

Analyses have also revealed that high temperature burn-in tests and baking before gas analysis alter the types and concentrations of gases of the ambient atmosphere within the integrated circuit packages, especially with fine leakers. For example, such tests have resulted in effects such as (1) increases in the concentration of hydrogen, (2) decreases in the concentration of water and (3) increases or decreases in the concentration of carbon dioxide.

The outgassing of package materials during a reliability stress testing at elevated temperatures is a possible source of contamination of the internal atmosphere that could be detrimental or degrading to the operational characteristics of the hermetically sealed electronic device. In other cases, the thermal shock tests (specified in military qualification procedures) had fractured the glass-to-metal seal in the lead frame of integrated circuit packages. These fractures, that allowed water vapor to enter into the package interiors during the subsequent water quench, went undetected in the hermeticity leak tests that followed.

Sources of Water Contamination during Manufacture of Sealed Circuit Packages

The contaminant of greatest concern in the internal ambient atmospheres of sealed integrated circuit and electronic component packages is water (both adsorbed and in the form of water vapor). It is generally believed that there is a relationship between the relative concentrations of oxygen and water that will penetrate a leaking package, with the ratio of oxygen to water content near seven to one. However, there are no general data to indicate at what concentration moisture has an adverse effect on the sealed electronic devices. The ideal situation would be that in which the internal gas atmosphere in the sealed integrated circuit package was pure nitrogen. The presence of appreciable concentrations of moisture makes the measurement of package leakage rates more difficult.

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PART 3. Techniques for Gross Leak Testing of Hermetically Sealed Devices

Definitions of *Gross* and *Fine* Leak Testing of Hermetic Seals

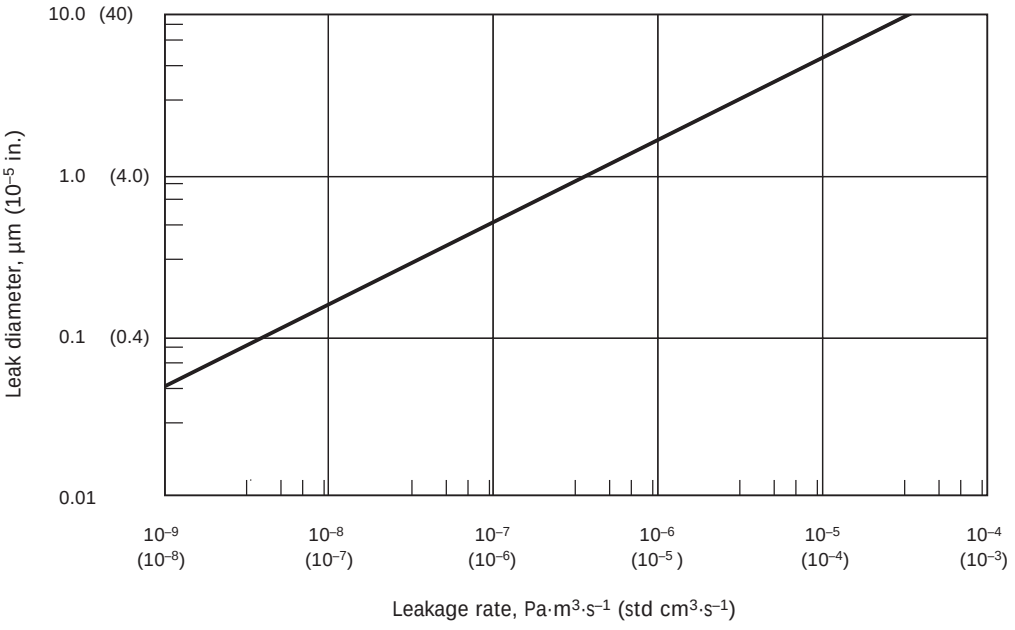
The *gross* end of the range of leakage rates of hermetic seals is defined as those devices with leakage rates grater than 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}). In spite of the fact that these leakage rates are called *gross*, the leaks are those that would occur with circular holes with diameters in the range of micrometers. Figure 2 shows the theoretical relationship between equivalent circular hole size and leakage rates. Actually, the leaks in hermetic seals are not circular holes, but rather consist of cracks or pores of various sizes. Thus, the task of leak testing such devices is more difficult than would normally be implied by the word *gross*. In hermetically sealed packages, overall leakage rates greater than 10^{-8} Pa·m^3·s^{-1} are typically found to be harmful.

The fine end of the range of hermetic seal leakage rates extends downward from 10^{-7} Pa·m^3·s^{-1} to an indefinite limit, perhaps as low as 10^{-12} Pa.m^3·s^{-1}. For high reliability uses, hermetically sealed devices with leakage rates greater than some prescribed value are rejected. This limit may be as small as 1×10^{-9} Pa·m^3·s^{-1} (1×10^{-8} std cm^3·s^{-1}). Leak test screening has been necessary for achievement of high reliability systems, not just for military uses but for many other uses. Typically, these leak tests may cause rejection of about two percent of the high quality devices in production tests, but in some cases rejection rates as high as 30 percent are found in critical military tests. Failures of hermetically sealed semiconductor packages have lead to about 13 percent of the operational failures that occur in high reliability integrated circuit electronic systems.

Although hermetic seal testing is both necessary and extensive in industry, there is still a lack of a sound technical basis for clear cut specifications for maximum allowable leakage rates.

It has been established by experiment and specified in the military standards, that all *wet* or *liquid immersion* gross leak test procedures interfere with the reliability of the dry gas leak testing of hermetic seals.

FIGURE 2. Theoretical relation of leakage rate to ideal circular hole leak diameter, assumed for hermetic seals. Empirical test data obtained with capillaries ranging from 8 to 30 mm (0.3 to 1.2 in.) in length.



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Major Leak Testing Techniques Used for Gross Leak Testing of Hermetic Devices

Gross leak testing techniques used for testing hermetically sealed electronic devices in the gross leakage rate range from 10^{-7} to 10^{-3} Pa·m^3·s^{-1} (10^{-6} to 10^{-2} std cm^3·s^{-1}) have included (1) liquid dye penetrant tests, (2) elevated temperature bubble tests, (3) back pressure bubble tests, (4) weight gain leak tests, (5) holographic leak tests and (6) krypton-85 pressurization techniques.

Each of the preceding leak tests will detect leaking devices. However, the choice of which test to use depends on the type of package used to enclose the device, as well as on the choice of additional fine leak tests to be used on the same device packages. Cleanliness is absolutely essential for accurate leakage testing with hermetically sealed device packages. Dye penetrant leak tests are used for glass enclosed devices (where the dye can be seen through the device enclosure in case of leakage) and for failure analysis of devices whose enclosures can be opened to reveal leakage paths during destructive failure analyses. Elevated temperature bubble testing is used for testing the gross leaks into the 10^{-5} Pa·m^3·s^{-1} (10^{-4} std cm^3·s^{-1}) range. Back pressure leak testing is used for leak testing into the 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}) leakage range. Weight gain leak tests are used for leak testing to the limit of gross leakage ranges of 10^{-6} Pa·m^3·s^{-1} (10^{-5} std cm^3·s^{-1}). The radioactive krypton-85 back pressurization is being used for gross leak testing of both devices with cavities and for zero cavity devices. The application with cavities uses an extension of the fine leak applications. The zero cavity technique uses the adsorptive gettering of krypton-85 gas by activated carbon that has been enclosed within the device.

Dye Penetrant Gross Leak Testing of Hermetically Sealed Electronic Devices

A visible dye liquid penetrant is commonly used for gross leak testing of clear glass hermetic packages for electronic devices. The technique used is to apply external pressures of 300 to 600 kPa for bombing times of the order of 3 to 10 h to the device packages when submerged in fluorescent dye or in a commercial dye penetrant liquid. The package exterior surfaces are then washed to remove excess penetrant from the outer surfaces. The clear glass devices are then examined visually for dye indications trapped within the cavities of the transparent device packages.

The use of dye penetrant for leak testing of opaque packages for electronic devices has not been found to be reliable. For example, 100 devices that indicated large leaks during a fine leak test were subsequently tested for gross leaks with the dye penetrant leak testing. A commercial dye penetrant was used and the devices were bombed during immersion in this liquid penetrant for 3 h at a pressure of 600 kPa (90 lb$_f$·in.$^{-2}$ gage). After their exterior surfaces were cleaned, these devices were placed in a vacuum chamber to produce a higher differential pressure. They were carefully observed for evidences of the dye leaking back out of the packages. After having been sorted into *good* and *bad* groups, these packages were repressurized and opened at atmospheric pressure to determine if there was any evidence of dye within the cavities. In these tests, 19 percent of the "good" devices (that had shown no evidence of leakage in the dye penetrant tests) were found to contain dye within the sealed cavities. Of the rejected "bad" devices that showed prior indications of leakage in the dye penetrant tests, 3 percent were found to have no dye within the cavities. After duplications of the dye penetrant tests on other devices produced similar unreliable test indications, it was concluded that the dye penetrant gross leakage test was not useful for leak testing of devices sealed in opaque packages.

Limitations and Advantages of Dye Penetrant Tests of Electronic Devices

The primary limitations of dye penetrant leak testing of hermetically sealed electronic devices are (1) interference caused by particulate matter in the dye penetrant liquids that plug up holes and (2) improper cleaning of the exterior surface of the devices under test, following pressurization, to remove the excess surface penetrant. Inadequate cleaning can result in rejection of good devices. Overcleaning can wash the dye out of the leakage paths in device packages, in the largest part of the gross leakage range.

However, the dye penetrant test technique is useful as a failure analysis technique that can be used not only to confirm the existence of the leak, but sometimes also to identify the leakage path through the device packages. If the cavity within the device package has not filled completely with the dye penetrant, the dye indications usually make the leakage path definable.

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Elevated Temperature Bubble Leak Testing of Hermetic Devices

Elevated temperature bubble testing is performed by immersing the hermetically sealed device package under test in a hot fluid and observing any indications of bubble emissions caused by gas escaping through leaks as a result of the increase in internal temperature and contained gas pressure. For microcavities, the normal technique is to immerse the devices in 125 °C (257 °F) perfluorocarbon liquid for a period of 30 s, observing any bubble emissions and rejecting the sealed package for any leakage indicated by a stream of bubbles. A stream of bubbles is defined as a visual indication of more than two bubbles originating from the same point on the package and typically rising through the immersion fluid.

In immersion bubble testing, most of the hermetically sealed devices that are rejectable will be characterized by a very small stream of bubbles that will start to be visible after about 15 s of immersion in the heated liquid bath and last until test object removal after 30 s. Because these bubble streams are sometimes very fine and hairlike in appearance, it is necessary to illuminate the immersion bath inspection zone with an intense light (such as the lamp used with a microscope or a projection lamp). The background against which the bubble emission stream is to be observed should be dull and nonreflective to provide high contrast for the illuminated bubbles. The observer should use an optical magnifier of at least 3× magnifying power that provides a field of observation large enough that all of the visible surface of the device being tested can be seen at one time. The angle of view should be at least within 30 degrees of the perpendicular to the area under observation. Inspectors should have adequate vision acuity and patience to persist in observation for the entire immersion period.

Control of Interfering Effects in Elevated Temperature Bubble Leak Testing

Effects that interfere with the ease and reliability of elevated temperature bubble testing of sealed devices include (1) contamination on the surfaces of devices under test that causes *false bubbling* or bubble emission from adsorbed gases or sources other than leaks, (2) particulate matter in the perfluorocarbon immersion bath that may look like a bubble stream and (3) bubbles generated from the heaters used in the immersion baths. Identification tags and coating are the most common causes of contaminants on the surfaces of devices under test. Lint is the major contaminant in the immersion bath itself.

Surface contamination of microcircuit packages can be eliminated by careful selection of the time and location during production processing of devices under test. Alternatively, the devices under test could be subjected to precleaning to remove surface contaminants and adsorbed gases before immersion in the bubble test bath. Cleanliness of the immersion bath fluid is best controlled by a circulated filtration system and by prerinsing of the test specimens. The problem of emission from heater elements can be eliminated by the immersion types of heaters or by sand placed so that external heaters are not in direct contact with the glass container used for the immersion fluid.

Back Pressure Bubble Emission Leak Testing of Hermetic Devices

Back pressure bubble testing is one of the more commonly used gross leak tests. Many persons start to use this test when they have the first flat tire on their bicycle, immersing the pressurized tire in water to see where bubbles indicate leak locations. Generally, the back pressure test is conducted by immersing a test specimen that has an internal pressure greater than 100 kPa (1 atm) into a liquid bath and by watching for streams of bubbles coming from it. The higher this internal pressure, the greater the chance of leak detection.

The normal technique used to provide back pressurization of sealed microcircuit packages is first to evacuate each device to a vacuum of 700 Pa (5 torr absolute) for a period of 1 h. Then the devices are pressurized when immersed in a perfluorocarbon liquid with a low boiling point, that is, 50 °C to 57 °C (122 °F to 135 °F). Pressurization occurs either (1) at an absolute pressure of 620 kPa (75 $lb_f \cdot in.^{-2}$ gage) for 2 h or (2) at a gage pressure of 200 kPa (30 $lb_f \cdot in.^{-2}$ gage) for 10 h. The test devices are then air dried for a period of about 2 to 4 min before leak test processing by the elevated temperature bubble testing technique.

In back pressure testing, the time between removal of the test devices from the pressurizing fluid and their immersion into the hot bath of bubble testing fluid must be carefully controlled. If the device is immersed too quickly, it will be covered with escaping bubbles (and look like carbonated water). This makes it very

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difficult to decide whether bubbles indicate true leaks. If the test devices are dried for an excessive time period, the fluid that had been forced in during pressurization will have time to escape and evaporate. The best range of waiting time between removal from the pressurization bath and immersion into the bubble testing bath is in the range of 2 to 5 min. However, the size of the sealed electronic package and its material also have to be considered to select the optimum wait time.

Characteristics of Perfluorocarbon Test Liquid Used in Weight Gain and Back Pressure Bubble Leak Testing

Figure 3 shows that the rate of flow of a perfluorocarbon test liquid is about 0.1 mg·min^{-1} under a pressure of 700 kPa (100 lb$_f$·in.$^{-2}$ gage) through a leakage path that would have a standard leakage rate for air of 5×10^{-7} Pa·m^3·s^{-1} (5×10^{-6} std cm^3·s^{-1}). The perfluorocarbon liquid shown in this example has a density of 1.7 g·cm^{-3} at 25 ºC (77 °F). This liquid boils at 50 ºC (122 °F).

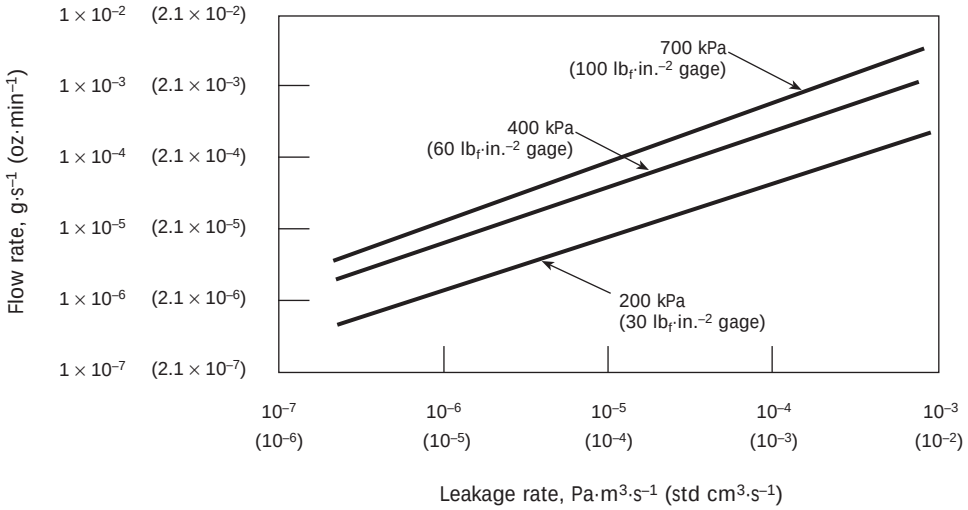
Thus, it is apparent that, if a sealed electronic device leaks and in-leakage of this fluid occurs during a bombing time of from 2 to 10 h, the pressure rise will be quite significant when the liquid is brought to a boil at 50 ºC (122 °F). This extends the range of leakage measurements to leakage sensitivities considerably better than those attainable with elevated temperature bubble tests made with no pressurization.

Weight Gain Leak Testing of Hermetically Sealed Electronic Devices

The measurement of weight gain of hermetically sealed electronic devices is not a widely used technique for their gross leak testing. The technique can be quantitative and the leakage rate can be determined if the bombing time and pressure are known because the rates of flow of the perfluorocarbon test fluid are known.

The test procedures used in weight gain leak tests include the following steps: (1) weigh each specimen to be tested, (2) pressurize the specimen in a perfluorocarbon liquid for a specific time period at a specified pressure, (3) reweigh the specimen to determine if liquid in-leakage has occurred and (4) compare the pretest and posttest weight values.

FIGURE 3. Leakage flow rates observed with liquid fluorocarbon test fluid under pressures of 200, 400 and 700 kPa (30, 60 and 100 lb$_f$·in^{-2} gage), passing through leaks with known atmospheric pressure air leakage rates. The gas leakage rates of the capillaries were measured using one atmosphere of helium at one end of the capillaries and a mass spectrometer helium leak detector at the other end. (These data were converted to equivalent rates of air leakage.) The fluorocarbon liquid leakage rates were measured by connecting one side of each capillary tube to a pressurized reservoir of fluorocarbon liquid, connecting the other end to an empty reservoir and measuring the amount of fluid that had passed through the capillary in 24 h. Five data points were obtained at each leakage rate and at each pressure.



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Before pressurization, the electronic devices to be leak tested are first weighed, usually to the nearest 0.1 mg (3.5×10^{-6} oz) or sorted by weight into a cell that has a span of 0.5 mg (1.8×10^{-5} oz). The devices are then normally subjected to a vacuum with a pressure of 650 Pa absolute (5 torr) for a period of 1 h, before pressurization. The test devices are then immersed in a liquid perfluorocarbon. The bombing time (or time of device exposure to the pressurized liquid) is typically in the range of 2 to 10 h, with bombing pressures of 300 to 600 kPa (45 to 90 $lb_f \cdot in.^{-2}$ gage). After completion of this pressurization period, the test devices are air dried for periods of 2 to 5 min, then reweighed to the same accuracy as that used during preweighing. If an electronic device gains more than 1 mg (3.5×10^{-5} oz) of weight or if its weight shifts by more than 0.5 mg (1.8×10^{-5} oz) cell, it is rejected. Devices that show no significant gain in weight between initial and final weight measurements are usually accepted for this gross leak test based on weight gain.

Radioactive Gas Gross Leak Testing

Krypton-85 tracer gas is used to detect gross leakage in devices with cavities > 0.5 cm^3 (> 0.03 $in.^3$) by pressurization at pressures from 150 to 600 kPa (20 to 90 $lb_f \cdot in.^{-2}$ absolute) for 36 s. The devices are measured for internally trapped tracer gas within 10 min after bombing. Large cavity devices such as relays are easily rejected up to 1 h after bombing.

Devices with cavities of 0.5 to 0.05 cm^3 (0.03 to 0.003 $in.^3$) are normally tested by pressurization at 300 to 600 kPa (45 to 90 $lb_f \cdot in.^{-2}$ gage), for 36 s, with the final evacuation of the pressurization chamber to 2.66 Pa (0.02 torr), instead of 66 Pa (0.5 torr). The devices should be measured for krypton-85 tracer within 10 min.

Very small cavity devices or zero cavity devices are tested for gross leaks through the use of charcoal.[4] The charcoal is added to the device cavity in quantities of 1 to 1.5 mg (3.5×10^{-5} to 5.3×10^{-5} oz) and acts as a gettering agent for the krypton-85 gas. This technique ensures the retention of the krypton-85 for over 30 min, allowing adequate time for testing.

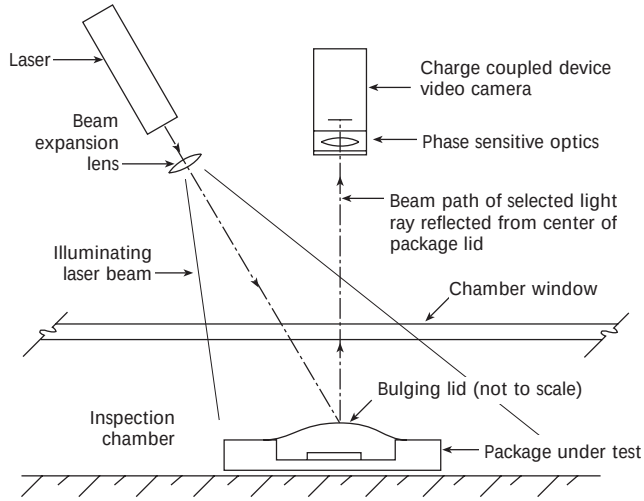
Holographic Leak Testing[5]

Holographic leak testing is a technique developed in the 1980s and limited by package type and leak rate range. Holography can be used to leak test sealed devices such as hybrid electronic packages, generally bodies with metal lids, by using laser light reflected off the lid of the package being evacuated. The technique measures the leakage rate of all packages on a tray simultaneously. Devices are placed in a chamber and the chamber is either pressurized or evacuated. The resultant change in pressure on the lid of the package will cause the lid of a package to undergo concave or convex deformation. Multiple hybrid electronic packages mounted on circuit boards can be leak tested by this method, which uses laser interferometry to measure microscopic deformations in the lid of the package (Fig. 4a).

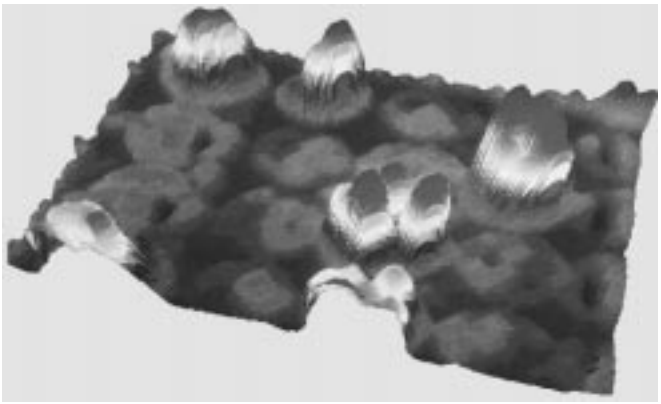
The package lid theoretically will not deform if there is a severe, gross leak in the seal, as the internal and external pressures will remain the same. For fine leaks, when a package lid is deformed due

FIGURE 4. Holographic leak testing system operation: (a) schematic of application to hybrid electronic packages with metal lids; (b) holograhic image of two pharmaceutical blister packages with ten cavities

(a)



(b)



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to external pressure change, the lid will slowly recover from deformation at a rate depending on the leakage rate. The rate of recovery of the lid deformation on a specific package design can be determined to reflect a specific leakage rate for that package. The larger the leakage rate, the more rapid the recovery. Holography is able to detect a single wavelength of laser light change in the surface deformation.

Gross leakage is quickly detected whereas fine leakage requires several minutes to detect a recovery (or change) in the lid deformation of a few micrometer (about 10^{-4} in.). Ideally, lid materials should be consistent in thickness and material. Application of the method to other device configurations can be more complicated. Very large hybrids may require evacuation of the chamber, if they have internal lid supports, whereas smaller lids and cylindrical cans are more difficult to read. The practical leak rate range of application appears to be from gross leaks to the 10^{-7} Pa·m^3·s^{-1} (10^{-6} std cm^3·s^{-1}) leakage rate range.

The technique may be adapted for rapid, full range leak testing to 10^{-8} Pa·m^3·s^{-1} (10^{-7} std cm^3·s^{-1}) in some production applications. Figure 4b shows the method's application to leak testing of pharmaceutical blister packages. The inspected package has laser milled holes of 15, 35 and 60 µm (6×10^{-4}, 1.4×10^{-3} and 2.4×10^{-3} in.) diameter. Figure 4b shows cavities leaking at rates between 5×10^{-5} to 1×10^{-6} Pa·m^3·s^{-1} (5×10^{-4} to 1×10^{-5} std cm^3·s^{-1}) in 0.1 s.

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PART 4. Fine Leak Testing of Hermetically Sealed Devices with Krypton-85 Gas

Major Leak Testing Techniques Used for Fine Leak Testing of Hermetic Devices

Basically, there are only two fine leak test techniques that are widely used in industry for leak testing of hermetically sealed devices: (1) the radioisotope tracer gas leak testing technique and (2) the helium tracer gas leak testing technique. Wide variance has existed in both the test conditions and the resultant leak test sensitivity limits, depending on such factors as the pressure differentials or bombing pressures, the low pressure conditions into which leakage occurs and the sensitivities of the leak testing instruments (used in vacuum or in air at atmospheric or other pressures).

In the past, many procedures and specifications used for fine leak testing specified a maximum leakage rate when measured at a differential pressure of 100 kPa (1 atm). The latter was not a realistic requirement for the helium test, because if the device were a *leaker,* the pressure differential of 100 kPa (1 atm) would not be maintained as time progressed after pressurization and during the leak test exposure. More recently, efforts have been initiated to develop more realistic and consistent standards for helium and radioisotope tracer gas fine leak tests of hermetically sealed devices.

Krypton-85 Fine Leak Test Methods for Hermetically Sealed Devices

In the radioisotope gas leak test technique, the tracer gas is usually the radioactive (but chemically inert) krypton-85 gas, typically diluted in gaseous nitrogen. The sealed hermetic devices are bombed or subjected to the radioactive gas and its carrier gas under pressure, nominally 300 to 800 kPa absolute (45 to 120 $lb_f \cdot in.^{-2}$), for specified periods of time. The maximum external pressure the device package can withstand must be determined in advance so that as high a bomb pressure as possible can be used to minimize the bombing time. In most cases, the bomb pressure and bombing time are specified in advance for specific types of components and package.

During the radioisotope leak testing, the packages are placed within the bombing chamber. Before introduction of the krypton-85 mixture, the chamber is evacuated to remove the air and prevent dilution of the krypton gas mixture. The bombing chamber is then backfilled with the radioactive tracer gas mixture to a specified pressure and the devices are exposed to this pressurized tracer gas mixture for a specified period of time.

The bombing time is chosen to permit a sufficient quantity of krypton-85 tracer gas to enter a leaking device to ensure detectability. The quantity of tracer required is about 10^{11} molecules of krypton-85. That concentration is sufficient to produce an easily measured radioactive emission through the walls of the device. Typically, in small cavity packages, the krypton-85 concentration represents about 1 part per hundred million parts of air inside the part. The radioactive gas pressurization mixture is then pumped out of the bombing chamber into a permanent tracer gas storage system.

Following completion of bombing and purging operations, the bombing chamber is opened and each package device is placed (either individually or in batches) within the well of a scintillation counting radiation detector system. They are easily tested by carrying the devices on a conveyor belt through a tunnel type scintillation crystal detector at rates of one per second. The radiation emitted from external surface adsorbed gas is typically beta radiation (electrons) that can be stopped by thin layers of absorbers. The more energetic gamma radiation produced by disintegrations of krypton-85 gas molecules contained within the packages can then be detected and counted with suitable scintillation counters.

The leakage rate is then calculated from the counting rate caused by emissions proportional to the amount of krypton-85 that has entered the device during the bombing step and still remains within the device package at the time of the scintillation counter measurement. This rate depends on the amount of radioactive gas introduced during bombing and on the loss of that gas through the leak following the bombing

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operation. Thus, measurements are being made during transient conditions of tracer gas pressure within the test devices. The transient conditions have little effect on the krypton-85 test, because very small partial pressures are needed for substantial detectability.

Radioactive Tracer Gas for Combination of Gross Testing and Fine Testing

One major advantage of the radioactive tracer gas leak test is that large quantities of hermetically sealed electronic devices can be bombed simultaneously and rate meter tests of gamma-ray emission can be made on the individual components or batches of components by automatic means. If time permits, essentially all of the radioactive tracer gas can escape before the scintillation counter radiation measurement, thus gross leakers could escape detection.

In devices of 0.05 cm^3 (0.003 $in.^3$) and larger cavities, the technique is very reliable for detection of gross or fine leakers. The gross and fine leak test are easily combined into a gross/fine combination test with a single pressurization. With typical radioactive gas machine concentrations, a gross/fine combination pressurization at 520 kPa (75 $lb_f \cdot in.^{-2}$) requires 12 to 15 min, to detect leaks from 10^{-2} to 10^{-9} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-1} to 10^{-8} std $cm^3 \cdot s^{-1}$). The measurement time or readout time should always be controlled to a maximum of 10 min for devices with cavities less than 0.5 cm^3 (0.03 $in.^3$) to ensure detection of gross leakers.

The radioisotope technique for leak testing of hermetically sealed packages is basically a more direct leakage test technique than helium leak testing because the radioactive gas is measured when it is inside the package. (In helium leak testing, the rate of escape of the tracer gas through the leak is being measured.) Smaller concentrations of radioactive tracer gas are sufficient for sensitive leak testing, so that the minimum detectable leakage rate is lower than that for the helium mass spectrometer test, typically 10^{-12} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-11} std $cm^3 \cdot s^{-1}$). Because detection is done with the devices at atmospheric pressure, the sample handling rates can be much greater than with helium testing, where the package must be placed within an evacuated closed environment before leakage measurements are made. However, it is considered proper practice to complete the readout in a ten minute time period.

Detection of Zero Cavity Device Leakage by Using Charcoal Gettering of Radioactive Tracer Gas

A device without any internal cavity has always presented a difficult and often expensive task to verify hermeticity, usually through extensive environmental testing. Both gross and fine leakers are easily detected in zero cavity devices through the tracer quantities of coconut shell charcoal placed on the inside of the device. The charcoal adsorbs large quantities of radioactive krypton-85 gas and easily retains the krypton-85 for sufficient time to ensure detection after bombing. The explosive industry uses small quantities of coconut shell charcoal, typically 1.5 mg (5.3×10^{-5} oz), in such devices as air bag squibs. The mixtures are compressed into cans at pressures as high as 125 MPa (1.8×10^4 $lb_f \cdot in.^{-2}$). A header is then compressed or soldered in place. The powder and charcoal mixture can be bombed in radioactive gas (in the open can configuration) and easily detected as a reject for 20 to 30 min after pressurization.

Electronic devices such as integrated circuits with less than 0.5 cm^3 (0.03 $in.^3$) cavities are tested for gross/fine sensitivities by adding coconut shell charcoal to the cavity. The charcoal is extremely light weight and bonds to the die bond material when fired. The radioactive gas gettering ensures detection of circuit packages with large gross leaks.

Leak Testing of Plastic Devices with Radioactive Tracer Gas

Low gas solubility plastic hermetic devices, both with and without cavities are tested for gross and fine leaks by using krypton-85 tracer gas. Krypton-85 gas emits both beta particles and gamma-rays. The beta emission normally is only detected from krypton-85 gas that is absorbed onto the surface of a device. The monitoring of surface desorption of good epoxies takes 3 to 8 min with krypton-85 pressurization for 1×10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-7} std $cm^3 \cdot s^{-1}$) sensitivity tests. Following the desorption of the surface gas, the internally trapped krypton gas is measured to detect a leaker.

Many types of relays use an epoxy coating over the glass feed-throughs to seal broken glass-to-metal seals. Such devices are easily tested with radioactive krypton gas by monitoring the beta radiation as a wait time, after which the

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internally trapped krypton-85 is detected to show a leaking device.

Zero cavity or extremely small cavity plastic devices such as plastic air bag squibs can be tested for gross and fine leaks through coconut shell charcoal added internally in quantities of 1.5 mg (5.3×10^{-5} oz). That allows wait time for the surface desorption to occur and then the device is measured for krypton radioactive tracer gas that was gettered and held by the charcoal.

Special Considerations in Radioactive Tracer Gas Leak Tests of Sealed Devices

The following precautions should be considered when selecting or using the radioactive tracer gas leak test procedure for tests of sealed devices.

1. As with helium leak testing, the radioactive tracer gas test should be performed before liquid immersion of test devices to prevent fluids from clogging the leak holes or reducing the measured leak rate.
2. Parts that are made of *sorptive* materials or that use adhesive labels are often rejected as *false leakers.* This problem is easily avoided with radioactive krypton tracer gas, which emits beta particles as well as gamma-rays. The beta radiation is only detected if the krypton-85 gas is on the outside of the device. In such cases, a short waiting period will allow surface gas to dissipate and after verification with the beta detector that the surface is clean, the device is reliably measured for internally trapped krypton with a scintillation detector.
3. In cases where a retest is required on sealed devices that have previously been subjected to leak testing with krypton-85 tracer gas, measurements should first be made for indications of gamma-ray emissions from residual radioactive tracer gas trapped on or within the device package. If there is any indication of krypton-85, the radioactivity reading must be recorded as a background radiation level for that test part. Then a standard test is applied. The background reading is subtracted from the final reading to establish the new leak rate.
4. Although the radioactive tracer gas leak testing technique may be applied on either a sampling basis or on 100 percent of all devices in a lot, the quantity of small cavity devices pressurized at one time during the bombing step should be limited to the number that can be checked for radioactivity at the counting station within 30 min following the required waiting time. However, it is always considered good practice to test batches of very small cavity parts within 10 min. At one part per second, that typically tests 600 devices per batch. Very small devices are tested in groups of three to five devices per analysis with the scintillation crystal, that increases the throughput to 180 to 300 devices per minute.
5. Sealed devices rejected during radioactive tracer gas leak testing obviously contain radioactive krypton-85. Because it is logical to control all radioactive materials, rejected or leaking devices should be destroyed, deactivated in a vacuum chamber or otherwise controlled. Most rejected devices contain such small quantities of radioactive tracer gas, they emit no measurable radioactive dosage to humans. Gross leakers will release that gas quickly, whereas fine leakers will generally require many hours to vent the gas. Puncturing or smashing those devices out of doors or in a well ventilated area will create no hazards to humans.
6. The possession of radioactive tracer gas leak detection systems requires a radioactive materials license from the regulatory body for that specific area. Each regulatory agency has a set of rules and regulations governing the use of such isotopes. The manufacturer of radioactive tracer gas equipment can provide information on the radiation technology and safe operation of such equipment.

Equipment for Radioactive Gas Leak Testing of Sealed Electronic Devices

In leak testing with radioactive tracer gases, special equipment is required for the storage, transfer and handling of the radioactive tracer gases, including storage vessels, pumps both for pressure and vacuum, valves and pressure measuring instrumentation. A typical krypton-85 gas handling system is depicted in Fig. 5. It contains a pressurization tank where the devices are bombed; a storage tank where the krypton-nitrogen gas mixture is stored; two vacuum pumps, one to evacuate the air from the bombing tank and one to return the krypton-nitrogen mixture to the compressor after bombing; and a compressor to transfer the tracer gas mixture from storage to the bombing chamber and then to compress the gas

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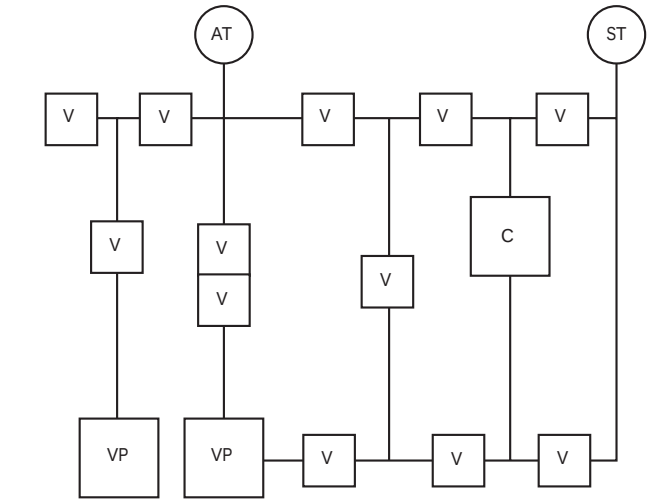
back into storage. These functional units are coupled with solenoid valves, pressure and vacuum sensors. The operation of such gas handling equipment is controlled by a logic system that is integrated with a series of both functional and safety sensors.

Measurement equipment is used to detect the radiation from devices that were nonhermetic and allowed radioactive gas to enter the device. Thallium activated scintillation crystals, housed in a 50 mm (2 in.) lead shield, are used to analyze the radiation from devices. The instrumentation is usually a rate meter that is measuring the signal from a photomultiplier tube coupled to the scintillation crystal.

FIGURE 5. Example of krypton-85 gas handling system.



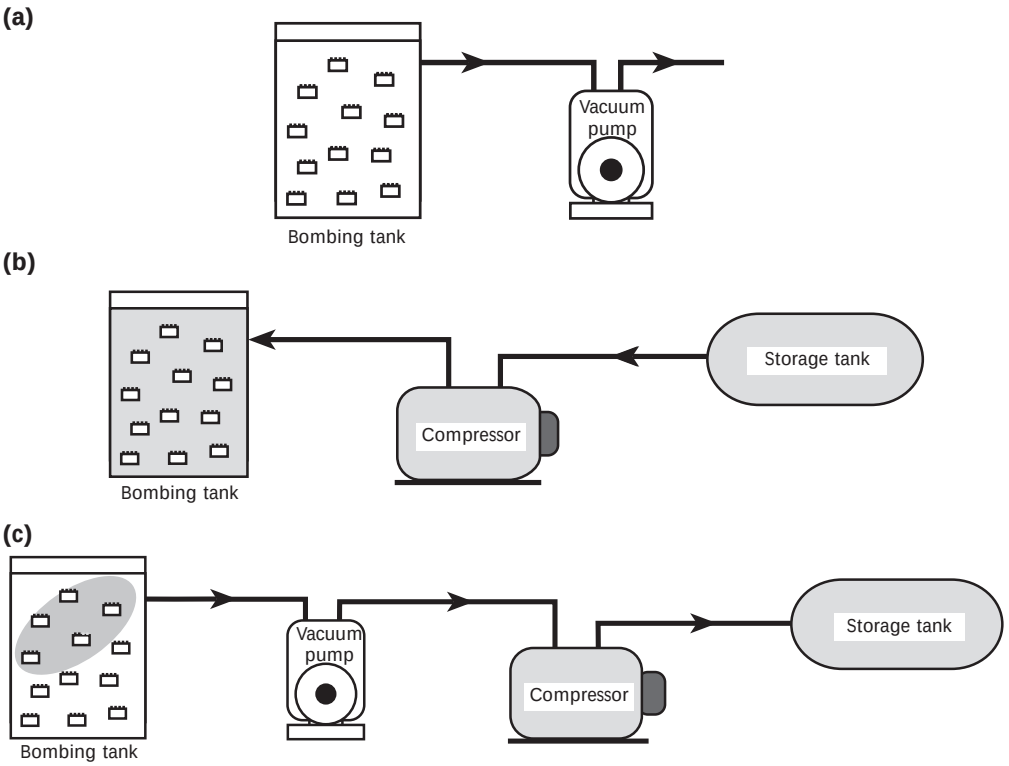
Legend
AT = activation tank
C = compressor
ST = storage tank
V = valve
VP = vacuum pump

Typical Radioactive Gas Pressure Bombing Cycle

The pressurization system is depicted in Fig. 6, showing only the major components of the system.

1. A test cycle begins with loading a batch of devices into the bombing tank.
2. The tank is closed and evacuated to a pressure of 65 Pa (0.5 torr), to prevent dilution of the radioactive tracer gas (Fig. 6a).
3. The tracer gas mixture is transferred from the storage vessel to the bombing tank. A compressor is used, if required, to compress the tracer gas to the preset bombing pressure (Fig. 6b).

FIGURE 6. Operation of krypton-85 gas system for leak testing: (a) evacuation; (b) bombing; (c) return of tracer gas to storage.



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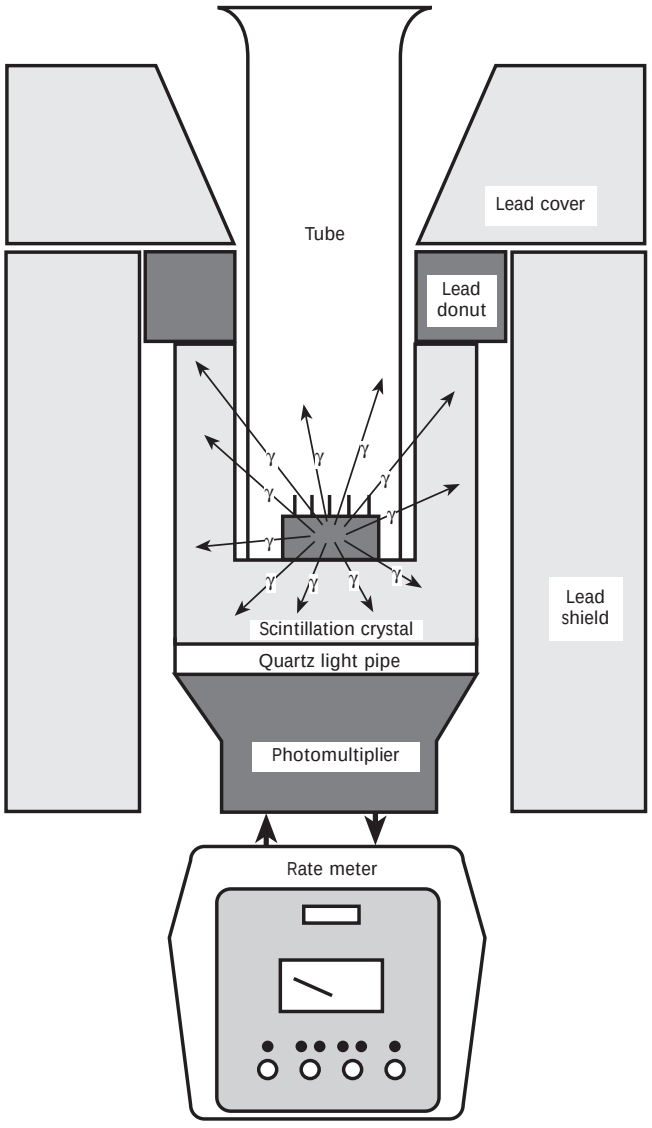
4. The devices then soak at the bombing pressure for the time necessary to achieve the desired sensitivity.
5. The tracer gas mixture is returned to the storage tank with a closed loop vacuum pump in tandem with a compressor (Fig. 6c).

The vacuum level achieved at the end of the cycle is either 65 Pa (0.5 torr) for a fine leak test or 260 Pa (2.0 torr) for a gross leak test. The higher venting pressure leaves a greater amount of gas inside of the gross leakers and allows them to be reliably detected. The bombing chamber is then vented to atmosphere and opened; the devices are removed and taken to the detector station and measured for any radiation from internally trapped krypton-85 gas that entered a leaker.

FIGURE 7. Radiation detection system.



Krypton-85 Radiation Counting Station

The counting station is where the krypton-85 tracer gas entrapped within a reject device is detected as a go/no-go measurement for pass/fail only; or measured quantitatively for an exact leak rate. The measurement of leakers is achieved by using a thallium activated sodium iodide crystal attached to a photomultiplier tube. Gamma radiation pulses from the krypton-85 trapped within a device produce quanta of light through ionization of the crystal (Fig. 7). The quanta of light are converted to current that in turn is read with a rate meter.

Typically a reject device will contain enough krypton-85 gas to produce a minimum of 1000 counts per minute that in turn is about 5×10^{11} molecules of krypton-85 gas within the part. (That usually represents a krypton-85 partial pressure of parts per million or parts per billion). The crystals are mounted within a 50 mm (2 in.) lead shield to minimize the atmospheric radiation and keep the background reading as low as possible. The time required to achieve a go/no-go signal in a scintillation crystal is 20 to 40 ms. Each device configuration is first measured with a *counting efficiency* in the crystal. The efficiency is identified as a *K factor* or the measurement of the number of counts per minute per microcurie of krypton-85 gas entrained within that device.

Sensitivity of Leak Testing with Radioactive Krypton-85

The sensitivity of leak testing with krypton-85 tracer gas covers the range from a visible hole, to as low as 10^{-14} Pa·m^3·s^{-1} (10^{-13} std cm^3·s^{-1}). Typically production or mass testing applications cover from a visible hole to 10^{-9} Pa·m^3·s^{-1} (10^{-8} std cm^3·s^{-1}). High sensitivities are achieved in some production applications to 10^{-12} Pa·m^3·s^{-1} (10^{-11} std cm^3·s^{-1}), by maintaining a high krypton-85 concentration, bomb times of a few hours and accurate readout.

Advantages of Leak Testing with Radioactive Tracer Gas

With krypton-85 tracer gas leak tests, it is necessary only to bomb or inject the pressurized tracer gas into the devices under test to measure leakage. By

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contrast, other tracer gas leak testing techniques require that the tracer gas first be forced into the device and secondly drawn out of the device to permit measurement of the leakage rate. The technique of bombing and not drawing the radioactive tracer back out of the device reduces the probability of missing leaks because it reduces the chance that the leak path will become blocked. Still another advantage of this technique of leak testing is that the rate of testing of sealed devices is many times faster than with other tracer gas techniques.

The beta (electron) emission from the krypton-85 can be used to determine whether tracer gas is adsorbed on the external surface of the test object or is contained wholly within the interior of the sealed test object. The radioactive emission from krypton-85 is more than 99 percent beta (electron) emission, which does not have sufficient particle energy to penetrate through most enclosure materials. A beta measurement can be made to avoid rejecting those devices with adsorbed external surface tracer gas only. If beta emission is minimal and gamma-ray emissions are observed, they indicate the presence of radioactive krypton-85 within the enclosure and thus indicate true *leakers*. The ratio of beta emissions to gamma emissions for externally adsorbed krypton-85 is typically of the order of 200 to 1. This ratio drops to the order of 1 to 1 where external surface adsorbed krypton-85 is minimal and radiation comes from inside the test object.

Beta Particle Counting to Detect Parts with Surface Contamination

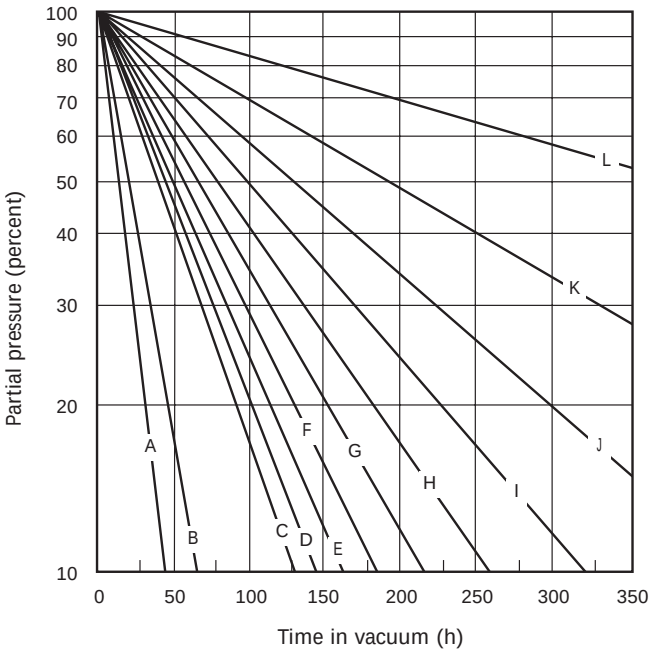
Routine checking of rejected parts with a thin window Geiger-Müller tube sometimes reveals surface contamination of the test parts. Comparison of rejected parts with acceptable parts of the same surface composition can be used to determine the significance, if any, of the surface contamination. Those parts with significant contamination can often be decontaminated with a brief exposure to heat. Frequently, such a heating cycle is routinely incorporated into the testing procedure of certain parts having organic coatings. It is quite reliable to test painted devices such as relays, by including an empty sample can, painted with the lot-of-relays but without any header or internal components. The can is measured after bombing in the radioactive tracer gas. When the radiation is gone from the can, the rest of the lot may be measured for internally trapped krypton-85.

Vacuum Decay Confirmation of Leak Rates

The radioactive tracer gas technique using krypton-85 gas provides perhaps the most absolute technique of confirming a leak rate. When a leaking device of known internal volume is bombed in krypton-85 gas, the device will collect a measurable quantity of krypton-85 gas. Quantitative measurement of the gamma reading provides an accurate measure of the number of krypton-85 molecules trapped in the device.

Because the partial pressure of krypton-85 is being measured, the exact leak rate may be verified by measuring the initial reading of krypton-85, placing the device in vacuum for several hours, reading the device and calculating the percentage of gas loss by using the equation $P_t = P_0\, e^{-kt}$, where k is the measured or calculated leak conductance ($cm^3 \cdot h^{-1}$) divided by the internal volume (cubic centimeter). A typical plot of vacuum decay curves is shown in Fig. 8.

FIGURE 8. Vacuum decay for 20 mm^3 (1.2×10^{-3} $in.^3$) package of krypton-85 for leaks A to L with range of conductances ($cm^3 \cdot h^{-1}$). One $cm^3 \cdot h^{-1} = 2.8 \times 10^{-4}$ $cm^3 \cdot s^{-1} = 0.8$ $oz \cdot day^{-1}$.



Legend

A = 3×10^{-7}	G = 6×10^{-8}
B = 2×10^{-7}	H = 5×10^{-8}
C = 1×10^{-7}	I = 4×10^{-8}
D = 9×10^{-8}	J = 3×10^{-8}
E = 8×10^{-8}	K = 2×10^{-8}
F = 7×10^{-8}	L = 1×10^{-8}

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Accuracy of Radiation Counting Techniques

With fixed conditions of bombing pressure, exposure time and concentration of radioactive krypton tracer, accuracy is limited by the efficiency of radiation detection, assuming no external surface adsorption of the gas. Small parts can be counted in a well or tunnel shaped thallium activated sodium iodide crystal scintillation counter. A state-of-the-art scintillation crystal counting station operates with a background count of 1000 to 1500 counts per minute from atmospheric radiation.

Detection of a device that has been bombed in radioactive krypton gas will collect a quantity of krypton-85 that reads 1000 to 1500 counts per minute above background. (Note that it is rare for leakers to be found that are marginal). Each increment of 1000 to 1500 counts per minute usually represent a leakage rate of one tenth of an order of magnitude

Such quantities of krypton-85 are quite accurately measured with state of the art equipment. A device that leaks in the fine leak range, $<5 \times 10^{-7}$ Pa·m^3·s^{-1} ($<5 \times 10^{-6}$ atm cm^3·s^{-1}), can be measured to accuracies of 0.1 of an order of magnitude, plus the normal cumulative errors associated with the process: i.e. the gas concentration, the pressure measurement, the krypton-85 reference sources etc. Thus a device may be measured for leakage with an absolute accuracy of about 0.4 of one order of magnitude.

Equation Used for Radioactive Gas Leak Testing

Equation 1 is used for the calculation of the testing parameters in performing a radioactive tracer gas leak test:

$$\textbf{(1)} \quad Q_1 = \frac{R}{SKP_d T}$$

where Q_1 is the leak rate sensitivity desired (Pa·m^3·s^{-1}); R is the reject point or rate meter reading above background, at which a part is rejected (this net reading should be at least equal to the normal ambient background reading of the counting equipment, typically 1200 to 1500 counts per minute net); $P_d = P_e^2 - P_i^2$ (where P_e is the external absolute pressure and P_i is the internal absolute pressure, in pascal); S is the specific activity or concentration of the radioactive gas mixture (this concentration measurement is normally measured at least monthly) in Bq·Pa^{-1}·m^{-3} (or µCi·atm cm^{-3}); t is pressurization or bombing time (second); and K is the counting efficiency of the device being tested (that is, the efficiency of measuring one microcurie of krypton-85 gas within the geometry of the exact part being tested, that will provide a correction for the thickness of the walls of the device as well as the positioning of the device relative to the scintillation crystal detector used to measure the radioactive tracer gas trapped in the device).

Functional Check on Krypton-85 Counting Station

The counting station used, for the detection of reject devices after bombing, is checked for function at least once per day. That check consists of placing a krypton-85 glass reference vial into the scintillation detector and ensuring that the amount of krypton-85 within that vial is being detected by the crystal rate meter system. A tolerance of ±10 percent is acceptable for such a check. In a well type crystal detector, the efficiency of detection is about 14 000 counts per minute per microcurie of krypton-85.

A becquerel (Bq) is one disintegration per second whereas a curie (Ci) is a quantity of disintegrations per second: 37 GBq = 1 Ci.

Counting Station Calibration

The counting stations are normally calibrated monthly by using a traceable krypton-85 glass reference source corrected for half life decay. Because the beta detectors are classified as qualitative instruments, they are normally checked for function only by using a cesium-137 source (or other beta source) and verified to be functional, because in the testing of devices they are normally used as a go/no-go indicator of surface gas contamination. The scintillation crystals, however, require a determination of accurate detectability for krypton-85 and assurance of stability. The rate meter used to measure the signal from the scintillation crystal can be calibrated electronically by using a pulse generator to verify signal detection accuracy and linearity, following manufacturer's procedures.

The functional accuracy of the rate meter is best determined as a crystal rate meter system. The operational stability of the crystal rate meter system must first be established by placing a krypton-85 reference vial into the crystals normal geometric reading position, as a device would be placed into a *well type* crystal for measurement.

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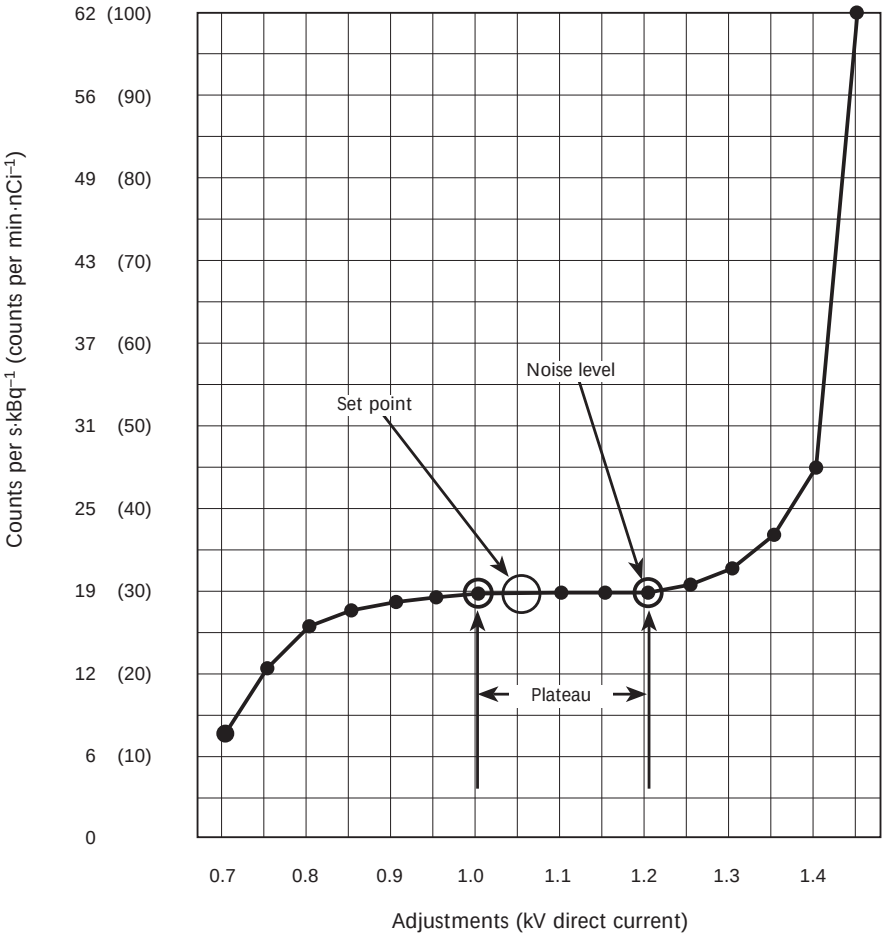
Most crystals operate at a high voltage of 1 to 1.5 kV. To establish the point of stability, the voltage is reduced to 0 and then increased in increments of about 100 V, with the reading of the reference source being recorded at each 100 V. A properly operating crystal will demonstrate a stable range over perhaps several hundred volts. That point is considered the *plateau* of that crystal. Such a plateau can be seen in Fig. 9. The high voltage is then set to a point that is usually at the midpoint of the plateau. In state-of-the-art counting system equipment, the system should remain stable at that voltage setting for years, without any significant changes to the efficiency of the system.

The resultant reading of the standard at that voltage, minus the background reading taken without the reference source in the well, will provide the actual efficiency of the crystal to measure krypton-85 gas contained in a reject device in that position within the crystal. A good quality crystal will provide reading efficiencies of 13 000 to 14 400 counts per minute per microcurie.

In large volume manufacturing facilities, the devices in test are read automatically by using a conveyor belt that carries the device through a tunnel in a scintillation crystal. That allows the radiation from a device to be read circumferentially as the device passes through the detector. Because only 20 to 40 ms are required to detect krypton-85 within a device, the belt may carry devices at speeds of 175 to 225 mm (7 to 9 in.·s^{-1}). A calibration of such detection systems requires the reference standard to be measured with the tunnel detection zone in a static state, the plateau to be established, the efficiency to be determined (as with the well crystal case above) and then the measurement to be read with the conveyor belt operating at full speed. Obviously, the dynamic efficiency of the system will be less than static. Tunnel systems achieve dynamic efficiencies from 8000 to 11 000 counts per minute per microcurie, for most devices.

FIGURE 9. Typical operating plateau for scintillation counter instrument. Detector sensitivity equals counts per second per nanobecquerel (counts per minute per microcurie).



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Determination of Counting Efficiency for Hermetic Devices

The efficiency of measurement of a quantity of krypton-85 gas within a hermetic device requires a representative sample of the device to be tested, to be filled with a known quantity of krypton-85 gas and then to be placed within the scintillation crystal in exactly the same position as it will be measured when tested. The sample used for the actual efficiency measurement must be of the same geometry, material and internal void as the actual parts to be tested with the radioactive gas.

Several techniques have been used to establish the counting efficiency of devices to be tested. A rather complicated technique involves the introduction of a very accurately known quantity of krypton-85 gas, of accurately known concentration, through a *tubulation* that is then sealed off. The device is then placed within the crystal and measured for detection efficiency, referred to as *K factor*. That is the measurement of the number of counts per minute per microcurie of krypton-85 gas within that device.

A second technique of determination involves the introduction of a sample of material that contains an accurately known quantity of krypton-85 gas. The device is then assembled into its final configuration and measured in the crystal. Both of these techniques are best performed by the radioactive gas equipment manufacturer. The equipment manufacturer can usually provide the user with the *K* factor for most devices that are manufactured, based on prior experiments, and *geometric approximation* of the device within a crystal. One rule is that the nuclear physics of krypton-85 gas only provides sufficient emission to achieve a maximum reading of 1.6×10^4 counts per minute per microcurie of krypton-85. Any indications of greater efficiencies are ambiguous and indicate errors in the technique or equipment used to determine the *K* factor.

Determining Specific Activity of Radioactive Krypton-85 Tracer Gas Mixture

The concentration of the krypton-85 radioactive gas mixture used for pressurization of the hermetic devices, must be accurately known to calculate the proper bomb time and pressure to be used. The krypton-85 gas is normally mixed in nitrogen (or air) at a total pressure of 0.7 to 1.0 MPa (7 to 10 atm). The concentration required to perform a proper test, is not critical. The concentration for testing plastic devices, and painted devices is usually maintained quite low, < $MBq \cdot Pa^{-1} \cdot m^{-3}$ (< 0.1 $mCi \cdot std\ cm^{-3}$). The concentration for testing nonabsorbent devices, high reliability devices etc., are usually > 1 $MBq \cdot Pa^{-1} \cdot m^{-3}$ (> 0.1 $mCi \cdot std\ cm^{-3}$).

The specific activity measurement is normally performed once each month. Two methods are commonly used with radioactive tracer gas systems. In one technique the gas mixture is sampled by withdrawing a sample of the gas into a glass vial, at a pressure of 250 Pa (2 torr). The volume of the glass vial must be precisely known and is usually about 3 mL = 3 cm^3 (0.18 $in.^{-3}$). The sample of gas is measured in a scintillation crystal and then compared to the measurement of a similar geometry glass reference vial of known quantity of krypton-85. The concentration of krypton-85 in $\mu Ci \cdot std\ cm^{-3}$ is calculated as the *specific activity* of the radioactive tracer gas system. A number of errors are inherently associated with this technique: the volume of the vial, the pressure of the sample, the accuracy of the counting station, the accuracy of the reference vial and the human errors introduced by the technician.

The second technique is a closed loop system built into the radioactive tracer gas handling system, which automatically samples the gas in the system every cycle that the machine operates. The concentration is electronically calculated and displayed on the operating control panel of the system. It involves the automatic sampling of the gas into a large, fixed volume chamber, at a pressure above atmosphere, and the continuous measurement of that sample with an integral radiation detector. The error associated with this technique is usually <0.001 of the hand sampling technique and is automatically performed every cycle of operation.

Calculating Leak Rates of Failed Devices

Following the bombing of the devices and sorting of rejects, the actual quantitative leak rates may be calculated for devices. Leak rate values greater than 5×10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-7} std $cm^3 \cdot s^{-1}$), are considered to be viscous flow of the gases and the gases of concern in leak detection systems are considered to have similar flow rates through the same leak path. Smaller leak rates are considered to be in the molecular flow regime. The leak rates are calculated based on the net radiation

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reading obtained on the device. Normally devices are rejected on a go/no-go basis. However, if the quantitative leakage value is desired on a device, the device should be read with the rate meter in the *long, time constant* position. That is, the actual reading of the device is averaged over a period from 1 to 10 s to ensure an accurate reading.

The ambient background of the equipment should also be read with equal accuracy and subtracted from the gross reading. The leak rate of the device is calculated by dividing the net reading by the reject value used in Eq. 1, times the test sensitivity Q_1. The leak rate value obtained will be for krypton-85 gas. If it is desired to convert a leak rate less than 5×10^{-8} Pa·m^3·s^{-1} (5×10^{-7} std cm^3·s^{-1}) to an equivalent leak rate for another gas such as air or helium, the krypton-85 leak rate would be converted based on the theory that the flow rate varies inversely with the ratio of the square roots of the molecular weights of the two gases of concern. Thus, to convert the krypton-85 leak rate to air, multiply by 1.712; to convert it to helium, multiply by 4.7.

It should be remembered that the actual leak rate of the device may be confirmed by applying techniques for vacuum decay confirmation of leak rates, described earlier.

Krypton-85 Reference Source for Calibration

The krypton-85 reference calibration is conducted by using a reference standard in the form of a glass vial of about 3.5 cm^3 (0.2 in.^{3}) volume, with a quantity of krypton-85 gas sealed inside and traceable to the United States National Institute of Standards and Technology. The half life of krypton-85 is 10.76 years, which allows for accurate correction by using standard half life equations or corrections from decay tables. The normal accuracy of such standards is ± 10 percent. The reference vials should be corrected for half life decay at least every six months by using the decay equation:

$$\textbf{(2)} \qquad A_t = A_0\, e^{-0.693\,\frac{t}{T_h}}$$

where A_t = amount of krypton-85 remaining at time t; A_0 = original amount at time 0; t = time passed from time 0 to time t (in unit of year); $T_{1/2}$ = half life of krypton-85 = 10.76 yr. This half life decay calculation has been converted to a *decay chart* as shown in Table 5. From that chart the quantity of krypton-85 within the vial can be calculated easily and accurately.

Pressurization System Calibration

The pressurization systems require maintenance and calibration at specific intervals to satisfy the metrology needs and to maintain a gas handling system required to reliably handle radioactive gas. The maintenance cycle includes replacement of valve seals, vacuum pump oil, compressor oil and normal wear items. The calibration steps cover the vacuum gages, pressure transducers and mechanical gages that control the actual test parameter accuracies. Most vacuum and pressure gages are reliable for periods of one year. The vacuum gages are calibrated at the critical stepping points of 0.066, 0.266 and 100 kPa (0.5, 2.0 and 760 torr). The pressure transducers are calibrated against a traceable mechanical gage for the normal operating range of the pressurization system.

TABLE 5. Decay of krypton-85 with half life of 10.76 yr.

Years	Months											
	0	1	2	3	4	5	6	7	8	9	10	11
0	1.000	0.995	0.989	0.984	0.979	0.974	0.968	0.963	0.958	0.953	0.948	0.943
1	0.938	0.933	0.928	0.923	0.918	0.913	0.908	0.903	0.898	0.893	0.889	0.884
2	0.879	0.874	0.870	0.865	0.860	0.856	0.851	0.847	0.842	0.838	0.833	0.829
3	0.824	0.820	0.816	0.811	0.807	0.802	0.798	0.794	0.790	0.785	0.781	0.777
4	0.773	0.769	0.765	0.761	0.756	0.752	0.748	0.744	0.740	0.736	0.733	0.729
5	0.725	0.721	0.717	0.713	0.709	0.705	0.702	0.698	0.694	0.691	0.687	0.683
6	0.679	0.676	0.672	0.669	0.665	0.661	0.658	0.654	0.651	0.647	0.644	0.641
7	0.637	0.634	0.630	0.627	0.624	0.620	0.617	0.614	0.610	0.607	0.604	0.601
8	0.597	0.594	0.591	0.588	0.585	0.582	0.578	0.575	0.572	0.569	0.566	0.563
9	0.560	0.557	0.554	0.551	0.548	0.545	0.542	0.539	0.537	0.534	0.531	0.528
10	0.525	0.522	0.520	0.517	0.514	0.511	0.509	0.506	0.503	0.500	0.498	0.495
11	0.492	0.490	0.487	0.485	0.482	0.479	0.477	0.474	0.472	0.469	0.467	0.464

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PART 5. Fine Leak Testing of Hermetically Sealed Devices with Helium Gas

Helium Fine Leak Test Methods for Hermetically Sealed Electronic Devices

The helium tracer gas leak test technique is the second of two widely used techniques for fine leak testing of hermetically sealed electronic devices. The helium mass spectrometer fine leak test is relatively simple and does not require compliance with regulations governing nuclear byproducts that produce ionizing radiations (such as krypton-85 tracer gas).

Helium is the lightest inert gas. Its molecules will penetrate through even the smallest leaks but will not clog the leak. Helium is not hazardous and is present in the earth's atmosphere only as a tracer element present in a concentration of about 5 $\mu L \cdot L^{-1}$ of air (about 1 part in 201000 in air). In normal production leak testing, the mass spectrometer leak test equipment provides capabilities for detecting leakage rates in the range from about 10^{-6} to 10^{-10} $Pa \cdot m^3 \cdot s^{-1}$ (10^{-5} to 10^{-9} std $cm^3 \cdot s^{-1}$).

With gross leakers it is possible that so much tracer gas may escape before mass spectrometer leak tests of the devices that little or no tracer gas is left to escape during the actual leak test period. Such loss of contained tracer gas makes leak detection much less reliable.

Principles of Helium Fine Leak Test Operation

In leak tests, the helium tracer gas is introduced into the devices under test either (1) initially during device manufacture just before or during the hermetic sealing process or (2) by back pressure (bombing) techniques applied any time after the hermetic sealing of the device has been completed. The tracer gas may vary from commercially pure 100 percent helium to a mixture of 10 percent helium with 90 percent nitrogen (if pressuring up is to be used and the cost of helium is a deterrent to its use). During tests, the sealed devices are placed in a test chamber that is evacuated and connected to a properly calibrated helium mass spectrometer leak. With proper calibration, the helium mass spectrometer leak detector can detect leakage rates in the range from about 10^{-7} to about 10^{-11} $Pa \cdot m^3 \cdot s^{-1}$ (about 10^{-6} to about 10^{-10} std $\cdot cm^3 \cdot s^{-1}$). The free volume of the evacuated test chamber containing the devices under test should be held to a practical minimum level. Any empty space within the chamber has an adverse effect on the limits of leakage sensitivity attainable with the helium mass spectrometer during leak tests of sealed devices with small internal helium filled volumes.

Operational Limitations of Helium Back Pressure Leak Tests

When the sealed test devices are placed in the evacuated chamber for leak testing, any helium gas previously injected into each sealed device may or may not have escaped through leaks in their enclosure. This can affect the leakage rate indicated by the mass spectrometer leak detector. The number of sealed devices that are removed in each lot from the (bombing) chamber for leak testing should be limited to a quantity that can be helium leak tested within a limited dwell time (typically 30 to 60 min maximum).

Devices that are gross leakers can lose essentially all of their contained helium tracer gas by leakage into air or vacuum environments in very short time periods. These could be missed entirely if the helium leak test were used alone during inspection. Thus, gross leakers should be separated from each lot of sealed test devices by gross leak tests (described above).

Lack of repeatability in helium fine leak testing of sealed devices can occur because, in most cases, the actual quantity of helium contained within each component (assuming that it is a leaker) is not known and cannot be calculated. (By comparison, with radioactive krypton-85 tracer gas of known concentration in nitrogen, the quantity present within the sealed test device is measured directly by scintillation or Geiger-Müller counter radiation measurements.) Thus, it is possible to calculate the true leakage rate by helium leak tests only if an appropriate theoretical relationship is available to relate internal gas pressure to the conditions of bombing and the delay between device removal from bombing pressure and its actual measurement of helium leakage with the mass spectrometer.

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This correlation between measured leakage rate and prior bombing conditions and subsequent time delays depends on knowledge of the following critical factors: (1) the mechanism of helium gas flow into the sealed test object during pressurization or bombing; (2) the pressure differential applied across the sealed boundary of the device enclosure during bombing; (3) the time of pressurization and external pressure of helium used during bombing, as well as the degree of vacuum (or internal gas pressure) existing within the device when bombing occurs; (4) the internal free volume of the sealed device to be tested; (5) the unfilled free volume in the evacuated test chamber while the test devices are contained in it; (6) the dwell time or delay between completion of pressurization during bombing and the time at which helium leakage measurements are made (also significant is the external gas pressure during dwell time, that is, the atmospheric air pressure or vacuum chamber pressure); (7) the leakage flow mechanism (molecular, viscous etc.) during the dwell time delay and the period of actual leakage measurement; (8) the actual helium concentration in nitrogen or air existing within the sealed test device at the completion of the bombing period.

The concentration could vary if mixed tracer gas (helium plus nitrogen) is injected into an evacuated test device during bombing or if 100 percent helium tracer gas is injected into sealed devices that already contain a significant air pressure (if previously stored in air or not subjected to evacuation before helium bombing).

Most leak testing documents used as helium leak testing standards or specifications for back pressure leakage testing are based on the assumption that leakage occurs in the molecular flow region and obeys simple exponential relationships, as indicated in Eq. 4 below.

Helium Fine Leak Testing of Devices Filled with Helium during Manufacture

The fine leak testing technique described here may be used for sealed electronic device packages that have been enclosed in such a fashion as to ensure that the internal cavity of the package contains a helium tracer gas content that provides a minimum of 20 kPa (0.2 atm) absolute partial pressure of tracer gas (100 percent helium) at a standard temperature of 25 °C (77 °F). A sampling inspection should be conducted on each 8 h work shift to verify that the specified amount of helium tracer gas is actually being sealed within each package. Within a maximum transfer time of 30 min after completion of the package sealing operation, the device under test is transferred into a chamber connected to an evacuating system and a helium mass spectrometer leak detector. Any tracer gas that leaks out is indicated by the leak detector as the measured leakage rate R_1. This measured leakage R_1 is converted to the equivalent standard leakage rate:

$$\textbf{(3)} \quad Q = R_1 \frac{P_i}{P_{\mathrm{He}}}$$

where Q is the equivalent standard leakage rate defined as that quantity of dry air at 25 °C (77 °F) in $Pa \cdot m^3 \cdot s^{-1}$ flowing through one or multiple leak paths when the high pressure side is at 100 kPa (1 atm) and the low pressure side is at a pressure not greater than 130 Pa (1 torr absolute); where the standard leakage rate is expressed in units of $Pa \cdot m^3 \cdot s^{-1}$ (or optionally, in std $cm^3 \cdot s^{-1}$); where R_1 is measured leakage rate defined as the leakage rate of a given package measured under specific conditions and using a specified (tracer gas) test medium; where P_i is total absolute internal gas pressure within the sealed device in pascal (or torr or bar); and where P_{He} is internal partial pressure of helium within test device, in the same pressure units as selected for P_i.

In many specifications, electronic devices with an internal cavity volume of 0.1 cm^3 (0.006 $in.^3$) or less are rejected if the equivalent standard leakage rate Q exceeds 5×10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-7} std $cm^3 \cdot s^{-1}$). Devices with an internal cavity volume greater than 0.1 cm^3 (0.006 $in.^3$) are rejected if the equivalent standard leakage rate Q exceeds 5×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-6} std $cm^3 \cdot s^{-1}$).

Fixed Helium Tracer Gas Fine Leak Test Technique

In the fixed technique for fine leak testing of sealed devices with helium tracer gas, the sealed electronic devices are leak tested under specific bombing time and testing time conditions specified for each size of internal device cavity (Table 6).

The bombing time is the time the devices are exposed to 99.5 to 100.0 percent pure helium tracer gas under the pressure of about 400 kPa for 2 h or a pressure of about 200 kPa for about 4 h. The maximum dwell time is the maximum time allowed after release of the bombing pressure, before the leakage is measured from the sealed device under

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test. This fixed technique is not used if the maximum standard leakage rate limit given in procurement documents is less than the limits specified for the flexible helium leak testing technique (described next).

Flexible Helium Tracer Gas Fine Leak Test Technique for Hermetic Seals

In the flexible helium fine leak test, the sealed electronic devices to be tested are subjected to a bombing pressure whose minimum level is 300 kPa (3 atm) of helium pressure. Values of bomb pressure, bombing exposure time and dwell time after bombing before leak testing are chosen so that actual measured tracer gas leakage rates for anomalous devices will be greater than the minimum detection sensitivity capability of the helium mass spectrometer leak detector. If the dwell time exceeds 60 min, graphs are plotted to determine an actual leakage rate R_1 value that will ensure overlap of leakage rates with those detectable with the gross leak test technique selected for subsequent tests of all devices.

For each combination of (1) sealed package internal free volume V, (2) bombing pressure P_E to which devices are exposed, (3) bombing time t_1, (4) dwell time t_2 following bombing and before leak testing and (5) maximum allowable equivalent standard rate Q (specified in procurement documents), theoretical relationships are used to calculate the rejection limit of the actual measured leakage rate R_1 by Eq. 4 described next.

Exponential Equations Assumed for Bombing and Leakage Transients

During the bombing operation, the pressure of helium tracer gas within the hermetically sealed device enclosure (if it is a leaker) is assumed to increase in accordance with an inverse exponential relationship:

$$\textbf{(4)} \qquad P = P_E \left(1 - e^{\frac{-Qt_1}{VP_0}\sqrt{\frac{M_A}{M}}} \right)$$

During the dwell time following bombing, the rate of leakage of helium tracer gas from leaking devices is assumed to follow a typical exponential decay transient:

$$\textbf{(5)} \qquad R_1 = \frac{QP_E}{P_0}\sqrt{\frac{M_A}{M}} \times \left(1 - e^{\frac{-Qt_1}{VP_0}\sqrt{\frac{M_A}{M}}} \right) \times e^{\frac{-Qt_2}{VP_0}\sqrt{\frac{M_A}{M}}}$$

where R_1 is measured leakage rate of tracer gas, helium, through the leak ($Pa \cdot m^3 \cdot s^{-1}$); Q is equivalent standard leakage rate in the same units as R_1; P_E is pressure of exposure in pascal (or in a unit called *atmosphere* [atm] = 101.325 kPa); P_0 is atmospheric pressure in the same unit as P_E; M_A is molecular weight of air = 28.7 $g \cdot mol^{-1}$; M is molecular weight of the tracer gas, helium = 4 $g \cdot mol^{-1}$; t_1 is time of exposure to P_E (second); t_2 is the dwell time between release of pressure and leak detection; and V is internal volume of the device package cavity (cubic meter).

Criteria for Rejection of Leakage Devices in Flexible Helium Leak Test

Unless otherwise specified, devices with an internal cavity volume of 0.01 cm^3 (6×10^{-4} $in.^3$) or less are typically rejected if the equivalent standard leakage rate Q exceeds 5×10^{-9} $Pa \cdot m^3 \cdot s^{-1}$ (5×10^{-8} std $cm^3 \cdot s^{-1}$). Devices with an internal cavity volume greater than 0.01 cm^3 (6×10^{-4} $in.^3$) and equal to or less than

TABLE 6. Fixed technique for leak testing of sealed devices by using helium tracer gas.

Internal Cavity Package Volume, cm^3 ($in.^3$)	Bombing Condition: Pressure kPa	Bombing Condition: Pressure ($lb_f \cdot in.^{-2}$)	Bombing Time (h)	Dwell Time (h)	Reject Limit: $Pa \cdot m^3 \cdot s^{-1}$	Reject Limit: (std $cm^3 \cdot s^{-1}$)
≤ 0.01 (≤ 6.1×10^{-4})	515 ± 15	(60 ± 2)	2.0 to 2.2	1	7×10^{-10}	(7×10^{-9})
0.01 to 0.40 (6.1×10^{-4} to 2.44×10^{-2})	515 ± 15	(60 ± 2)	2.0 to 2.2	1	2×10^{-10}	(2×10^{-9})
≥ 0.40 (≥ 2.44×10^{-2})	515 ± 15	(60 ± 2)	2.0 to 2.2	1	2×10^{-9}	(2×10^{-8})
≥ 0.40 (≥ 2.44×10^{-2})	310 ± 15	(30 ± 2)	2.0 to 2.2	1	1×10^{-9}	(1×10^{-8})

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0.4 cm^3 (0.024 $in.^3$)are typically rejected if the equivalent standard leakage rate Q exceeds 1×10^{-8} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-7} std $cm^3 \cdot s^{-1}$).

Devices with an internal volume greater than 0.4 cm^3 (0.024 $in.^3$) may be rejected if the leakage rate Q exceeds 1×10^{-7} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-6} std $cm^3 \cdot s^{-1}$).

Simplified Equation for Calculation of Leakage Rates after Helium Bombing

The quantity $\sqrt{(M_A \cdot M^{-1})} = \sqrt{(28.7/4)} = 2.7$, so Eq. 5 can be simplified as Eq. 6:

$$R = 2.7 \frac{QP_E}{P_0} \left(1 - e^{-2.7 \frac{Qt_1}{V}} \right) \times e^{-2.7 \frac{Qt_2}{V}} \quad (6)$$

In a similar way, the actual leakage rate for a device that has been filled with helium to the pressure P during manufacture can be found from the simplified Eq. 7:

$$R_1 = 2.7 \frac{QP_E}{P_0} e^{-2.7 \frac{Qt_2}{V}} \quad (7)$$

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CHAPTER

Leak Testing Techniques for Special Applications

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PART 1. Techniques with Visible Indications of Leak Locations[1]

Purposes of Leak Testing to Locate Individual Leaks

Leak testing for the purpose of locating individual leaks is required when it is necessary to detect, locate and evaluate each leak. Unacceptable leaks then can be repaired and total leakage from a vessel or system brought within acceptable limits. Methods for detecting and locating individual leaks are generally quantitative only in the sense that the lower limit of detectable leak size is determined by the sensitivity of the leak detecting indicators and test methods used. Thus, only rather crude overall leakage rate information could be approximated by adding the leakage rates measured for the detectable leaks.

Many different leak detecting, locating and measuring techniques and devices are available. The selection of test equipment, tracer gas and leak detection method is influenced by the following factors: (1) the size of the leaks to be detected and located; (2) the nature and accuracy of leak test information required; (3) the size and accessibility of the system being tested; (4) the system operating conditions that influence leakage; (5) the hazards associated with specific leak location methods; and (6) the ambient conditions under which leak location tests are required to be carried out. Wind, stratification effects and lack of air circulation can influence leak sensitivity and personnel.

Classification of Methods for Locating and Evaluating Individual Leaks

Methods for locating and evaluating individual leaks can be categorized in various ways, including by types of leak tracer used in the detection, location and possible measurements of individual leaks. A primary division is that between liquid tracers and more sensitive gaseous tracers. Leak location techniques that depend on tracer gas properties are listed below in general categories, in order of increasing leak sensitivity and complexity of test techniques.

1. Leak location techniques independent of any characteristic properties of the tracer gas include those that use, for example, candles, liquid and chemical penetrants, bubble testing and sonic or ultrasonic leak tests.
2. Leak location techniques involving use of tracer gases with easily detectable physical or chemical properties include gases with thermal conductivities or chemical properties differing from those of the pressurizing gas, for example, gaseous halogen compounds and gases having characteristic radiation absorption bands in the ultraviolet or infrared spectral ranges.
3. Leak location techniques involving tracer gases with atomic or nuclear properties providing easily detectable leak signals include helium and other inert gases having specific charge-to-mass properties that permit their sensitive detection by mass spectrometers and include gaseous radioactive isotopes detectable with particle counters and radiation detectors.

Techniques for Leak Location with Dyed Liquid Tracers

Testing for leaks by use of dyed liquid tracers is a nondestructive testing process closely related to the liquid penetrant testing process used to detect discontinuities open to the surface in test objects. For leak testing, however, the liquid penetrant is applied to one side of the enclosing wall of a test object or test system and, after allowing adequate time for the penetrant to seep through leaks, visual testing for leak location is carried out on the opposite side of the enclosure wall. Note that, in this type of test, pressurization of the test object is not usually required. The migration of the liquid tracer through the leak passageway is not due to an applied pressure differential. Instead, the physical forces required to cause the liquid to penetrate through the leak are provided by surface wetting capillary action and by surface tension effects characteristic of the liquid

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penetrant tracer and of the material surfaces that this tracer touches.

Significant differences exist between liquid tracers used in leak testing and liquid penetrants used for detection of surface discontinuities in test objects. These differences exist mainly in connection with the way applicable indicator dyes are used, their concentrations and the techniques for augmenting the detectability of leak indications.

Characteristics of Liquid Tracers for Locating Leaks

Liquid leak tracers are typically composed of a vehicle or carrier such as oil or water and of a tracer dye system incorporated into the liquid carrier to enhance visibility of leakage indications. The dye solubility in various carrier liquids as well as the coloration power of the dyes in dilute or in concentrated solutions in the carriers become important factors that control the sensitivity and ease of use of the liquid tracers. Nonfluorescent visible color dyes that may exhibit intense coloration in concentrated solutions usually lose their coloration power rapidly as they are extensively diluted in a solvent. For water base tracers, basic dyes provide the most intense colors. Solvent dyes usually yield the strongest colors for oil soluble tracer systems.

However, a fluorescent dye that has a reasonable efficiency in the conversion of ultraviolet radiation to visible light is usually much more effective as a leak tracer than is the average visible color dye. This occurs partly because of the greater response of fluorescent dyes in dilute solutions and partly because of the enhanced visibility or brightness contrast of fluorescent leak indications seen against a dark background (see Fig. 1). In closeup applications where leak testing can be carried out with an ultraviolet lamp under subdued white light conditions, it is often possible to get brightness contrast values for fluorescent leak indications that exceed several hundred to one. These conditions provide high levels of tracer sensitivity and excellent visibility of the leak indications.

Components of Fluorescent Tracer Dyes

Two broad categories are used in classifying fluorescent dyes with respect to their effectiveness in tracer usage in leak testing. *Sensitizer dyes* are materials that can yield strong fluorescent response in thin liquid films and at practical dye concentrations of about 0.5 percent or more. *Color former* dyes are materials used mainly in combination with sensitizer dyes for the purpose of shifting the color response by cascading the fluorescence. Color former dyes, when used alone, usually do not provide an acceptable level of thin film response but are sometimes extremely effective as bulk fluorescence tracers. The classification of dyes used in fluorescent tracers follows: (1) water soluble sensitizer dyes, (2) water soluble color former dyes, (3) oil soluble sensitizer dyes and (4) oil soluble color former dyes.

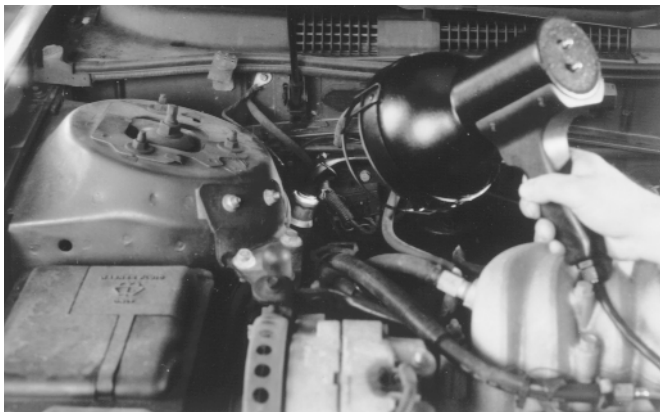
Advantages of Water Phase Fluorescent Leak Tracers

Water is the most plentiful and the least expensive solvent liquid available. Where

FIGURE 1. Leak testing using fluorescent tracer liquids: (a) fluid is added to air conditioning system; (b) ultraviolet radiation causes leaking fluid to fluoresce.

(a)

(b)



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the use of water as a tracer carrier is permissible, economic considerations alone would call for its use. Other considerations in favor of water include cases where water would ultimately be used as the contained liquid in a tank or pipeline being tested. In such cases, the use of an oil phase tracer liquid would be undesirable in view of the necessity for cleaning the oily tracer liquid off the test surfaces before letting water in. When the mode of bulk fluorescence is used in leak testing, water phase tracers are often preferred. With water phase leak tracers, the fluorescence response can be detected at dye concentrations on the order of a few parts per million or less. This level of leak (dye) sensitivity is greater by several orders of magnitude than the sensitivity attainable with oil phase tracer materials.

Applications of Water Phase Fluorescent Leak Tracers in Leak Testing of Boilers and Tanks

Water phase fluorescent racers are frequently used for detecting microleaks in water or steam systems. In this use, water containing a dissolved fluorescent tracer dye is introduced into the boiler, tank or system under test. The outside of the test object is then inspected in near ultraviolet radiation (365 nm wavelength mercury vapor radiation) for locations of points of leakage. In the case of large tanks, it is often found that leaks will show up only when the large tank is filled with water. In this case, the filled tank is subject to the stresses of weight and hydrostatic pressure incurred in normal usage of the tank. Stress may act to open leaks that otherwise are too small for easy detection or that exist only under stress. Pipelines, boilers, valves and other systems may also exhibit leakage only under conditions of pressure.

Large leaks may be readily detectable by a wetness that surrounds the points of leakage. Small leaks are more difficult to localize because the water carrier may evaporate as it exudes from the leakage point. Such small leaks will, of course, carry out some of the tracer dye, causing a buildup of dry dye around the point of leakage. In many cases, this accumulation of dye cannot be readily detected, because its fluorescence is quenched in the solid state. Virtually all of the useful water soluble fluorescent dyes exhibit concentration quenching or solid state quenching of fluorescence, at least to some degree.

Solvent Developers to Enhance Leak Indication of Water Phase Fluorescent Tracer

A useful technique for detecting points of leakage in boilers, pipelines and valves uses a water phase fluorescent tracer containing a special dye system that can be developed by application of a suitable spray solvent developer material. This process will work if a water activated color dye such as fluorescein, a water soluble fluorescent dye, is used. However, fluorescein yields indications having brightness response much lower than that of more efficient sensitizer dyes.

In this leak locating technique, the fluorescent tracer dye is dissolved in water, this fluorescent water is introduced into the system under test and the system is pressurized. If necessary, the test system is allowed to stand under pressure for several days. Then a liquid solvent developer is sprayed lightly over the external surfaces, with particular attention to joints, weldments and other regions where leaks might occur. Inspection is carried out in darkness or under subdued white light while the fluorescence is excited with ultraviolet radiation (similar to those widely used in fluorescent liquid penetrant testing for discontinuities in test objects).

Microtraces of dye accumulations at points of leakage will dissolve in the thin coating of developer liquid applied to surfaces under inspection. These minute amounts of fluorescent tracer dye dissolved in the developer can then undergo a transition, from the nonfluorescent solid state where fluorescence response is quenched to a high level of fluorescent brilliance. The technique of using liquid developers to enhance leakage indications obtained with water phase fluorescent tracers is sensitive enough for detection of leaks with submicroscopic cross sections.

Characteristics of Oil Phase Fluorescent Leak Tracers

The second important category of dyed liquid leak tracers is that of the so-called *oil phase tracer.* This type of leak tracer uses as its vehicle or carrier a low viscosity solvent liquid, preferably an aromatic oil. The fluorescent dye or dyes used in such tracers must be oil soluble in nature. Most of the available oil soluble sensitizer and color former dyes are readily soluble in aromatic solvents, much more so than they are in aliphatic oils or distillates. Some of the most efficient fluorescent sensitizer dyes are oil soluble in character. An oil-and-solvent soluble coumarin dye is used in many liquid penetrant and leak

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tracer formulations; however, certain vehicle materials such as the silicones and fluorocarbons will not support fluorescence with this coumarin type of oil phase fluorescent dye. Other proprietary dyes are available that require little or no support for their fluorescence.

Significance of Thin Film Fluorescence Response Characteristics of Leak Tracers

In cases where leaks are extremely small, the amount of dyed liquid tracer that can migrate through the leakage path is correspondingly small. Detection of such small leaks often depends on the fluorescence of extremely thin layers of dyed liquid that form indications at each point of leakage. The visible color response, as well as the fluorescence response of thin films of leaking dyed liquids, follows the same laws as similar films of color contrast or fluorescent liquid penetrants. The tracer sensitivity can be measured and calibrated as a function of the concentration of the indicator dye. The fluorescence response is about a linear function of the dye concentration. In some cases, it is even possible to estimate the dimensional magnitude of the leak through techniques similar to those used in evaluation of liquid penetrant testing.

The detectability of a given leak condition by dyed liquid tracers depends on at least two factors. First, the leakage passageway must be large enough to permit a visible amount of the dyed tracer liquid to pass through the leak during the test. Second, the dye solution (its brightness in thin films) must be dimensionally sensitive so that fluorescence or color response can be seen in the thin film exudation from the point of leakage. It is obvious that a large leakage path cross section will permit a greater flow of tracer liquid than would a small or restricted leakage path. Accordingly, when the detection of extremely small leaks with liquid tracers is desired, it is often necessary to use a dye tracer system that is characterized by a high level of sensitivity. (This is not always required, however, as explained below.)

Evaporative Leak Tracers to Detect Very Small Leaks

Sometimes microleaks are found in connection with surface porosity or other conditions under which it is extremely difficult to verify the leak condition. Where the leak is extremely minute, the rate of migration of the dyed liquid tracer through the leakage path may be slow that no films of the dyed liquid can appear at the exit point of leakage. If the solvent liquid of the tracer system is volatile, it will evaporate as it exudes from the leak outlet and a deposit of dry dye will accumulate. This state is sometimes obtained only after a time longer than projected for the test. The length of time required for leak indications to form by solvent evaporation will depend on the size of the leak and the concentration of dye in the tracer liquid. Small leaks in large tanks can be economically detected by taking advantage of the evaporative leak tracer technique. Fairly low concentrations of dye can be used and it is necessary only to allow sufficient time for an accumulation of dye to leak around a point of exit. In many cases, it may be necessary to use a solvent developer to enhance water phase evaporative tracer leak indications, as described earlier.

Limitations of Dyed Liquid Tracers for Leak Detection and Location

Liquid tracers, generally analogous to liquid penetrant testing media, have several basic limitations in comparison with gaseous leak tracers. In many cases, liquid cannot be introduced into the tank, pipeline or component under test. Vacuum chambers or vacuum pump systems fall into this category because liquid contamination and residues left after evaporation might serve as sources of virtual leaks or cause outgassing, which could be very hard to clean up from the vacuum system. As noted earlier, liquids can act to clog leaks so that more sensitive gas tracers cannot pass through the leaks. Refrigeration systems or equipment and air ducts may require leak testing without the use of liquids.

An obvious limitation results from the similarity of liquid penetrant tracers and liquid leak tracers. Liquid penetrants can be applied to test surfaces, where they enter surface connected discontinuities such as cracks and porosity. After cleaning of excess penetrant from these surfaces, the liquid penetrant tracer then exudes from the discontinuities (which typically do not penetrate through the wall thickness of the test material). Exudations and indications produced from discontinuities that do not penetrate through the wall or pressure boundary can form indications that are similar in appearance and characteristics to leakage indications formed by similar tracers.

Certain types of hermetically sealed components, such as electronic devices, are often tested by applying pressurized tracer systems to their external surfaces (as in helium bombing) so that some tracer

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can enter the test part through any leak passageways. Later, the tracer can escape from the interior volume and pass outward through the pressure boundary to form leakage indications.

This technique is less feasible when liquid tracers are used for leak testing. In most cases, the liquid would be an undesirable contaminant of the interior regions of sealed test objects. Secondly, the entry and later exudation of liquid tracer from leaks through the pressure boundary cannot be reliably discriminated from surface indications that do not penetrate the enclosure wall. Finally, as noted previously, exposure of test surfaces to liquids that might seep into and clog leaks can result in severe difficulties if later leak tests are conducted with gaseous tracers, because prior leak clogging with liquids can prevent the gas tracer from passing through the leaks.

Techniques for Leak Location with Gas Phase Dye Tracers

Gas phase tracers, used with dye indicators, have been developed to a high level of leak detection sensitivity. Either a visible color or a fluorescent dye can be used to augment the sensitivity of a gas phase leak tracer process. Detectors consist of a dye and a tracer gas that will react with each other to produce a change of color or fluorescence in the dye. A simple technique of gas phase leak testing with dye indicators is to pressurize the system to be tested with ammonia gas. Note that ammonia gas is possibly dangerous to use in leak testing unless it is carefully controlled and exhausted from work areas to avoid injury to personnel. With ammonia gas as the leak tracer, strips of pH indicator paper, moistened with water, can be used as probes to search out any points of ammonia gas leakage. This technique provides a medium-to-high leak test sensitivity for the detection of gas leaks but is awkward to use and ammonia fumes are unpleasant or toxic for test operations.

Ammonia Gas Leak Detection with Purple Dye Indicators

Bromocresol purple dye is used in chemical reaction leak testing with ammonia gas tracers. The dye is sprayed or brushed onto the outside surfaces of pressure vessel welds and allowed to dry. Upon drying, bromocresol purple turns into a yellow chalklike powder coating. The vessel under test is then pressurized with ammonia gas. If a leak exists, it is indicated by a change in the color of the powdered dye from a light yellowish green color to vivid purple. After testing, the dye is removed with an air hose, by a water wash or by wiping the test surface with a cloth. The concentration of ammonia gas can be as small as 50 μL of ammonia gas to 1 L of air (50 $\mu L \cdot L^{-1}$). Problems encountered with this leak testing technique are ensuring uniform adherence of the dye to the weld areas and the complete removal of the dye after completion of the leak test.

Gas Phase Leak Testing with pH Sensitive Dye Indicators

Various visible color and fluorescent dyes are sensitive to the concentration of active ions, either acid or alkaline. These ions determine the hydrogen potential (or pH), which indicates the ion content of pure water. The pH value for a solution can be shifted by addition of an acid or an alkali. A number of useful indicator dyes are quite sensitive to small changes in pH of the liquid in which they are dissolved. While oxidation reduced indicators are feasible, the most direct way to use these effects is to use a pH sensitive liquid and a tracer gas that will produce a change in pH of the liquid it contacts. This type of gas phase leak indicator is a liquid material coated onto areas suspected of having leaks. The indicator material then reveals the presence of leaks by fluorescent or visible color indications. A suitable indicator dye for producing visible color leak indications is phenolphthalein. However, numerous proprietary dye indicators are available that yield satisfactory fluorescence or color response as the result of very small changes in pH.

Most of the pH sensitive dyes can be dissolved in pure water or in water and alcohol mixtures. Glycols are effective as solvents for these dyes in many cases. In formulating the dye indicators, sufficient acid (such as hydrochloric acid) or base (such as sodium hydroxide) is added to the dye solution to shift the pH to a value close to the color change point of the indicator dye. The concentration of the indicator dye may be varied within substantial limits. At the lower extreme, changes of color or fluorescence may be detected with dye concentrations as low as one part per million (1 $\mu L \cdot L^{-1}$) in the solvent. The practical upper limit is a saturated solution of the dye. The solvent must be polar in nature so that it can ionize dissolved acids or bases. Examples of suitable solvents include various lower alcohols, glycols, glycol ethers and water. Alcohols and glycols are useful with water

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insoluble indicator dyes or where volatility or refractive index properties are advantageous. It has also been found that the sensitivity of the leak detection process can be greatly enhanced by dissolving the indicator dye in a highly purified, deionized water from which all traces of buffer salt residues have been removed. This allows the pH sensitive tracer dye to switch easily from one color or fluorescent state to another.

Selection of Tracer Gas for Leak Testing with pH Sensitive Dye Indicators

Two basic materials are used in leak testing and location with the gas phase tracer and pH sensitive dye indicator: (1) a vapor source and (2) an indicator paint. Ammonia vapor could be used, of course, but it represents a severe personnel hazard. Proprietary gas phase tracers have been developed that will yield a color change in response to a very minute quantity of vapors given off by simple amine liquids such as ethylene diamine or propylenediamine. Many simple amines have vapor pressures on the order of 1 kPa (10 torr) at room temperature. In normal use, the vapor concentration of such tracers cannot exceed 10 parts in 760 (the partial pressure ratio) or about 1.3 percent. Even this concentration would normally occur only quite close to the work area or within a pressure chamber. Thus, for low vapor pressure amines, the hazard to leak testing personnel may be considered to be negligible. During leak testing, the vapor source may be placed inside the test chamber or system, either in a shallow cup or on a cloth pad or sponge. Even without pressurization of the chamber, the vapors from the amine liquid may often diffuse through extremely small microleaks, pinholes, porosities in seals, welds and brazed joints. Of course, a small amount of pressurization will act to accelerate the leakage of the vapors.

Formulation of pH Sensitive Indicator Paints for Coating Test Surfaces

The indicator paint used to indicate leak locations must contain a suitable indicator dye. The unbuffered pH of the dye solution must be carefully adjusted to a point just below the critical pH of fluorescence or color change. Also, a thickening agent and an evaporative dilutent should be included so that a paint coating that is applied to a test surface will become partially dry and form a greasy or pasty coating on the test surface. A typical proprietary leak tracer paint is a liquid that can be brushed onto the test surfaces where it dries to a pale blue greaselike coating. Vapors of the amine tracer that diffuse through leakage points act to trigger a color change in the pasty coating so that bright red spots are formed to indicate leak locations.

Chemical Fumes Leak Locator Techniques

The chemical smoke or fumes leak indicator technique depends on a chemical reaction between a tracer fluid and a chemical reagent to produce visible leak indications. Various nonstandardized chemical indicator leak tests exist, of which a few representative examples are described next. Tracer gases or vapors used in chemical indicator leak testing include ammonia, hydrogen sulfide and carbon dioxide. Both ammonia and hydrogen sulfide are considered hazardous or toxic gases and should be used with proper safety precautions. The chemical indicator techniques are static leak testing techniques used fundamentally as leak location procedures. The longer the leak test is run, the more sensitive it becomes. A major advantage of these tests is their relatively low cost because no expensive instrumentation is required. Major limitations of these tests include the possibility that reactive chemicals might damage materials or parts of systems being tested or present hazards to personnel.

Chemical Indicator Leak Detector Techniques Using Ammonia Tracer Gas

Ammonia (NH_3) gas has found industrial use as a gas phase leak tracer. It is chemically basic and corrosive and it is usually prohibited at concentrations exceeding 75 $\mu L \cdot L^{-1}$. The corrosive action of ammonia is exhibited strongly on brass parts. The ammonia tracer can be introduced as an anhydrous ammonia gas or by placing a cloth saturated with liquid ammonia in the pressurized space. The sensitivity of the leak tests will depend on the concentration of the ammonia gas and will increase with higher concentrations. Hydrogen chloride vapor can be introduced by probing the atmosphere in the vicinity of a leak with an open bottle of hydrochloric acid or with a swab wetted with 0.1 normal hydrochloric acid. (Note that hydrochloric acid is a dangerous chemical and should not be brought into contact with the skin or eyes or inhaled through nose or mouth.) Where leaks are present, the leakage of ammonia tracer gas can be revealed by a white chemical fog or mist of ammonium chloride that forms when

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the ammonia comes into contact with the hydrogen chloride vapor. This technique should be used only with good ventilation because of the obnoxious characteristics of both hydrogen chloride vapor and ammonia gas.

Another technique for use of ammonia in a chemical indicator leak test is to use sulfur dioxide (SO_2), as from a sulfur candle, as the revealing reactant. Ammonia combines with sulfur dioxide gas to produce a white mist of ammonium sulfide. Sulfur dioxide is not quite so irritating or corrosive as hydrogen chloride; however, sulfur dioxide is still an obnoxious gas and should be used only in well ventilated test areas.

A third gas that can be used as a revealing reactant for leaking ammonia vapor is carbon dioxide (CO_2). Carbon dioxide is not as sensitive as either hydrogen chloride or sulfur dioxide when used for leak location with ammonia tracer gas but has the advantage of being noncorrosive.

Chemical Indicator Leak Detector Techniques Using Carbon Dioxide Tracer Gas

Another chemical indicator leak location technique uses carbon dioxide tracer gas to fill the vessel or system under test. The leak indicator consists of agar-agar solution loaded with sodium carbonate and phenolphthalein. This bright red spray solution yields a stable red film when sprayed onto test surfaces. Leaking carbon dioxide causes the formation of white spots at points of leakage. The volume of red agar-agar film that is changed to a white color is directly proportional to the amount of leaking carbon dioxide that enters the indicator film. To apply this indicator film, the agar-agar solution is preheated and stored at temperatures of 65 to 70 °C (149 to 158 °F), at which temperature the agar is liquid. For application to test surfaces, the hot agar solution is poured into a preheated sprayer bottle and sprayed with preheated compressed air. The spray nozzle should be held about 0.6 m (2 ft) from the test surface. A single coating should be applied in one pass and multiple coatings should be avoided. Spraying should always be done in a horizontal direction, never vertically. After completion of leak testing, the agar-agar film can be removed by a jet of high velocity air. The air jet will lift the agar film from the test surface, leaving the surface in a clean, dry condition.

Formulation of Agar-Agar Film Solution Sensitive to Carbon Dioxide Tracer Gas

The spray solution sensitive to leaking carbon dioxide tracer gas is formulated (by weight) of 1.0 part of agar-agar to 0.15 part of phenolphthalein. It is important to obtain the proper viscosity of agar-agar solution, which should be between 0.5 and 1 mPa·s (5 and 10 atm) at the spraying temperature, about 65 to 70 °C (149 to 158 °F). To prepare the spray solution, the dry powders of agar-agar, sodium carbonate and phenolphthalein should be blended thoroughly in proper proportions. The dry mixed powders should be added to cool distilled water while stirring to disperse the solid constituents. The resulting mixture should be stirred constantly while heating to temperatures between 96 and 98 °C (205 to 208 °F), either on a hot plate or with a steam bath. When the solids have dissolved completely and a clear solution has been obtained, it should be allowed to cool to a temperature between 65 and 70 °C (149 to 158 °F). At this storage temperature, the indicator film solution can be stored in a closed glass container sealed to exclude air and the small quantities of carbon dioxide in the atmosphere.

Miscellaneous Additional Leak Detector Techniques Using Chemical Indicators

Numerous additional techniques of leak testing using chemical indicators of leaking tracer gases of liquids have been proposed and used on occasion. Several of the following examples involve use of hazardous tracer gases such as hydrogen sulfide or acetylene and should be avoided where feasible. Hydrogen sulfide has been used as a tracer gas to locate leaks in containers. The indicator solution responsive to hydrogen sulfide is a five percent solution of stannous chloride. The leak locations are then shown by brown stains of stannous sulfide. Because of the poisonous nature of hydrogen sulfide, this is not a very popular leak testing technique. An alternative chemical that responds to hydrogen sulfide by the formation of precipitates consists of indicator solutions containing silver salts or lead salts.

Leaks in natural gas pipelines have been located by the reactions of silver salt solutions with acetylene tracer gas. Acetylene is also a hazardous gas that explodes spontaneously at pressures above 200 kPa (30 $lb_f \cdot in^{-2}$). Acetylene forms explosive mixtures with air or oxygen in almost any proportions.

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Anhydrous copper sulfate has been used as a chemical leak indicator where water is used as the tracer fluid (as in detection of leaks from water lines or water leaking during hydrostatic tests). When leaking water contacts a developer film of anhydrous copper sulfate, it shows blue indications at locations of water leakage. An alternative technique of indicating water leakage is a lime wash or similar type of coating applied over external surfaces of test vessels or test systems within 24 h after hydrostatic pressure testing.

Typically, only large leaks such as centerline cracks or pinholes can be indicated by this technique. Fine check cracks may not be indicated when water under pressure is used as a tracer. Sometimes nearly penetrating discontinuities may enlarge sufficiently under hydrostatic pressure to seep water. This water seepage will produce wetness indications in the lime wash indicator, but interpretation of these indications may be difficult.

Fluorescent or Visible Tracer Dyes in Hydrostatic Test Fluids

Fluorescing dye indicators can be added to the pressurized liquid (usually water) used in hydrostatic pressure tests. The dye may provide bulk fluorescence or can more appropriately be of the type that provides intensified fluorescence after evaporation of the carrier liquid. In some cases, a developing fluid or film may be applied to the external surfaces of the test object where leakage is possible or suspected. During or following hydrostatic pressure tests (which often serve as proof tests simulating application of service stresses), the test operator can visually examine all welds under strong white light (if visible color dyes were used) or under near ultraviolet radiation (if fluorescent dyes have been used in the pressurizing fluid). Slow, continuing seepage from small leaks thus can be indicated by brilliant fluorescent indications.

Smoke Bomb Techniques for Locating Leaks

Gas and smoke bombs (Fig. 2) can be used for detecting and locating leaks. Generally speaking, a volume of smoke sufficient to fill a volume five or six times larger than the volume of the vessel or system to be tested is required for a smoke test. Medium size volumes such as pressure vessels can be tested by closing all vents, igniting a smoke candle or smoke bomb and placing it inside the vessel to be tested. All openings in the test system should then be closed. Almost immediately, smoke will be seen issuing from the larger leaks present in the test object. The order of escaping smoke will also assist in pinpointing the locations of leak exits. When smoke tests are made on steam generating boilers and similar equipment with volumes of 2.8×10^3 m^3 (10^5 ft^3) or larger, it is frequently desirable to apply air pressure to the interior volume of the system under test.

Smoke candles can be obtained that provide from 100 m^3 (4×10^3 ft^3) of smoke in 30 s to 4×10^3 m^3 (1.4×10^5 ft^3) of smoke in 2 or 3 min. The color of the smoke may vary from white to gray, depending on the smoke density and available lighting. The smoke used in leak location is generated by chemical reaction, contains no explosive materials and has been assumed to be nontoxic.

FIGURE 2. Smoke generators: (a) titanium tetrachloride smoke stick; (b) dye based smoke candles.

(a)

(b)

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Halide Torch for Visual Detection of Halogen Vapor Leaks

The halide torch consists of a burner connected to a tank of gas or alcohol. Some of the air required for combustion is drawn into the flame (chimney fashion) through a tube near the bottom of the burner. A flexible inlet extension of this tube is used as a probe to locate leaks. If the leak is large enough to be detected by this technique, a bright green flame color (characteristic of copper) appears when the open end of this detector probe tube passes near the point of halogen tracer gas leakage. The halide torch permits locating leakage down to about 200 to 300 mL (8 to 10 oz) of refrigerant-12 or refrigerant-22 gas per year. This corresponds to a leakage rate of about 1×10^{-4} Pa·m^3·s^{-1} (1×10^{-3} std cm^3·s^{-1}) based on an air flow of 1 L·s^{-1} (2 ft^3·min^{-1}) and a halide torch sensitivity to refrigerant gas estimated as 100 µL·L^{-1}. Refrigerant-12 gas (CCl_2F_2) is considered to be the best tracer gas with respect to leak sensitivity, vapor pressure, inertness and safety.

General Advantages and Limitations of Visible Indicators of Leak Locations

Leak testing techniques providing visual indications of leak location have several common advantages and limitations. In most instances, tests that produce visual images are psychologically satisfying because the observer sees direct visual evidence of the existence of each leak. In many cases, this evidence can build up in magnitude or intensity as leak test time or observation time increases. This continuity (or repeatability) of test indications is also reassuring. The leak indications do not disappear as long as the leakage continues. (Some types of electronic instrumentation used for leak detection, described below, show only transient indications as tracer gas is encountered and then lose sensitivity if exposure to this tracer gas continues.) In general, most observers having leak indications pointed out to them in typical cases can soon learn to see similar indications and evaluate them as leaks. Operators can be trained to use visual leak indicator test techniques rapidly, develop confidence in these test indications and demonstrate the test signals to management and other personnel readily and convincingly. Where motivation toward process improvement or to repair existing leaks is needed, these psychologically effective leak location test techniques have many true advantages.

The precision of leak location of most of these visual leak indicator tests far exceeds that of many gas tracer or detector probe tests, where there is no instantly visible evidence of the leak locations. Often the response of these detection instruments can be so slow that the probe has been moved well away from the leak locations before the instrument signals that a leak has been detected. The operator must then go back and search slowly to find the individual leak (or multiple leaks) that produces the delayed test signal. Similarly, after an intense tracer gas signal has entered such electronic detection instruments, a time delay is often required to clean the test instrument and restore its sensitivity to traces of leaking gas.

By contrast, however, the visual leak indicator tests do not in themselves ensure that a test object is free from all leaks (because some leaks might exist in areas unexamined or overlooked during testing). They also are not appropriate for measurement of leakage rates, even though rough comparisons of leakage rates can be made from the visual leak signal. The visual indicator techniques also typically lack the very high sensitivity to small leaks provided by electronic instrumentation responsive to leak tracer gases. Liquid leak penetration media in general cannot pass through the smallest leaks that are readily shown by tracer gas flows. Thus, leak sensitivity of visual leak indication tests is typically significantly less than the best sensitivity attainable by basic techniques of leakage measurement or by leak probing and tracing with electronic leak detector instruments.

A final testing advantage of visible leak indicator techniques is their possible application to systems in service or to systems pressurized with liquids or gases during proof tests. Addition of tracer dyes to water or oils in storage tanks or hydraulic systems can reveal transient leak locations that might not be detectable under normal leak testing conditions. These simple tests are often used to find and permit repair of large leaks before undertaking more costly tests with highly sensitive leak detection systems.

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PART 2. Primary Containment Leakage Rate Testing in the United States Nuclear Power Industry

The primary containment completely encloses the reactor vessel. It is designed to retain its integrity as a radioactive material barrier during and following accidents that release radioactive material into the primary containment volume. Primary containment is considered to be intact when it can be demonstrated that the total containment leakage rate is less than the maximum allowable leakage rate (*La*) when the containment is maintained at the calculated peak accident pressure (P_{ac}). Values of L_a and P_{ac} are specified in each plant's technical specifications or *final safety analysis report* (FSAR).

Maximum allowable leakage rate L_a is calculated such that 10 *CFR* Part 100, dose limits are not violated under post accident design basis accident conditions.[2] The calculations assume conservative weather conditions and direct leakage of nuclides from primary to secondary containment. The WASH-1400 report[3] specifies the amount and type of nuclear sources terms that would be contained in such leakage. Most or all plants in the United States use allowable leakage rates based on WASH-1400 source terms.

The values of L_a and P_a are plant specific. For United States plants, L_a values range between 1.5 and 6.3 $L \cdot s^{-1}$ (200 and 800 std $ft^3 \cdot h^{-1}$). P_a values range between 60 and 450 kPa (9 and 65 $lb_f \cdot in.^{-2}$ gage). This results in plants being allowed an equivalent sharp edged orifice hole diameter of between 1.59 and 4.76 mm (6.25×10^{-2} and 1.875×10^{-1} in.) for the entire containment structure.

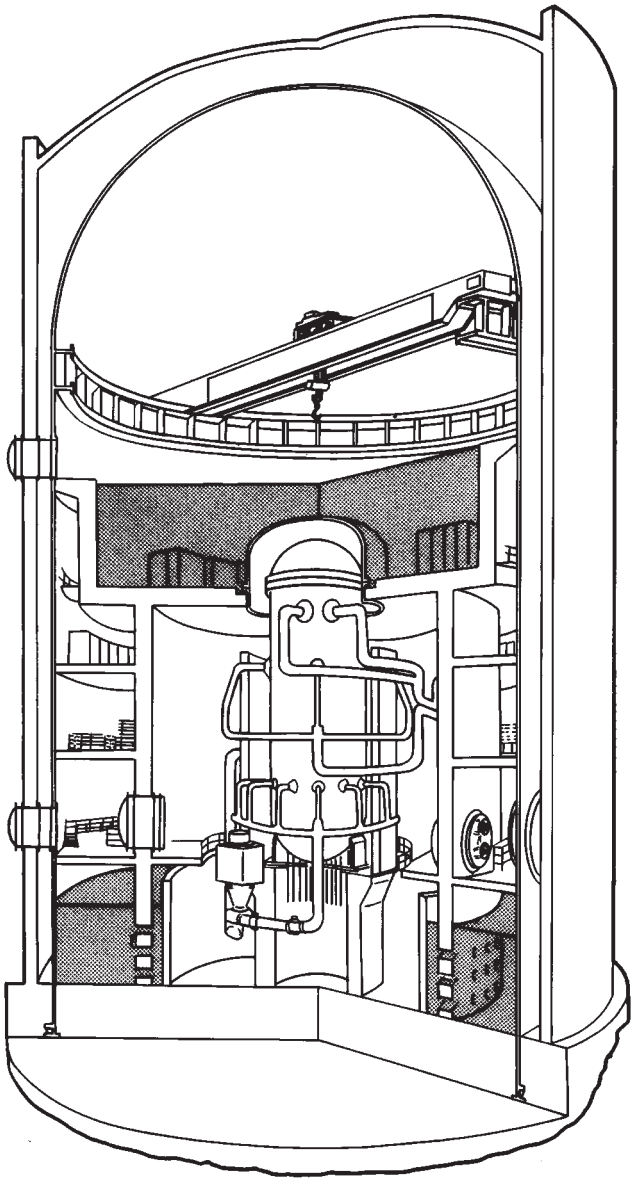
Leakage rate for test purposes is defined as that leakage that occurs in a unit of time, stated as a percentage of weight of the containment air volume at the leakage rate test pressure that escapes to the outside atmosphere in 24 h.

Three types of leakage rate tests are performed in the nuclear power industry, Type A, B and C testing. The Type A test is a test of the entire containment structure (Fig. 3) and all of its potential air leakage pathways; this is also referred to as the *integrated leakage rate test* (ILRT). Type B tests are intended to detect local leaks in nonvalve type penetrations such as hatches, flanges and metallic bellows. Type C tests are intended to measure leakage rates through valves. Type B and C testing is also known as *local leakage rate testing* (LLRT).

Integrated Leakage Rate Testing

This is a pressure decay test of the entire containment structure. The integrated leakage rate test has six distinct phases: pressurization, stabilization, leakage rate

FIGURE 3. Multibarrier containment vessel for boiling water reactor nuclear power plant.



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measurement, the verification test and depressurization.

First, the containment structure is pressurized with clean dry air up to the peak accident pressure P_{ac}. Following a temperature stabilization period of at least four hours, the leakage rate measurement portion of the test may begin.

The total containment dry air mass is typically measured at 10 min intervals for at least 8 h. The slope of the least squares fit line of these masses with respect to time is proportional to the leakage rate. The 95 percent *upper confidence limit* (UCL) of this slope calculated using a single sided Student *t* distribution is used for comparison against the acceptance criteria.

Because the total containment pressure changes very little over the test duration, a constant value of leakage rate is expected to be measured. The leakage rate of change must be less than a specified value. A check is also performed on the amount of scatter of the dry air masses.

For the Type A test to be considered acceptable, its 95 percent upper confidence limit, its linearity and the data scatter must all be within their acceptance limits.

The verification test is performed immediately following the successful completion of the Type A test. A metered leakage rate of about L_a is induced from the containment. Then the containment leakage rate is again measured for at least 4 h. The new leakage rate is expected to equal the Type A test leakage rate plus the induced metered leakage rate. If agreement within 0.25 L_a cannot be shown, then the verification test is considered a failure and the Type A test must be repeated.

The total containment dry air mass M is calculated using Eq. 1:

$$M = 144 V \frac{P - P_v}{RT} \quad (1)$$

where P is the average total containment air pressure. One or more absolute pressure transmitters may be used. The average value from those transmitters is P.

P_v is the containment vapor pressure. At least three (typically ten) relative humidity or dew point sensors are distributed throughout containment. Values from these sensors and their companion temperature sensors, (relative humidity only) are used along with psychrometric tables to calculate the partial pressure of water vapor in the air. Each sensor is assigned a volume fraction and its reading is weighted using this fraction. P_v is the calculated total containment volume weighted vapor pressure.

T is the containment temperature. At least 10 and typically 25 temperature sensors are distributed throughout the containment. Each sensor is assigned a volume fraction and its temperature reading is weighted using this fraction. T is the calculated total containment volume weighted temperature.

V is the free air volume of containment. In most cases, this value is assumed to be a constant throughout the test. In some plants where significant water level changes occur, the free air volume is updated periodically. In all cases, the size of the structure is assumed to be constant. This is a valid assumption for most modern thick walled plants. The original design thin walled steel globes experienced volume changes due to diurnal effects significant enough to impact the leakage rate measured.

R is the perfect gas constant of air. Because containment pressures are always far less than the critical pressure of air, this is an excellent assumption.

Details of integrated leakage rate testing methodology may be found in ANSI/ANS-56.8-1994, *Containment System Leakage Testing Requirements*.[4]

Local Leakage Rate Tests (LLRTs)

Penetrations, process lines and other pathways that have the potential to allow gaseous leakage from inside to outside the primary containment under normal or postaccident conditions are considered to be Appendix J pathways.[5] These pathways must be subject to periodic local leakage rate testing.

The leakage rate through each component when subjected to a pressure differential of at least P_{ac} is measured and compared against an acceptance criterion for that individual component. That leakage rate is also added to the total leakage rates from the other entire Appendix J[5] pathways to ensure that the total is less than that plant's specified limit. These tests are also used to satisfy ASME Section XI[6] requirements for inservice testing.

Local leakage rate tests are typically performed using either the flow makeup or pressure decay methods. Other techniques such as reference volume, tracer gas detection or soap solution are also sometimes used.

In all local leakage rate tests, the tested barrier is pressurized with clean dry air or nitrogen. For the entire duration of the test, the test pressure differential may not drop below P_{ac}. If the test pressure aids the barrier in sealing, then the pressure differential across the barrier may never exceed 1.1 P_{ac}. A barrier may be tested in a direction in reverse of the direction of expected post accident pressure only if

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such a test can be shown to yield conservative results.

When a pressure decay test is performed, the test volume's temperature and pressure must be recorded at the beginning and at the end of the test. The minimum duration allowed for a pressure decay test is 15 min after stable conditions have been achieved. There is no minimum duration for flow makeup tests; however, test data are required to be obtained only after stable conditions have been achieved.

Performance Based Testing Programs

Containment leakage rate testing in the nuclear power industry is performed in accordance with the federal regulation *10 CFR 50, Appendix J.*[5] This rule has two options, Option A calls for Type A, B and C tests to be performed at fixed intervals, Option B allows for the test intervals to be determined for each component based on its performance history.

Under Option A, no fewer than three Type A tests must be performed within every ten year interval for inservice inspection. Poor performance does not require the test interval to be shortened. Although two consecutive test failures will require no more than about an 18 month intervals between future Type A tests until two consecutive passing tests have been achieved. Type B and C tests are required to be performed every 24 months regardless of component performance. Containment airlocks and ventilation system valves have more stringent testing requirements due to their industry wide poor performance history and due to their greater safety significance.

Under Option B, the baseline test interval for Type A testing is 48 months. Upon the completion of two consecutive successful tests, the interval may be extended up to ten years. Type B and C tests have a baseline test interval of 30 months. Passing two consecutive tests allows for extension of the test interval up to 60 months. If a Type B barrier passes three consecutive tests its interval may be extended up to 120 months. Airlocks, ventilation valves and a specified small population of other valves are excluded from the performance based program and must be tested at more frequent fixed intervals.

The basis for performance based testing programs is contained in the *NEI Industry Guideline Document 94-01 Revision D.*[7]

As-Found Testing

For a performance based program to be effective, the components in the program must be tested following some significant length of service prior to any repairs or adjustments. This is referred to as *as-found testing.* All components are required to be as-found tested under Option B. Repairing or adjusting a component before its periodic test generally results in the component being returned to its baseline interval, as does replacement or significant modification.

Reduced Pressure Testing

When most nuclear plants were tested before operation, the Type A test was performed twice — once at full pressure P_{ac} and once at half pressure. These two results along with the full pressure test acceptance criteria were then used in a correlation specified in 10 *CFR* 50 Appendix J to formulate a half pressure test acceptance criterion.[5] The plant then had the option in future operational tests to perform the Type A test either at full pressure or at half pressure with a more stringent acceptance criterion.

Review of test data from the last 20 years has shown that no accurate, conservative or reliable correlation may be derived. It is possible to specify a conservative correlation between full and half pressure leakage rates through fixed size orifices. Unfortunately, due to a large number of resilient seals, the size of the leakage pathways is a function of applied pressure. This makes a reliable correlation not possible.

The recent Option B to Appendix J recognizes this fact and no longer allows reduced pressure type A testing.

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PART 3. Leak Testing of Geosynthetic Membranes[8]

Purpose of Destructive and Nondestructive Seam Testing

Seam testing is routinely performed as an integral part of the construction quality control and quality assurance programs for installation of geosynthetic membranes or pond liners. Seam testing routinely consists of destructive testing of seam samples in the laboratory and nondestructive seam testing in the field. The following discussion describes the purpose of destructive and nondestructive testing of seams, introduces the various nondestructive test techniques, discusses the procedures for the most common techniques in detail and recommends means for testing bumps and other areas difficult to seam.

As part of quality control and quality assurance monitoring of geosynthetic membrane liners, destructive and nondestructive testing is conducted to establish the strength and continuity or seams, respectively. Destructive testing involves cutting out a section of seam and sending it to an offsite laboratory for strength testing. Destructive testing is not discussed herein. Information on this type of test can be found in the literature.[9-12]

Nondestructive testing is performed to evaluate continuity of the seam. Discontinuities in a seam can serve as potential sources of leakage. A discontinuity 0.1 cm^2 (0.016 $in.^2$) in area can result in 1250 L (330 gal) per day of leakage through a geosynthetic membrane overlying a free draining subbase. Therefore, it is important that possible seams be 100 percent nondestructively tested. Geometric constraints will not allow all seams to be tested but the length of seam that cannot be tested needs to be kept to a minimum. Special construction quality assurance procedures need to be followed for seams not tested.

Nondestructive Testing Methods

Nondestructive test methods are summarized in Table 1.[8,13,14] Of these methods, the vacuum box, air pressure and air lance tests are routinely used because of their simplicity. The *probe test* is typically used for small lengths of seam because it is time consuming.

Vacuum testing and *air lance testing* can be performed by construction laborers with minimal training. The vacuum test requires some specialized equipment. The air pressure test requires a more skilled individual but can be performed using equipment purchased at a hardware store. Vacuum testing and *air pressure testing* are used for stiff geosynthetic membrane such as high density polyethylene (HDPE) and very low density polyethylene (VLDPE) geosynthetic membranes. The air lance test is used for flexible geosynthetic membranes such as polyvinyl chloride (PVC) and chlorosulfonated polyethylene (CSPE) geosynthetic membranes.

Ultrasonic testing is not commonly used, primarily because specialized equipment and an experienced operator are required.

A conductive wire is incorporated into the geosynthetic membrane seam during installation. The spark test requires a technician familiar with installing the conductive wire. An improperly installed wire may be more detrimental to the seam integrity than not testing the seam. Because specialized equipment is required, the *spark test* is usually used in areas that cannot be tested through other nondestructive test methods.

The *ponding test* is an effective procedure for testing the primary geosynthetic membrane of a double liner systems in sumps and at pipe penetration. The test is conducted by filling the sump with water. In the case of pipe penetrations, it may be necessary to build a small dike around the penetration using sand bags or other temporary barriers. Liquid in the leak detection system is an indication of leaks in the primary liner. If a leak is detected, the sump is drained and the geosynthetic membrane is visually monitored for discontinuities. If possible, the seams are tested by using one of the methods in Table 1.

The *electrical leak location method*[15,16] is used to find holes in the parent material as well as discontinuities in the seams of geomembrane liners. The test is used to find holes in an inservice liner that is known to be leaking or is incorporated as part of a construction quality assurance monitoring program. After the liner system is completely installed, the potential for further damage is minimal. The test is conducted with the liner under

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hydrostatic load or under the load of the protective cover soil. The electrical leak location method is the only method that can reliably and accurately locate leaks in landfill liners covered with protective soil.

A voltage is applied between one electrode in the material above the liner and either another grounded electrode or an electrode in the leak detection zone between double liners. If leaks are present in the geomembrane liner, then electric current will flow through the leaks. The electrical potential field will be altered, and leaks are identified as characteristic zones of measured high electric potential. These variations in the electrical potential are measured by using specialized equipment to scan the submerged or soil covered liner. Different types of equipment are used for surveys with soil on the liner, for surveys by personnel wading in water on the liner and for surveys using a towed sensor for deep or hazardous water.

Vacuum Test

The vacuum test is the most common nondestructive test method for stiff geosynthetic membranes joined with a single seam. The vacuum test consists of applying a vacuum to a portion of the seam using a box with a transport window. A soapy solution is applied to the liner within the box. Any leaks in the liner will cause the soapy solution to bubble portion of the liner. The equipment required to perform the vacuum test consists of the following

1. a vacuum box assembly consisting of a rigid housing, a transparent viewing window on top of the housing, a soft synthetic rubber gasket attached to the bottom of the housing, a Egort hole or valve assembly through which the vacuum is applied, and a vacuum gage;
2. a vacuum tank and pump assembly equipped with a pressure controller and pipe connections;
3. a pressure/vacuum hose with fittings and connections;
4. a soapy solution; and
5. an applicator for the soapy solution.

Some boxes are available with small electric vacuum pumps mounted directly on the box. It is important that the pump have sufficient power to establish the required negative pressure, typically on the order of 34 kPa (5 $lb_f \cdot in.^{-2}$).

TABLE 1. Methods for leak testing of geosynthetic membranes.[8,13,14]

Test Method	Speed	Recording Method	Operator Dependency	Operation
Air lance	fast	manual	high	Air is blown through nozzle at a seam. Disbonds are indicated where membrane vibrates.
Air pressure (dual seam)	fast	manual	low	In double seamed membrane, intermediate channel is pressurized and pressure is measured. Sections up to 100 m (330 ft) can be tested.
Electric sparking (to ground)	fast	manual	low	Leakage of high voltage current (15 to 30 kV) to ground causes spark, indicating pinholes or other discontinuity in thermoplastic liner.
Electric wire	fast	manual	low	Conductive wire is embedded in seam and connected to probe. Amplitude of about 20 kV is used to indicate discontinuities in membrane.
Electrical leak location	fast	manual or automatic	low to moderate	Current flows through leak from electrode in containment area to electrode on other side of liner. Separate instrument is used to scan for leakage location.
Probe (mechanical point)	slow	manual	very high	Seam is poked with stiff instrument such as screwdriver. Results are qualitative and not very reproducible.
Ultrasonic	moderate	automatic	moderate	Ultrasonic techniques (impedance, pulse echo and through-transmission) are applicable to fused, not adhesive, seams and are reliable when conducted by experienced operators over small areas.
Vacuum box	slow	manual	moderate	Segments of seam are isolated in vacuum box for bubble testing.

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In general, the steps to be followed in vacuum testing of a seam are as follows.

1. If vacuum testing a fusion seam, the flap must be cut off with a hand held cutter with a hooked blade to expose the seam for testing. This technique is not recommended.
2. Energize the vacuum pump and reduce the tank pressure to about 34 kPa (5 $lb_f \cdot in.^{-2}$) below atmospheric pressure.
3. With a soapy solution, wet a strip of geosynthetic membrane that extends 150 mm (6 in.) beyond the area to be covered by the vacuum box.
4. Place the box over the wetted area.
5. Close the bleed valve and open the vacuum valve.
6. Push down on the box to create a leak tight seal.
7. For a period of not less than 15 s, examine the geosynthetic membrane through the viewing window for the presence of soap bubbles.
8. If no bubble appears after 15 s, close the vacuum valve and open the bleed valve. Before moving the box over the next adjoining area, place a mark (with a marker that will not damage the geosynthetic membrane) on the geosynthetic membrane at the leading edge of the viewing window, then move the box over the next adjoining area so that the last mark on the geosynthetic membrane is at the rear of the viewing window, and repeat the process. Often the outline of the box is left in the soapy solution applied to the geosynthetic membrane. In this case, it is not necessary to mark the geosynthetic membrane.
9. If soap bubbles appear, the area is marked, repaired and retested in accordance with the requirements of the project documents.

It is important that a good seal be established between the vacuum box and the geosynthetic membrane. Otherwise, air can pass beneath the gasket and create bubbles within the vacuum box. If this situation occurs, it is difficult to detect leaks in the geosynthetic membrane seam. When testing geosynthetic membrane it is often necessary to replace the gasket periodically.

Therefore, it is useful if two vacuum boxes are available. One box can be used on the day the gasket on the second box is being replaced.

The 15 s testing interval stated above is an approximate time. The vacuum box may need to be held longer or can be released a few seconds sooner. The important point is to make sure a good seal has been obtained, the vacuum is developed in the box and the entire surface in the box is carefully viewed for bubbles It is readily apparent when the vacuum in the box is developed because the geosynthetic membrane will pull up into the box. It should be noted that the vacuum box is most effective with recently installed geosynthetic membranes. When retesting pond liners the vacuum box may not be effective because the hydrostatic pressure created by liquid in the pond has pressed the geosynthetic membrane liner against the soil. As a result, the vacuum in the box may not be strong enough to pull the liner up, thereby allowing air to pass through any leaks. In this case, the electric leak location method is probably the most efficient approach to check for leaks.

Air Pressure Test

The air pressure test is used with seaming processes that produce a double seam with an enclosed channel. The testing consists of pressurizing the channel with air. Leakage is detected if the air pressure cannot be maintained.

The equipment required for the air pressure test consists of the following:

1. an air pump that is equipped with a pressure gage that can generate and sustain a pressure between 175 and 210 kPa (25 and 30 $lb_f \cdot in.^{-2}$) and that is mounted on a cushion to protect the geosynthetic membrane;
2. putty or similar material used for sealing the ends of the channel;
3. two pairs of vice grips;
4. a hose with fittings and connections; and
5. a sharp hollow needle or other air pressure feed device.

The air pressure test typically entails the following procedures.

1. Cut a small opening at both ends of the seam to be tested.
2. Seal both ends of the air channel with putty.
3. Insert the needle or other approved pressure feed device through the putty into the channel created by the double rack fusion seam process.
4. Clamp both ends of the air channel with the vice grips.
5. Place a protective cushion between the air pump and the geosynthetic membrane.
6. Energize the air pump to a pressure between 25 and 210 kPa (4 and 30 $lb_f \cdot in.^{-2}$), close the valve and sustain the pressure for not less than 5 min.
7. If the loss of pressure exceeds 23 kPa (3 $lb_f \cdot in.^{-2}$) for 1.5 mm (0.06 in.) or thicker geosynthetic membranes or 31 kPa (4 $lb_f \cdot in.^{-2}$) for geosynthetic membranes thinner than 1.5 mm (0.06 in.) or if the pressure does not

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stabilize, the length of seam is considered unacceptable. A shorter length of seam can be tested to isolate the unacceptable area.
8. To verify that there is airflow through the entire channel, remove the seal at the end of the channel away from the air source and observe the loss of pressure on the gage.
9. If on checking for complete airflow it is found that there is a blockage in the tunnel, the seam must be repaired.
10. Remove the needle or pressure feed device and patch the holes at both ends of the seam.

Air pressure testing is preferred to vacuum testing because long lengths of seam can be tested quickly. Air pressure testing is especially efficient for testing smooth geosynthetic membrane. It has been observed in the field that, for textured geosynthetic membrane, blockage of the air channel occurs because the asperities of the textured surface often become attached together during the seaming process. As a result, it is some times more efficient to vacuum test textured geosynthetic membrane seams.

Air Lance Test

In the air lance test, a hollow wand with a nozzle is used to blow air at the geosynthetic membrane seam. Unseamed portions are detected because the geosynthetic membrane will vibrate.

The equipment used for the air lance test consists of the following: (1) an air lance wand equipped with a 2.4 mm (0.09 in.) hose fitting and with quick connect fitting connections, (2) an air hose with quick connect fittings, (3) a regulator and (4) an air compressor.

The steps of an air lance test are as follows.

1. Pass air at 210 kPa (30 $lb_f \cdot in.^{-2}$ gage) minimum to 275 kPa (40 $lb_f \cdot in.^{-2}$ gage) maximum pressure through the wand.
2. Hold the wand at a 45 degree angle to the field seam and about 50 mm (2 in.) above the geosynthetic membrane.
3. Move the wand over the seam so that the air is directed toward the seam edge and toward surface of the upper and lower geosynthetic membrane surfaces to detect unbonded areas.
4. Vibration of the geosynthetic membrane indicates unbonded areas within the seam or other undesirable seam conditions that need to be patched in accordance with the project specifications.
5. The patches should also be tested using the air lance test.

The air lance method is efficient for testing geosynthetic membranes that are 0.75 mm (0.030 in.) or thinner. For thicker geosynthetic membranes, it is sometimes more difficult to detect unbonded areas.

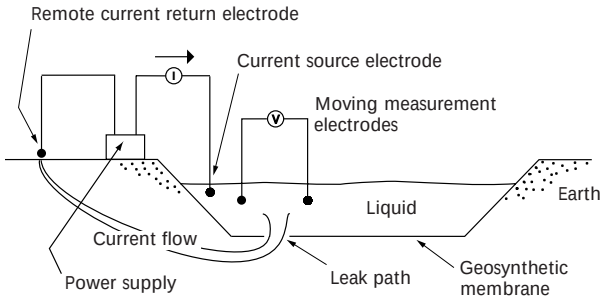
Electrical Leak Location Test[15]

The high voltage electrical leak location survey method is used to locate leaks in geosynthetic membrane lined facilities. Electrical leak location surveys locate leaks that were not previously detected using other test techniques or were caused during the placement of the protective soil cover. On average, 22.5 leaks per 10 000 m^2 (nine leaks per acre) are located when this method is used to test geosynthetic membrane lined facilities.[16] The method and equipment can locate leaks after protective soil cover is placed over the liner and is a very cost effective way to check liner installation quality or quickly solve a leakage problem

The electrical leak location method detects electrical paths through the geosynthetic membrane liner caused by water leaking through the leaks (see Fig. 4). A voltage is connected to one electrode placed in the water or soil covering the liner and to an electrode placed in the leak detection zone for double lined systems or in earth ground for single lined systems. Electrical current flowing through the leaks in the liner produces localized anomalous areas of high current density near the leaks. These areas are located by making electrical potential measurement scans throughout the survey area.

With the proper implementation of equipment and survey procedures the electrical leak location method can detect and locate very small leaks. The leak signal amplitude is proportional to the amount of electrical current flowing through the leak. To maximize this current, a high voltage power supply with safety circuits can be used. The high voltage power supply produces a

FIGURE 4. Electrical leak location method for leak testing of geosynthetic membranes (pond liners).



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proportionally higher leak signal to provide optimum leak detection.

Electrical leak location surveys are conducted with *water* or *soil* covering the geosynthetic membrane (Fig. 5). For manual surveys where *water* covers the liner (Fig. 5a) the water depth must be between 150 and 750 mm (6 and 30 in.) in depth when surveying the bottom floor area. The manual survey system consists of a portable electrical probe and associated instrumentation. The operator wades in the water and systematically scans the submersed liner to locate any leaks.

Survey lanes on the bottom are scanned with overlapping coverage. The survey operator moves the probe laterally across a 2.5 m (8 ft) span, advances about 0.3 m (1 ft) and then moves the probe laterally in the opposite direction. In this manner, the total area of water covered liner is surveyed with the probe passing within about 150 mm (6 in.) or less from every submerged point on the liner. The liner field seams that can be located are double checked. When detected, leaks are located to within 25 mm (1 in.) or less and immediately marked.

If the water in the geosynthetic membrane lined impoundment cannot be lowered or the water in the pond is too dangerous for personnel to wade then a remote survey method is used to survey the liner. The remote survey method uses a probe that is towed back and forth across the impoundment. The probe cable is connected to the leak location instrumentation. The probe is designed to scan along the bottom of the pond as survey personnel pull the electrode across the pond. Survey scans are typically made on 0.6 m (2 ft) spacings, so the probe will pass within 0.3 m (12 in.) of any potential leak.

For best results, for any type of electrical leak location survey, electrical conduction paths through or around the liner should be eliminated or insulated. Penetrations such as leachate collection lines should be constructed of nonelectrical conducting materials.

An electrical leak location survey of *soil* covered geosynthetic membrane (Fig. 5b) is a very effective means for finding leaks that occur while placing a protective soil cover over the geosynthetic membrane liner or that occur after the liner installation.

Because a geosynthetic membrane liner of a landfill has protective soil covering the liner, point-by-point electrical potential measurements are made instead of the continuous potential measurements used for surveys in water. The measurements are made on the soil surface using special electrodes. The electrical potential data are recorded in a portable data acquisition logger and then downloaded to a portable computer for processing and data analysis.

To survey a liner, an electrical conduction path through leaks in the liner to the soil subgrade or leak detection layer must be established. Therefore, the soil must have sufficient moisture to allow for electrical contact with leak. This can be achieved by wetting the soil after placement on the high density polyethylene (HDPE) liner and letting the water percolate to the liner surface. The electrical measurements are then conducted on the moist soil. In addition, a synthetic drainage layer located between two high density polyethylene liners must be flooded with water. This is not required if the drain layer is made of a natural material such as sand, gravel or clay.

In addition, for single liners, the protective soil cover placed on the primary liner should not contact earth ground. This can be prevented by leaving an area of liner material temporarily exposed at the top of the side slope or along the edge of the liner.

FIGURE 5. Electrical leak location method for geosynthetic membranes: (a) survey through liquid; (b) survey of floor of landfill liner with side slope in background.

(a)

(b)

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The survey is conducted by making potential measurements along survey lines spaced at regular intervals across the bottom floor area of the landfill. The potential measurements are typically made with a dipole electrode separation of 0.75 m (2.5 ft) on survey lines spaced 0.75 m (2.5 ft) apart. Therefore, data are collected on a 0.75 m (2.5 ft) grid pattern. When a suspect area is indicated in the processed data, manual measurements are taken to further localize the leak.

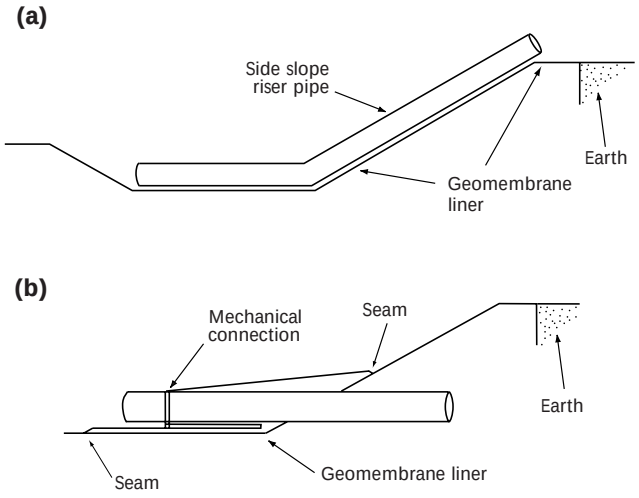
Sumps and Pipe Penetrations

Most lining system have a sump or a low point from which liquid is removed. The preferred method of liquid removal is to pump directly from the collection point as shown in Fig. 6a.[8] In some cases, it is necessary to penetrate the lining system with a pipe, as shown in Fig. 6b.[8] In both cases, liquid will always be standing on the lining system Therefore, it is important that the seams in these areas are continuous. Sumps often have steep sides that make nondestructive testing difficult. Seaming around pipe penetrations and connecting the liner to the pipe is also difficult. Therefore, it is recommended that all seaming operations in such areas be continuously monitored.

Also, some form of nondestructive testing needs to be performed. An appropriate place to use the spark test is the seams around a pipe penetration. The liner is usually fastened to a pipe by a mechanical connections such as a hand clamp. To ensure the clamp forms an effective seal between the pipe and the boot, it is recommended that water be ponded above the top of the pipe. This method is effective only for the top or primary liner of a double liner system.

The purpose of nondestructive testing of geosynthetic membrane seams is to ensure continuity of the weld. A number of nondestructive test methods are available. The most common methods are the vacuum, air pressure and air lance tests. Other available nondestructive test methods are the ultrasonic, electrical leak location spark and probe tests. Sumps and other low lying areas of a lining system may have liquid continuously standing on the lining system. Therefore, it is important that nondestructive testing of these areas be performed. In these areas, simple ponding tests may be performed to determine if more sophisticated nondestructive tests are required.

FIGURE 6. Placement of sump pipe for liquid removal: (a) side slope riser pipe; (b) pipe penetrating geosynthetic membrane liner. The connection of geosynthetic membrane to pipe is a common source of leakage. Seams too close to pipe may be difficult to test with vacuum box.



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Part 4. Residual Gas Analysis[17]

Leak testing of some type is required for any device or system that must contain or exclude a gas or liquid. These products range from tiny integrated circuit packages to systems as large as nuclear power plants and 16 000 m^3 (100 000 barrel) petroleum tanks. Many techniques are available for leak testing.[18,19] Electronics technology has made the electronic gas analyzers suitable for the factory environment, with simplified data presentation, stable control circuits and the possibility of operation by relatively unskilled personnel.

The helium mass spectrometer leak detector has become a commonly used piece of shop and field equipment for product quality control leak testing and for maintenance of all types of piping systems and many other applications. The residual gas analyzer (RGA) has become a commonly used device for continuous monitoring of thin film vacuum deposition and other manufacturing processes because it provides dynamic, real time information on the relative amount of the various gases in the system. A residual gas analyzer can be used to warn when too much of an unwanted gas appears, if a material is not sufficiently outgassed, if there are other indications of both poor and good process conditions or if general vacuum system performance needs to be monitored.

Purposes of Residual Gas Analysis

The application of residual gas analysis for leak testing components or systems is similar to that of a helium mass spectrometer, but the residual gas analyzer scans over a user selectable range of ionized gas particles instead of one gas and therefore offers various advantages.

1. Problems in the test system can be identified when the product is leak tested in a vacuum chamber. Chamber leakage, chamber outgassing, product leakage and virtual leakage can be identified.
2. Many tracer gases can be used — for example, the one most economical, the one normally inside the sealed part or the one that provides the best residual gas analyzer response.
3. Multiple parts can be tested in one vacuum chamber by using a different tracer gas in each part.

Residual Gas Analyzer Operation

Various manufacturers offer a popular radio frequency quadrupole residual gas analysis systems with sophisticated electronics for programming and displaying total system pressure and partial pressure of various gas species, data reduction and control and alarm modes, as well as interface ports for external computer or multipoint recorder connection.

Analytical laboratory mass spectrometers are available that can discriminate between particles with differences in relative atomic (A_r) mass as small as 0.01 A_r. The portable residual gas analyzer can resolve down to 1 A_r, suitable for most factory applications. The residual gas analyzer measures the partial pressure of gases by a three stage process of ionization, mass separation and detection. The gas ions are sorted by their mass-to-charge ratio and, therefore, each species will produce a peak proportional to its partial pressure in the vacuum system and its mass-to-charge ratio. Hence, for quantitative leakage measurements, the residual gas analyzer and the complete leak test system must be calibrated for the particular gas of interest.

The quadrupole residual gas analysis includes a filament, electron multiplier and other components that may decrease in performance if allowed to accumulate contamination. When the multiplier gain decreases by a factor of 200, the residual gas analysis head must be cleaned or elements must be replaced. Various filament materials designed for specific types of service are available to reduce this problem. This is similar to the maintenance required for a helium mass spectrometer.

Residual Gas Analysis Leak Testing

The instrument must have a mass range suited for the leaking gases to be detected. Table 2 lists some of the cracking pattern peak values of typical gases and vapors suitable for leak detection and vacuum system performance analysis.

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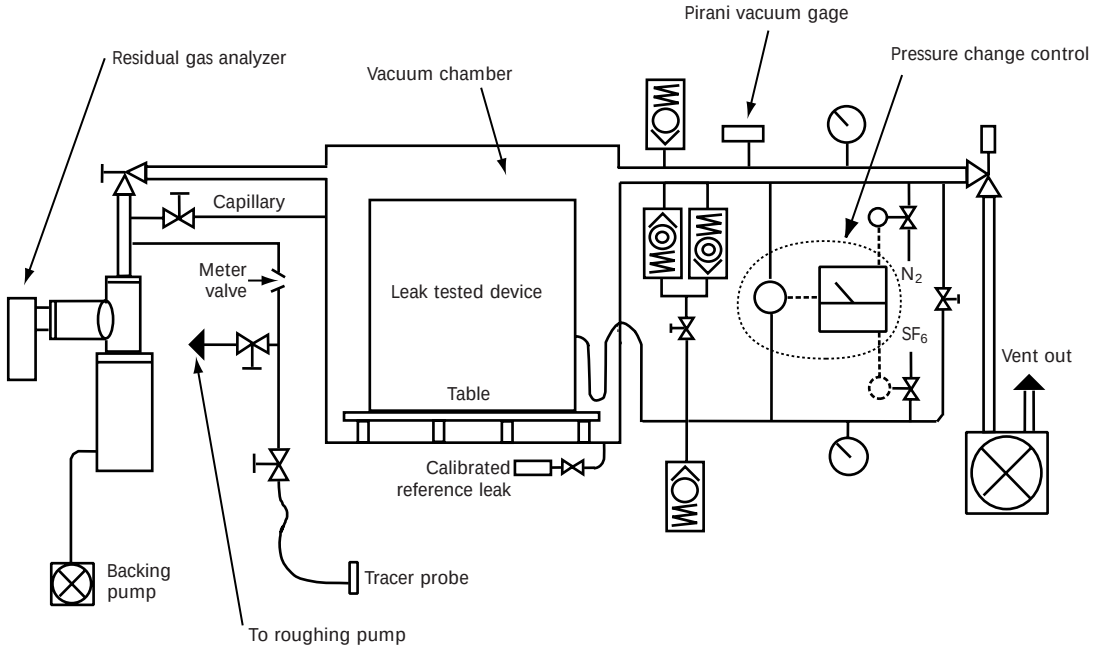
The residual gas analyzer may be used to detect leaks in a product filled with a suitable gas by simply placing the product in a vacuum chamber, evacuating the chamber and analyzing the residual gas for the product tracer gas. When using two or more tracer gases simultaneously, the operator must be sure that each has its own distinct peak of sufficient magnitude. Various products with different tracer gases may be tested at one time with the residual gas analysis system because each tracer gas mass number will identify the leaking product. The sensitivity of a helium leak test system can be increased by evacuating the vacuum chamber with a special cryogenic pump that does not pump the helium tracer gas. Leaks may be located with a residual gas analyzer by using the detector probe method, as shown in the system piping schematic (Fig. 7).

A vacuum chamber was designed and constructed to test a large missile guidance head. A mounting platform on drawer slides is provided so that the 127 kg (280 lb) head can be easily positioned, secured and then pushed into the chamber. After the vacuum test, the platform in the extended position places the head in a convenient location for leak sniffing and repair. The entire test system weighs 2.9 Mg (6.4×10^3 lb) and measures $2.29 \times 1.78 \times 1.68$ m ($90 \times 70 \times 66$ in.) high. The $1.37 \times 1.37 \times 1.52$ m ($54 \times 54 \times 60$ in.) vacuum chamber is

TABLE 2. Residual gas analysis cracking pattern peak values for gases and vapors.

Gas or Vapor	Relative Molecular Mass
Helium	4
Water vapor	18
Oxygen	32
Argon	40
Ethanol	31
Methanol	31
Isopropyl alcohol	45
Acetone	43
Petroleum pump oil	55 or 77
Fomlin Y-25™	69
705 DP oil™	78
Polyether DP oil™	51
Toluene	91
Sulfur hexafluoride	127

FIGURE 7. Residual gas analysis system for leak testing and backfill.



Legend

= compound pressure gage

= pressure relief valve, 100 kPa (15 $lb_f \cdot in.^{-2}$)

= pressure relief valve, 10 kPa (1.5 $lb_f \cdot in.^{-2}$)

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electropolished 304 stainless steel and the O-ring sealed door is nickel plated aluminum, providing a relatively low outgassing structure.

The control panel contains the residual gas analyzer control unit, the guidance head backfill pressure control and gages, chamber vacuum gage, control valves and the residual gas analyzer turbo pump power supply. Figure 7 shows how the residual gas analyzer radio frequency head and analyzer assembly, chamber gas sample admittance valves, turbo pump and piping of the system are interconnected.

Principle of Operation

The pressure differential permitted between the inside and outside of the guidance head is limited to 10.4 kPa (1.5 $lb_f \cdot in.^{-2}$) because of its structural design. This explains the differential pressure control system provided to ensure that this differential will not be exceeded during evacuation and backfill of the chamber and the guidance head.

The chamber and guidance head are evacuated to remove the air and the guidance head is backfilled with sulfur hexafluoride leak tracer gas to 10.4 kPa (1.5 $lb_f \cdot in.^{-2}$). The residual gas analyzer valve is then opened to the chamber and the residual gas analyzer control unit cathode ray tube display presents the relative partial pressure of the residual gases in the chamber. If sulfur hexafluoride (relative molecular mass M_r (SF_6) = 127) is indicated, the actual leak rate is calculated from the system calibration data.

The means of calibrating a system is similar to that outlined in the *ASME Boiler and Pressure Vessel Code*, Sec. 5, Art. 10.[20] A calibration leak standard is connected to the vacuum chamber opposite the leak test sample port, and the residual gas analyzer response to the tracer gas from the leak is recorded. A full range leak rate versus detector response curve is plotted by testing with leak standards of various leak rates. The response of this residual gas analyzer system to a 1×10^{-5} $Pa \cdot m^3 \cdot s^{-1}$ (1×10^{-4} std $cm^3 \cdot s^{-1}$) helium leak standard in the vacuum chamber was a 20 percent increase in the partial pressure in 20 min. This response is very good for such a large vacuum chamber. Also, the cleanup time was 20 min.

Because it responds to any tracer gas, residual gas analysis is more versatile than helium mass spectrometry for many leak testing applications on small to medium systems. It can be used also for analyzing systems that cannot be inspected with the helium mass spectrometer. If large quantities of helium are required, the residual gas analyzer may also provide the most economical method of leak detection because it uses a less expensive tracer gas. The vacuum system trouble shooting capability and process control functions make the residual gas analyzer a valuable tool for many vacuum processes.

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16

CHAPTER

Leak Testing Glossary

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Introduction

Most of the definitions in this glossary are adapted from the text in this volume and from the *Nondestructive Testing Handbook*, second edition.[1-10] The definitions in this glossary have been modified to satisfy peer review and editorial style. For this reason, references given in this glossary should be considered not attributions but rather acknowledgments and suggestions for further reading.

The definitions in the *Nondestructive Testing Handbook* should not be referenced for inspections performed according to standards or specifications or in fulfillment of contracts. Standards writing bodies take great pains to ensure that their documents are definitive in wording and technical accuracy. People working to written contracts or procedures should consult standards when appropriate.

This glossary is provided for instructional purposes. No other use is intended.

Definitions

absolute pressure: Pressure above absolute zero value, or pressure above that of space empty of all molecules. Equal to sum of local atmospheric pressure and gage pressure.[1,10]

absolute temperature: Temperature above absolute zero value. Absolute zero temperature is expressed as 0 K or –273.15 °C (–460 °F).

acceptable quality level (AQL): Maximum percent defective (or the maximum number of units with rejectable anomalies per hundred units) that, for the purposes of sampling tests, can be considered satisfactory as a process average.[8,10]

acceptance criteria: Standard against which test results are to be compared for purposes of establishing the functional acceptability of a part or system being examined.[10]

acceptance level: Test level above or below which test objects are acceptable in contrast to *rejection level*.[4,10,12]

acceptance standard: Specimen similar to test object and containing natural or artificial discontinuities that are well defined and similar in size or extent to the maximum acceptable in the product. See *standard*.[4,6,7,10]

accumulation test technique: Detecting the total amount of leakage by enclosing the component under test within a hood, bag, box, shroud or container. For pressure testing, any gas leaking from the component accumulates in the space (volume) between the component and the enclosure. For vacuum testing, any gas leaking into the component accumulates in the leak detector sampling the evacuated component. Accumulation of tracer gas in a measured time period provides a measure of the leakage rate.[1,10]

accuracy: Degree of conformity of a measurement to a standard or true value.[1,10]

acoustic emission: In leak testing, elastic waves resulting from the flow of fluids through leaks in the frequency range 30 to 100 kHz.[5,10]

acoustic emission leak testing: Leak test method that uses acoustic emission.[10]

adsorption pump: Pump that creates a vacuum by collecting gas on the interior surfaces of the pump. Pressures of 2 Pa (20 μbar) are readily attained. The pump has a finite capacity but may be regenerated for additional use.[11]

air flow: In leak testing, flow of air from the probe inlet to the sensitive element of the halogen leak detector that carries the tracer gas from the leak to the sensing diode.[1,10]

alkali ion diode: Kind of sensor for halogen gases. In this device, positive ions (cations) of an alkali metal are produced on the heated surfaces (usually platinum) of the diode. One electrode is at a negative potential and attracts cations that are released when a halogen gas passes between the sensor electrodes. Provides an output current to operate the indicator on the halogen leak detector.[1,10]

ambient temperature: Temperature of surrounding atmosphere. Also called *atmospheric temperature* or *dry bulb temperature*.[10]

anomaly: Instance of variation from normal material or product quality.[4]

artificial discontinuity standard: See *acceptance standard*.

artificial flaw standard: See *acceptance standard*.

artificial source: In acoustic emission, a point where elastic waves are created to simulate an acoustic emission event. The term also defines devices used to create the waves.[5,10]

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***ASNT Recommended Practice No. SNT-TC-1A*:** A set of guidelines for employers to establish and conduct a nondestructive testing personnel qualification and certification program. *SNT-TC-1A* was first issued in 1968 by the Society for Nondestructive Testing (SNT, now ASNT) and has been revised every few years since.[10]

atmosphere or **atmospheric conditions:** See *standard atmospheric conditions.*

atmospheric pressure: Ambient pressure caused by the weight of the earth's atmosphere. Because the weight of the earth's overlying atmosphere decreases with increase in altitude, atmospheric pressure decreases with elevation. Also called *barometric pressure.* At sea level, *standard barometric pressure* is taken as 101.325 kPa (14.696 $lb_f \cdot in.^{-2}$). It is also equal to the pressure exerted by a mercury column 760 mm (29.92 in.) high — that is, equal to 760 mm Hg (29.92 in. Hg) or 760 torr.[1,10]

automated system: Acting mechanism that performs required tasks at a determined time and in a fixed sequence in response to certain conditions.[8,10]

background contamination: Tracer gases in a test system that initiate a response from the leak detector and that may or may not be attributable to a leak.[1,10]

background signal: Steady or fluctuating output signal of a test instrument caused by the presence of acoustic, chemical, electrical or radiation conditions to which the sensing element responds.[1,10]

backstreaming: Movement of pumping fluids from the pump back to the vacuum chamber.[11]

baffle: System component, typically a plate, that condenses pump fluids before they reach the vacuum chamber and returns fluid to the pump.[11]

barometer: Pressure gage used to measure the atmospheric pressure at a specific location.[1,10]

barometric pressure: Ambient pressure caused by the weight of the Earth's atmosphere.[1,10] See atmospheric pressure.

bell jar: Kind of evacuated test chamber. See *vacuum pressure testing.*

black light: Disfavored term for *ultraviolet radiation.*

black light filter: Filter that transmits ultraviolet radiation between 320 and 400 nm wavelengths while absorbing or suppressing the transmission of the visible radiation and hard ultraviolet radiation with wavelengths less than 320 nm.[6,10,13]

Bourdon tube: See *quartz Bourdon tube gage.*

bubble leak testing: Pressure test where leakage is indicated by formation of bubbles by escaping gas. Methods include immersion, vacuum box and bubble solution tests.[1]

capillary action: Tendency of liquids to penetrate or migrate into small openings, such as cracks, pits or fissures. The positive force that causes movement of certain liquids along narrow or tight passages.[2,10]

calibrated leak: Instrument or specimen providing leakage at known rate for purposes of reference or comparison. See *standard.*

carrier fluid: (1) Fluid that acts as a carrier for the active materials. (2) Fluid in which fluorescent and visible dyes are dissolved or suspended, in liquid penetrants or leak tracers.[2] (3) Liquid vehicle in which fluorescent or nonfluorescent magnetic particles are suspended for ease of application.[6,10,13]

certification: Process of providing written testimony that an individual is qualified. See also *certified.*[8,10]

certified: Having written testimony of qualification. See also *certification.*[8,10]

choked flow: Phenomenon where, while pressure downstream is gradually lowered, velocity through an orifice increases until it reaches the speed of sound in the fluid (also known as *sonic flow*).[1]

cleanup time or **cleanup:** Time (time constant) required after a tracer gas has ceased to enter a leak test system, for the system to reduce its signal output to 37 percent of the signal indicated before the tracer gas had ceased to enter the leak testing system.[1,10]

code: Standard enacted or enforced as a law.[8,10]

cold cathode ionization gage: Ion gage in which the ions are produced by a cold cathode discharge, usually in the presence of a magnetic field that lengthens the path of electrons between the cathode and anode.[1,10] It has a range of 1 Pa to 0.1 mPa (10 mtorr to 1 µtorr).[11]

cold light: Obsolete word for *fluorescence.*[8,10]

cold trap: Device that condenses vapors and prevents oil or water molecules from entering a vacuum chamber.[1]

color contrast dye: Dye that can be used in a penetrant to impart sufficient color intensity to give good color contrast indications against the background on a test surface when viewed under visible light.[2,10]

color contrast penetrant: Penetrant incorporating a dye, usually nonfluorescent, sufficiently intensive to give good visibility to discontinuity indications under visible light.[2,10]

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complete testing: Testing of an entire production lot in a prescribed manner. Sometimes complete testing entails the inspection of only the critical regions of a part. One hundred percent testing requires the inspection of the entire part by prescribed methods. Compare *sampling, partial*.[8,10]

conductance: Flow characteristics of a tube, manifold or leak path expressed in $m^3 \cdot s^{-1}$.[11]

cryogenic pump: Pump that condenses chamber gas on a cold surface of 4 to 80 K (–269 to –194 °C). Cooling is provided by liquid gas such as liquid helium or by refrigeration.[11]

defect: Discontinuity whose size, shape, orientation or location make it detrimental to the useful service of its host object or which exceeds the accept/reject criteria of an applicable specification.[6,14] Some discontinuities may not affect serviceability and are therefore not defects.[2] All defects are discontinuities.[2] Compare *discontinuity* and *indication*.[10]

detector probe: Adjustable or fixed device through which air and/or tracer gas is drawn into the leak test instrument and over the sensing element or detector. Also called a *sampling probe* or a *sniffer probe*.[1,10]

detector probe test: Pressure leak test in which the leakage of a component, pressurized with a tracer rich mixture, is detected by scanning the test object boundary surface with a detector probe connected to an electronic leak detector. Leakage tracer gas is pulled from the leak through the probe inlet to the sensing element to cause a visible or audible signal on the indicator of the leak test instrument.[1,10]

diffusion: Process by which molecules intermingle as a result of concentration gradients or thermal motion.[2] Spreading of a gas through other gases or solids within a volume.

diffusion pump: High vacuum pump with no moving mechanical parts that uses a vapor jet to sweep gas from the vacuum chamber and achieve pressures as low as 1 nPa (10 ptorr).[11]

discontinuity: Interruption in the physical structure of a part.[10] A discontinuity may or may not be considered a *defect*.

displacement pump: Mechanical pump that physically sweeps gas out of a volume and creates a vacuum. Rotary piston and rotary vane pumps are two examples. A displacement pump can achieve pressures in the 0.1 to 1.0 Pa (10 to 1 mtorr) range.[11]

drift (electronic): Change in output reading of an instrument, usually due to temperature change.

dry bulb temperature: Alternate term for ambient or atmospheric temperature.[1,10]

dynamic testing: Testing in which the system under test is pumped continuously.

elastomer: Natural or synthetic rubber gasket material used to make a vacuum tight seal in a vacuum system.[1]

equivalent standard leakage rate: See *standard leakage rate*.

examination, general: Test or examination of a person's knowledge, typically (in the case of nondestructive testing personnel qualification) a written test on the basic principles of a nondestructive testing method and general knowledge of basic equipment used in the method. (According to ASNT's guidelines, the general examination should not address knowledge of specific equipment, codes, standards and procedures pertaining to a particular application.) Compare *examination, practical* and *examination, specific*.[10]

examination, practical: In certification of nondestructive testing personnel, a hands-on examination using test equipment and sample test objects. Compare *examination, general* and *examination, specific*.[10]

examination, specific: In certification of nondestructive testing personnel, a written examination that addresses the specifications and products pertinent to the application. Compare *examination, general* and *examination, practical*.[10]

flammability: Tendency to combust, considered to be characteristic of liquids having flash point below 60 °C (140 °F) and a vapor pressure not exceeding 275 kPa (40 $lb_f \cdot in.^{-2}$) at 37.8 °C (100 °F).[1]

flash point: Lowest temperature at which vapors above a volatile, combustible substance ignite in air when exposed to an ignition source.[6,10,14]

flaw: Imperfection or unintentional discontinuity. See also *defect* and *discontinuity*.[2]

flow measurement: Determining the extent of leakage by measuring the rate of flow of gas into or out of a system or component under test.[1]

fluorescence: Emission of visible light from a material in response to ultraviolet or X-radiation. Formerly called *cold light*.[8,10]

foam leak test: Bubble leak test technique in which the tracer gas blows a hole through a blanket of foam covering the test object, thus indicating the location of the leak.[1]

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fore pump: Mechanical pump in a helium mass spectrometer that performs initial evacuation of a system to a pressure of 0.1 Pa and then accepts the exhaust from the high vacuum pump such as a diffusion pump. The forepump lowers pressure to less than 10 kPa into which the diffusion pump can exhaust its gas.[1]

gage pressure: Pressure above or below atmospheric pressure at the measurement location.[1,10]

gas ballast: Gas (air) admitted into the pumping chamber of a mechanical pump and inhibiting condensation of vapors in the chamber.[1]

getter: Reactive material that traps gas and removes it from a vacuum chamber. Several metals such as titanium, zirconium and tantalum can form getters for gases.[11]

halide: Compound of two or more elements, one of which is a halogen.[1,10]

halogen: Any of the nonmetallic elements — fluorine, chlorine, bromine and iodine — or any gaseous chemical component containing one or more of these elements.[10]

halogen detector probe test: Pressure leak test in which the leakage of a component, pressurized with a halogen rich mixture, is detected by scanning over the test object boundary surface with a probe connected to a halogen leak detector. Halogen gas is pulled from the leak through the probe inlet to the sensing element to cause a visible or audible signal on the indicator of the leak test instrument.[1,10]

halogen leak detector: Leak detector that responds to tracer gases containing halogen. Normally not very sensitive to the elemental halogen gases but very good when used with a gas that contains halogen. Also called *halogen sensitive leak detector* or *halide leak detector.*[1,10]

halogen standard leak: Standard leak in which the contained gas is a halogen tracer gas compound.[1,10]

heated immersion test: Bubble test where the heating causes buildup of internal pressure in a test object and the formation of bubbles at leak sites.[1]

helium: Monomolecular, noble gas with atomic weight of four, commonly used as tracer gas in leak testing. Because of helium's small molecular size and rarity (5 $\mu L \cdot L^{-1}$ in air) it is an excellent tracer gas.[1]

helium leak detector: Leak detector that responds to helium tracer gas.[1,10]

helium mass spectrometer leak detector: Mass spectrometer constructed to be peaked or tuned for response to helium gas.[10]

hermetic seal: Fusion seal that is leaktight.[1]

holes: Any void remaining in an object as a result of improper manufacturing processing. Often called *gas holes, cavities* or *air locks.*[2,10]

hood test: Quantitative leak test in which a test object under vacuum test is enclosed by a hood filled with tracer gas so as to subject all parts of the test object to examination for leakage at one time. A form of dynamic leak testing in which the entire enclosure or a large portion of its external surface is exposed to the tracer gas while the interior is connected to a leak detector, with the objective of detecting leakage or measuring its total rate.[1,10]

hot thermionic ionization gage: Absolute pressure gage that monitors ion current proportional to gas density at pressures less than 0.1 Pa (1 mtorr). Electrons produced by a heated filament (usually of tungsten or iridium and often thorium coated) ionize the gas and produce a positive ion current that flows to a wire collector. This current is proportional to gas density over the absolute pressure range below 100 mPa (1 mtorr) for a given gas composition.[1]

ideal gas: Gas that obeys the laws of thermodynamics for ideal gases. Also called perfect gas.[1,10]

immersion leak testing: Test object is pressurized and then submerged in detection fluid. The formation of bubbles from the object indicates a leak; the absence of bubbles indicates leaktightness.[1]

implosion: Collapse of pressure boundary or wall of a containment vessel or structure when evacuated and subject to atmospheric or higher external pressures.[1]

indication: Nondestructive testing discontinuity response that requires interpretation to determine its relevance. Compare *defect, discontinuity* and *false indication.*[8,10]

indication, discontinuity: Visible evidence of a material discontinuity. Subsequent interpretation is required to determine the significance of an indication.[2,10]

indication, false: Indication produced by something other than a discontinuity. Can arise from improper test procedures.[6,10]

indication, nonrelevant: Indication due to misapplied or improper testing. May also be an indication caused by an actual discontinuity that does not affect the usability of the object (a change of section, for instance).[2,10]

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indication, relevant: Indication from a discontinuity (as opposed to a nonrelevant indication) requiring evaluation by a qualified inspector, typically with reference to an acceptance standard, by virtue of the discontinuity's size or location.[8,10,16]

inert gas: Gas that does not readily combine with other substances. Examples are helium, neon and argon.[1,10]

infrared: Below red, referring to radiation of frequency lower than the color red. See *infrared radiation*.[9,10]

infrared and thermal testing: Nondestructive testing that uses heat or infrared radiation as interrogating energy.

infrared camera: Radiometer that collects infrared radiation to create an image.[9,10]

infrared radiation: Radiant energy below the color red, of wavelengths longer than 770 nm, between the visible and microwave regions of the electromagnetic spectrum.[8,9,10,17]

infrared thermography: See *thermography*.

inlet: Opening, flange, connection or coupling on a leak detector or leak testing system through which tracer gas may enter from a leak in a test object.[1,10]

integrated leakage rate test (ILRT): Leakage test performed for an entire system or component by pressurizing the system to the calculated peak containment internal pressure related to the design and determining the overall integrated leakage rate.[1,10]

interpretation: Determination of the significance of test indications from the standpoint of their relevance or nonrelevance. The determination of the cause of an indication or the evaluation of the significance of discontinuities from the standpoint of whether they are detrimental or inconsequential.[2,10]

ion current: Current that flows at all times from the positive emitter (heater) to the negative cathode collector of the heated anode (alkali ion) halogen vapor detector. This current increases in the presence of halogenated gases.[1,10]

ion pump: Pump that combines electric and magnetic fields to ionize gas and trap the gas inside the pump, thus removing it from the vacuum chamber.[11]

ionization gage: High vacuum gage that depends on the measuring of electrical current resulting from ionization of gas. Examples include thermionic ionization gages (Bayard-Alpert), cold cathode gages (Penning or Philip) and alphatron gages.[1]

IR: Infrared and thermal testing.

isobaric: Having constant barometric pressure.[1]

Knudsen number: Ratio of mean free path to characteristic dimension of the system.[11]

laminar flow: Class of viscous flow where velocity distribution of fluid in a cross section of a tube is parabolic.[1]

laser: Acronym (*l*ight *a*mplification by *s*timulated *e*mission of *r*adiation). The laser produces a highly monochromatic and coherent (spatial and temporal) beam of radiation. A steady oscillation of nearly a single electromagnetic mode is maintained in a volume of an active material bounded by highly reflecting surfaces, called a resonator. The frequency of oscillation varies according to the material used and the means of initially exciting or pumping the material.[8,10,17]

leak: Opening that allows the passage of a fluid.[1,10,18]

leak detector: Device for detecting, locating or measuring leakage.[1,10]

leak testing (LT): Nondestructive testing method for detecting, locating or measuring leaks or leakage in pressurized or evacuated systems or components.[1,10]

leakage: Measurable quantity of fluid escaping from a leak.[1,10]

leakage design basis accident: Calculated peak containment internal pressure related to the design basis accident.[1,10]

leakage rate: Quantity of leakage fluid per unit time that flows through a leak at a given temperature as a result of a specified pressure difference across the leak.[1,10] See *throughput*.

leaker penetrant: Penetrant especially designed for leak detection.[2]

leech box: Double compartmented box in which the outer compartment is evacuated and then the inner compartment is pressurized to produce a pressure differential across the test boundary under the inner compartment.[1]

level, acceptance: In contrast to rejection level, test level above or below which, depending on the test parameter, test objects are acceptable.[2,10]

level, rejection: Value established for a test signal above or below which, depending on the test parameter, test objects are rejectable or otherwise distinguished from the remaining objects.[2,10] See *level, acceptance*.

lid stiffness: In leak testing of hermetically sealed packages, the material characteristic that defines the amount of lid deflection from a specific pressure differential.

LT: Leak testing.

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magnetic sector: Permanent magnet that separates the ion species in the spectrometer tube of the helium mass spectrometer.[1]

manifold: Collection of vacuum hardware such a valves, piping and chambers connected together to form a test system.[1]

manometer: Instrument for measuring pressure (or pressure differentials) of gases and vapors.

manual zero: Control on a test instrument that allows the user to zero the instrument panel meter.[1,10]

masking: Covering of a portion of a test object so as to prevent tracer gas from entering leaks that may exist in the covered section.[1,10]

mass flow rate: Weight, moles or number of molecules passing through a system as function of time.[1]

mass spectrometer leak detector: Mass spectrometer with design factors optimized to produce an instrument that has high sensitivity to a single tracer gas.[1,10]

mass-to-charge ratio: Ratio of mass (kilogram) to electrical charge (coulomb) of a molecule,[1] or the atomic mass of the molecule divided by the atomic charge of the molecule.[11]

mean free path: Average distance a gas molecule travels between successive collisions with other molecules in the gas or vapor state.[1,10]

mechanical pump: Mechanical device with pumping fluid and seals that physically removes a portion of the gas from a system with each revolution of the armature. A mechanical pump can pump a chamber down to about 0.1 Pa (1 mtorr).[1]

micro: Prefix that divides a basic unit of measure by one million.[2,10]

mole: Molecular weight of a substance, in gram (gram mole).[1]

molecular flow: Phenomenon occurring when mean free path length of gas molecules is greater than the largest cross sectional dimension of a leak or the tube through which flow is occurring.[1,10]

molecular weight: For a gas, the mass of 22.4 L (0.8 ft^3) at standard conditions.[1]

motion feedthrough: Function provided by rotary or linear drives that penetrate the vacuum boundary to operate valves, pumps or perform other functions inside the vacuum system.[11]

NDC: Nondestructive characterization.

NDE: (1) Nondestructive evaluation. (2) Nondestructive examination.[8,10]

NDI: Nondestructive inspection.

NDT: Nondestructive testing.

nondestructive characterization (NDC): Branch of nondestructive testing concerned with the description and prediction of material properties and behaviors of components and systems.[10]

nondestructive evaluation (NDE): Another term for *nondestructive testing.* In research and academic communities, the word *evaluation* is often preferred because it emphasizes interpretation by knowledgeable personnel.[10]

nondestructive examination (NDE): Another term for *nondestructive testing.* In the utilities and nuclear industry, *examination* is sometimes preferred because *testing* can imply performance trials of pressure containment or power generation systems.[10]

nondestructive inspection (NDI): Another term for *nondestructive testing.* In some industries (utilities, aviation), the word *inspection* often implies maintenance for a component that has been in service.[10]

nondestructive testing (NDT): Determination of the physical condition of an object without affecting that object's ability to fulfill its intended function. Nondestructive testing techniques typically use a probing energy form to determine material properties or to indicate the presence of material discontinuities (surface, internal or concealed). See also *nondestructive evaluation, nondestructive examination* and *nondestructive inspection.*[10]

optical leak testing: Leak testing method using optical means such as holographic laser interferometry. Optical leak testing is used for microelectronic and pharmaceutical packaging.

outgassing: Forms of gas coming from material in a vacuum system. Includes gases adsorbed on the surface, dissolved in material and trapped in pockets and those due to evaporation.[1,10]

overall integrated leakage rate: Total leakage through all leakage paths including containment welds, valves, fittings and components that penetrate a primary reactor containment system, expressed in weight percent of contained air mass per day.[1,10]

partial pressure: Pressure a gas would exert if alone in a container.[1]

parts per million (ppm): Concentration of a specific gas in another gas or gas mixture. For example, a tracer gas concentration might be 10 ppm in air or nitrogen. The more specific term $\mu L \cdot L^{-1}$ is preferred, to indicate proportion by volume.[1,10]

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penetrant: Liquid capable of entering discontinuities open to the test surface and adapted to the penetrant test process by being made highly visible in small traces. Fluorescent penetrants fluoresce brightly under ultraviolet light and visible penetrants are intensely colored to be readily visible on developer backgrounds when illuminated with visible light.[2,10]

penetrant leak testing: Technique of penetrant testing in which the penetrant is applied to one surface of a test material while the opposite surface is tested for indications that would identify a leak or void passing through the material thickness.[2,10]

permeation: Passage of fluid into, through and out of a solid barrier having no holes large enough to permit more than a small fraction of molecules to pass through any one hole.[1]

Pirani gage: Bridge circuit that measures the effect of gas conductivity changes corresponding to pressure variations. Measures pressure from atmospheric down to 0.1 Pa (1 mtorr).[1]

pressure differential: Difference in pressure between two sides of a pressure boundary.[1]

pressure proof testing: Test of system at pressure considerably above the allowable working pressure to demonstrate structural capability.[1]

pressure testing: Technique of leak testing objects pressurized with a tracer gas with the subsequent detection and location of any existing leaks with a sampling probe (a qualitative test). Tests performed by increasing the pressure inside a test boundary to a level greater than the surrounding atmosphere and detecting leakage by systematic examination of the outside of the test surface. Leaks are located at time of detection; however, it is impossible to accurately determine a total leakage rate for the object being pressure tested.[1,10]

probe: In leak testing, the physical means for sensing a gaseous leak, typically a tube having a fine opening at one end, used for directing or collecting a stream of tracer gas. Detector probes are used for pressure testing and tracer probes are used for vacuum testing.[1,10]

probe gas: Tracer gas that issues from a fine orifice in a tracer probe so as to impinge on a restricted (small) test area.[1,10]

process control: Application of quality control principles to the management of a repeated process.[8,10]

process testing: Initial product testing to establish correct manufacturing procedures and then by periodic tests to ensure that the process continues to operate correctly.[2,10]

proportioning probe: Probe that can vary the tracer gas concentration in the sample at the sensor, typically by mixing pure air with sample gas from the probe inlet port. Ratios of mixture between 100 percent pure air (obtained from an outdoors source or by filtering ambient air through charcoal) and 100 percent leak sample gas are attainable without great changes in total flow from the probe. The proportioning probe used in halogen leak testing lets the user operate in an atmosphere with up to 1000 $\mu L \cdot L^{-1}$ tracer gas background contamination. It proportions the amount of atmosphere allowed to enter the probe with its own (recirculating) fresh air supply.[1,10]

pumping speed: Volumetric speed at which gas is transported, expressed in cubic meter per second.[11]

pure air supply: In leak testing, air that has been cleaned of halogen contamination by means of an activated charcoal filter. This term is sometimes also used to describe any nonreactive gas, such as nitrogen, that contains no halogen contamination and to which the leak detector is not sensitive.[1,10]

qualification: Process of demonstrating that an individual has the required amount and the required type of training, experience, knowledge and capabilities. See also *qualified*.[8,10]

qualified: Having demonstrated the required amount and the required type of training, experience, knowledge and abilities. See also *qualification*.[8,10]

quality: Ability of a process or product to meet specifications or to meet the expectations of its users in terms of efficiency, appearance, longevity and ergonomics.[8,10]

quality assurance: Administrative actions that specify, enforce and verify a quality program.[8,10]

quality control: Physical and administrative actions required to ensure compliance with the quality assurance program. May include nondestructive testing in the manufacturing cycle.[8,10]

quartz Bourdon tube gage: High precision pressure measuring instrument containing a quartz helical Bourdon tube.[1,10]

radioactivity leak test: Leak test using radioactive tracer gas such as krypton-85, detected by its radioactivity.[1]

recommended practice: Set of guidelines or recommendations.[8,10]

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Recommended Practice SNT-TC-1A: See *ASNT Recommended Practice No. SNT-TC-1A.*

reference standard leak: *Calibrated leak* for reference purposes. See also *standard.*

relevant indication: See *indication, relevant.*

repeatability: Ability to reproduce a detectable indication in separate processings and tests from a constant source.[1,2,10]

response time: Time required for a leak detector signal to reach a specified value after the application of a step input.[1,11,19] The signal reaches 63 percent of final value in one time constant.

response factor: Response of a halogen leak detector to 3×10^{-7} Pa·m^3·s^{-1} (3×10^{-6} std cm^3·s^{-1}) of tracer refrigerant-12 or less, divided by the response to the same quantity of another tracer gas. Thus, the actual leakage rate of a detected leak will equal the indication of the detector multiplied by the response factor of the specific halogen tracer gas used. The response factor of a mixture of tracer and nontracer gases will be the response factor of the tracer divided by the fraction of tracer gas in the test gas (by volume).[1,10]

Reynolds number: Number expressing the relative quantity of gas flowing in a pipe.[11]

roots blower: Blower that uses two lobed rotors mounted on parallel shafts in conjunction with mechanical pumps to obtain greater pumping speeds and lower pressures.[11]

rotameter: Meter that uses a float and a tapered glass bore to measure flow.[11]

sampling probe: See *detector probe.*

sampling, partial: Testing of less than one hundred percent of a production lot.[8,10]

sampling, random partial: Partial sampling that is fully random.[8,10]

sampling, specified partial: Partial sampling in which a particular frequency or sequence of sample selection is prescribed. An example of specified partial sampling is the testing of every fifth unit.[8,10]

sensitivity: Measure of a sensor's ability to detect small signals. Limited by the signal-to-noise ratio.[7,10]

sensitivity of leak detector: Response of a leak detector to tracer gas leakage (typically panel meter pointer deflection in scale divisions; leak sensitivity is measured in units of Pa·m^3·s^{-1} or std cm^3·s^{-1}).[1,10]

sensitivity of leak test: Smallest leakage rate that an instrument, technique or system can detect under specified conditions (implies minimum detectable leakage rate).[1,10]

SI: International System of Units (*Le Systeme Internationale d' Unites*), a system of measurement based on seven units: meter (m), kilogram (kg), second (s), kelvin (K), ampere (A), candela (cd) and mole (mol).[4,10,20]

signal: Response containing relevant information.[4,10,11]

sniffer probe: See *detector probe.*

sniffer test: See *detector probe test.*

SNT-TC-1A: See *ASNT Recommended Practice No. SNT-TC-1A.*

soak time: In leak testing, the period of time between when the system or component reaches test pressure and either when the leak detector solution is applied to the surface or when the leak detector is used to scan that surface.[21]

solution film: Thin continuous film of bubble solution used in bubble testing.[11]

sorption pump: Pump consisting of a sieve and liquid nitrogen with ability to pump to 0.1 Pa (1 mtorr).[11]

specification: Set of instructions or standards invoked by a specific customer to govern the results or performance of a specific set of tasks or products.[8,10]

spectrometer: In the helium mass spectrometer, the basic device that sorts the charged gaseous particles by species in accordance with molecular weight.[1]

standard: (1) Physical object with known material characteristics used as a basis for comparison or calibration; *reference standard*. (2) Concept established by authority, custom or agreement to serve as a model or rule in the measurement of quantity or the establishment of a practice or procedure.[7,22] (3) Document to control and govern practices in an industry or application, applied on a national or international basis and usually produced by consensus. See also *acceptance standard* and *working standard.*[4,8,10,11]

standard atmospheric conditions: Atmospheric pressure of 101.325 kPa (14.6959 lb_f·in.$^{-2}$). Temperature of 0 °C (273 K, 32 °F or 492 °R). The density of dry air at these conditions is 0.120 41 kg·m^{-3} (0.075 17 lb·ft^{-3}) at sea level.[1,10]

standard barometric pressure at sea level: See *atmospheric pressure.*

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standard leak: Device that permits a tracer gas to be introduced into a leak detector or a leak testing system at a known rate to facilitate tuneup and calibration of the leak detector or test system.[1,10]

standard leakage rate: In optical leak testing of hermetically sealed packages, the quantity of dry air at 25 °C (77 °F) flowing (in atmospheric $cm^3 \cdot s^{-1}$) through a leak or multiple leak paths when the high pressure side is at 100 kPa (1 atm or 760 torr absolute) and the low pressure side is at pressure not greater than 100 Pa (1 torr absolute).[23] An *equivalent standard leakage rate* of a given sealed package, with a measured leakage rate, is the leakage rate, of the same package with the same leak geometry, that would exist under the standard leakage rate conditions.

static testing: See *accumulation test technique.*

structural integrity test (SIT): Test that demonstrates the capability of a vessel to withstand specified internal pressure loads.[1,10]

surface tension: Characteristic of liquids where the outer surface contracts to the smallest possible area.[1]

temperature: Measure of the intensity of particle motion in degrees celsius (°C) or degrees fahrenheit (°F) or, in the absolute scale, kelvin (K) or degrees rankine (°R), where increment of 1 K = 1 °C = 1.8 °R = 1.8 °F. Compare *heat.*[9,10]

Tesla coil: High voltage spark coil (several thousand volt).[1]

test piece: Part subjected to testing.[10]

test quality level: See *level, rejection.*

test ring: Ring specimen typically made of tool steel, containing artificial subsurface discontinuities used to evaluate and compare the performance and sensitivity of magnetic particles.[6,10,12]

test surface: Exposed surface of test object.[2,7,10]

thermal: Physical phenomenon of heat involving the movement of molecules. Compare *infrared radiation.*[9,10]

thermal conductivity: Heat transfer capability, as of a gas.[1]

thermal conductivity vacuum gage: Instrument that operates on principle that as gas molecules are removed from a system, the amount of heat transfer by conduction is reduced. This relationship is used to indicate absolute pressure.[1,10]

thermal disorption: Release of gases or vapors from the interior wall of a vacuum system by heat.[11]

thermal equilibrium: Condition of an object wherein temperatures throughout the object remain constant.[9,10]

thermocouple gage: Device that incorporates a thermocouple to measure gas conductivity changes corresponding to pressure variations from 0.1 Pa (1 mtorr) to atmospheric pressure (100 kPa or 760 torr).[1]

thermography: Imaging or viewing of object or process through sensing of *infrared radiation* emitted by it. Temperature patterns on the material surface produce corresponding radiation patterns. Thus, heat flow by both conduction and radiation may be observed and used to locate material discontinuities.[9,10]

throughput: Quantity of gas, or total number of molecules at a specific temperature, passing a section of a vacuum system per unit of time. See *leakage rate.*[1,10]

torr: Unit of pressure nearly equal to 133.322 Pa (1.000 mm Hg).[1,10]

tracer: In leak testing, a gas that is sensed as it escapes from confinement.[1,10]

tracer gas: Gas that can be detected by a specific leak detector and thus disclose the presence of a leak in a system. Also called *search gas.*[1,10]

tracer probe test: Leak test in which a tracer gas is applied by means of a probe to an accessible test surface on an evacuated test object so that the area covered by the tracer gas is localized. A leak detector in the line to the vacuum pump enables individual leaks to be located when they admit tracer gas.[1,10]

tracer standard leak: Standard leak in which the contained gas is a tracer gas compound.[1,10]

transition flow: Phenomenon that occurs when the mean free path of gas is about equal to the cross sectional dimension of a leak or the tube through which flow is occurring.[1,10]

trap: *Cold trap.*

turbomolecular pump: Molecular turbine that drives gas out of a vacuum chamber, achieving a high vacuum pressure in the 10 nPa (0.1 ntorr) range.[11]

ultimate pressure: Lowest pressure that can be achieved in a vacuum chamber after cleaning and baking.[11]

ultrasonic: Pertaining to acoustic vibration frequencies greater than about 20 kHz.[7,10,21]

ultrasound leak test: Leak test that detects ultrasound in the 40 kHz range from gas flowing through the leak path.[1]

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ultraviolet radiation: Electromagnetic radiation or light energy in the near ultraviolet range with wavelengths from 320 to 400 nm, just below the wavelengths of visible light. Also a term for the ultraviolet light source used in fluorescent nondestructive testing. Sources often have a predominant wavelength of 365 nm.[2,6,8,10,13]

upper confidence limit: Calculated value constructed from sample data with the intention of placing a statistical upper boundary on a true leakage rate.[1,10]

vacuum: Space containing gas at a pressure below atmospheric pressure.[1,10]

vacuum box: Device used to create a differential pressure over an isolated area of a weld or of a pressure boundary that cannot be directly pressurized.[1,10]

vacuum box leak testing: Technique of bubble testing where a vacuum box is used to create a pressure differential across a boundary. A viewing window allows observation of bubble formation.[1]

vacuum grease: Substance commonly used to attain a seal and to lubricate devices such as stopcocks and moving boundary penetrations.[1]

vacuum pressure testing: Leak testing procedure in which the test object containing tracer gas is placed in an evacuated enclosure and the tracer gas is detected after entering the enclosure.[1,10] Also called *bell jar testing*.

vacuum testing: Method of testing for leaks in which the object under test is evacuated and the tracer gas is applied to the outside surface of the test object.[10]

vapor: Gaseous form of, for instance, water or oil.[1]

vapor pressure: Pressure exerted by the vapor of a liquid when in equilibrium with the surface of the liquid at a specified temperature. These limiting pressures can restrict the levels of pressurization of enclosures with these tracer gases during pressure leak testing and can also limit the vacuum obtainable in presence of these liquids (for example, water or solvents).[1,2,10]

variable standard leak: Device that permits a tracer gas to be introduced to the leak detector at a rate adjustable by the operator.[1,10]

vent: Valve in a vacuum system for letting air into a vacuum chamber.[1]

verification test: Tests intended to confirm the capability of leak test technique and equipment to determine leakage rate.[1,10]

virtual leak: Emission of gas in a vacuum system that results from condensible or trapped gases. They gradually evaporate from surfaces or escape from pockets, raising the absolute pressure in the same manner as a real leak.[1,10]

viscosity: Coherent characteristic of fluids that causes resistance to flow.[1]

viscous flow: Flow of gas or gas mixtures through a leak or duct under conditions such that the mean free path is smaller than the cross section of the leak or opening. Viscous flow may be either laminar or turbulent and is most likely to occur during leak tests at atmospheric or higher pressures. With vacuum conditions, the flow of tracer gases to the leak detector element is usually by diffusion, resulting in slow response to leaks being probed by a tracer jet.[1,10]

working standard: Workpiece or energy source calibrated and used in place of expensive reference standards. In the calibrating of photometers, the standard would be a light source.[8,10]

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CHAPTER 17

Leak Testing Bibliography

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Introduction

This bibliography lists published works cited in the references at the end of chapters, as well as other works not cited elsewhere in this volume. A listing in this bibliography is not to be construed as any sort of endorsement or recommendation of the technique, service or equipment described.

The bibliography is divided into sections, and a published work is generally cited only once. The reader therefore is urged to look in more than one section of the bibliography. A publication on an acoustic technique, for example, may be found under *Acoustic Leak Testing* or *Standards and Practices* or *Aboveground Storage Tanks* but not under more than one of those headings.

The bibliography headings are the following.

General Works
  Engineering
  Measurement Units
  Nondestructive Testing
Leak Testing, General and Miscellaneous
Leak Testing Methods
  Acoustic Leak Testing
  Bubble Testing
  Helium Leak Testing
  Optical Leak Testing
  Thermographic Leak Testing
Leak Testing Safety
Standards and Practices
Leak Testing of Storage Systems
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