

ANSI/AWS A4.3-93
An American National Standard

**Standard Methods for
Determination of the Diffusible
Hydrogen Content of Martensitic,
Bainitic, and Ferritic Steel Weld Metal
Produced by Arc Welding**



American Welding Society

Keywords — Diffusible hydrogen, gas chromatography, mercury displacement, arc welding, steel, shielded metal arc welding, gas metal arc welding, flux cored arc welding, submerged arc welding

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Standard Methods for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic, and Ferritic Steel Weld Metal Produced by Arc Welding

Superseding ANSI/AWS A4.3-86

Prepared by
AWS Committee on Filler Metal

Under the direction of
AWS Technical Activities Committee

Approved by
AWS Board of Directors

Abstract

A standard 25 x 12 x 80 mm test specimen and method of preparation are set forth, along with two standard methods of diffusible hydrogen analysis, mercury displacement and gas chromatography. The methods are suitable for shielded metal arc welding, gas metal arc welding, flux cored arc welding, and submerged arc welding using welding conditions and electrodes given in several applicable American Welding Society filler metal specifications.



American Welding Society

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Foreword

(This Foreword is not a part of ANSI/AWS A4.3-93, *Standard Methods for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic and Ferritic Steel Weld Metal Produced by Arc Welding*, but is included for information only.)

A number of test methods for determining weld metal diffusible hydrogen have been used over the years. In particular, media for collecting hydrogen have included glycerin, paraffin, and mercury, with glycerin being by far the most popular in the United States. A previous AWS A5 Task Group, chaired by R. A. LaFave, investigated the collection of hydrogen over glycerin in 1982-1983. That Task Group concluded that, whereas collection of hydrogen over glycerin is simple and inexpensive, the results suffer from severe variability. This is due in part to the solubility of water, atmospheric gases, and hydrogen in glycerin.

Collection of hydrogen over glycerin, when using very low hydrogen weld metals, has also been shown to give null readings, while collection over mercury using the method of ISO 3690 was giving readings greater than zero. The method of ISO 3690 is only suitable for coated electrodes. Recognizing this, the International Institute of Welding (IIW) has been developing a draft standard with a larger specimen size suitable for other welding processes but still using collection of hydrogen over mercury as the reference method. This draft has also introduced analysis by gas chromatography as giving results equal to those of collection over mercury.

The AWS A5 Task Group, in 1983 and 1984, chaired by D. J. Kotecki, considered adopting the method of the IIW draft standard. However, after round robin testing employing the IIW specimen size and other sizes, the Task Group rejected the IIW specimen size for two reasons. First, the small size required reorientation of the test specimen depending upon welding heat input. Second, the small size resulted in hydrogen volumes almost too small to measure when very low hydrogen weld metals were tested. As a result, the Task Group selected a specimen size appropriate to generating a significant volume of hydrogen gas for measurement and not requiring specimen reorientation for welding heat input within the realm of electrode classifications envisioned for use with this standard.

This is the first revision of this specification as shown below:

ANSI/AWS A4.3-86, *Standard Methods for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic, and Ferritic Steel Weld Metal Produced by Arc Welding*.

Comments and suggestions for the improvement of this standard are welcome. They should be sent to the Managing Director, Technical Services Division, American Welding Society, 550 N.W. LeJeune Road, P.O. Box 351040, Miami, Florida 33135. Official interpretations of any of the technical requirements of this standard may be obtained by sending a request, in writing, to the Managing Director, Technical Services Division, American Welding Society. A formal reply will be issued after it has been reviewed by the appropriate personnel following established procedures.

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Standard Methods for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic, and Ferritic Steel Weld Metal Produced by Arc Welding

1. Scope

This standard prescribes a standard weld test assembly, a standard method of test specimen preparation, and two standard methods of analysis for determination of diffusible hydrogen from martensitic, bainitic, and ferritic steel weld metals. The methods of preparation are suitable for shielded metal arc, gas metal arc, flux cored arc, and submerged arc welding processes. Extension of the methods of preparation to other processes, such as gas tungsten arc or plasma arc welding, are possible.

It is not the intent of this standard directly herein to classify arc welding electrodes, fluxes, and gases according to the hydrogen content of welds produced from them. However, it is the intent of this standard that it be used as the standard test method for classification purposes to be referenced in individual filler metal specifications prepared by the AWS Committee on Filler Metal and its subcommittees.

In addition to its use for electrode classification purposes, it is the intent of this standard that it be used for quality conformance testing of arc welding electrodes, fluxes, and gases.

It is recommended that this standard be used for developing and reporting research results so that the results may be directly compared with those from other laboratories.

For purposes of electrode classification and quality conformance testing, any requirement established based on the use of this standard must also be based on a reference atmospheric condition. If a reference condition is not specified in conjunction with diffusible hydrogen requirements in the individual consumable standards, the recommended reference condition in 6.6 shall be used.

2. Units of Measure

At the present time, U.S. customary units of measurement are normally the primary units of AWS documents, including the standards and specifications prepared by the Committee on Filler Metal. However, these units are awkward for expressing hydrogen values, whereas the S.I. units are not. Furthermore, the practice of reporting diffusible hydrogen values in S.I. units is practically universal, including within the United States.

Therefore, the S.I. units of measurement are chosen as the primary units of measurement for this standard, except for welding parameters specified by filler metal specifications. U.S. customary units are included parenthetically, except for diffusible hydrogen values and for measurements directly used in computing diffusible hydrogen values. Only S.I. units are, in practice, used for diffusible hydrogen values and for measurements directly used in computing them.

3. Preparation of Weld Test Assemblies

3.1 Test Assembly Dimensions. Each weld test assembly shall consist of a starting weld tab, a test specimen at the center, and a run-off weld tab, all held in a copper clamping fixture (Figures 1A, 1B, and 2). Four such weld test assemblies shall constitute a complete test. The material for all three pieces of a weld test assembly shall be nonrimming quality steel of grade ASTM A36 or SAE 1020. In case of dispute, ASTM A36 steel shall be used as referee material. All three pieces for a weld test assembly shall have a cross section of 25 mm (wide)

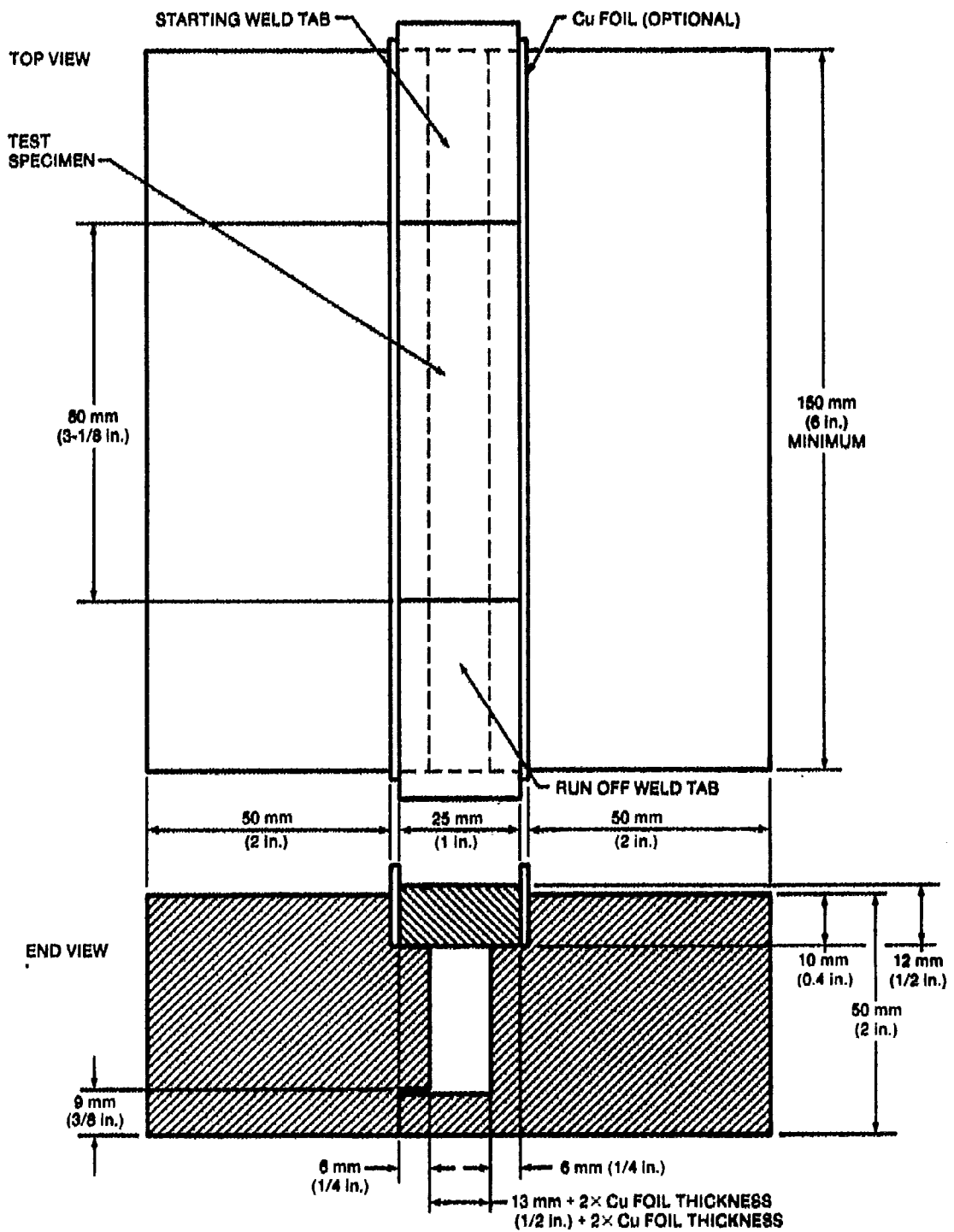


Figure 1A — Weld Test Assembly in Suggested Copper Clamping Fixture

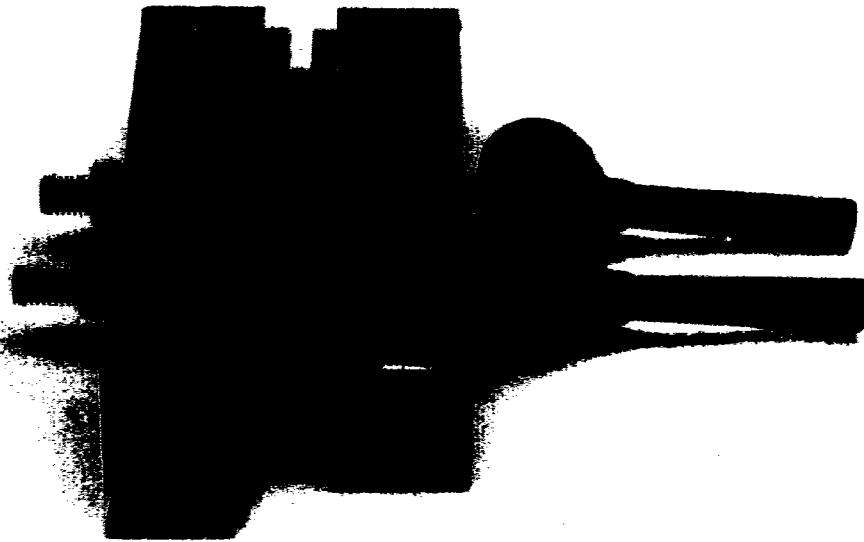


Figure 1B — Alternate Copper Clamping Fixture

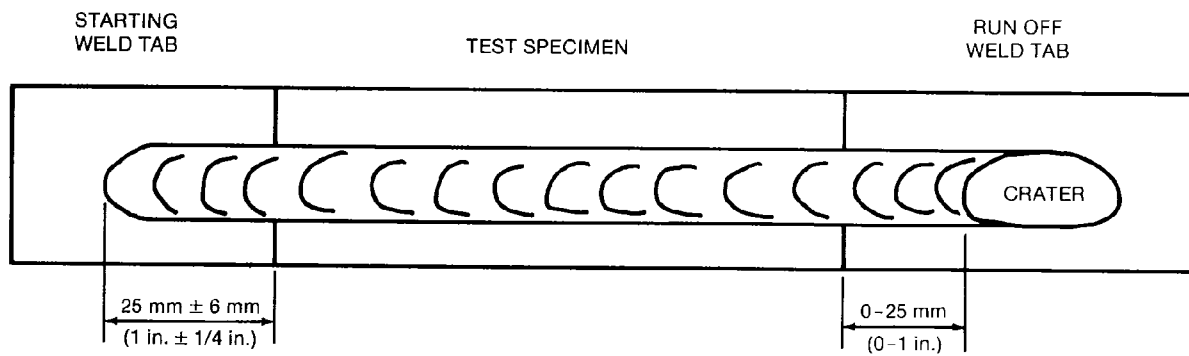


Figure 2 — Weld Test Assembly After Welding, with Correct Dimensions for Weld Bead Length on the Weld Tabs

x 12 mm (thick) ± 2 mm (1 x 1/2 in. $\pm 1/16$ in.) with perpendicular edges. The width of the three pieces (25 mm dimension) shall be sufficiently uniform that a copper alignment clamp will hold all three parts of the weld test assembly firmly. A convenient base metal form is hot-rolled bar stock of this same cross section.

The length of the center test specimen shall be 80 mm ± 5 mm (3-1/8 in. $\pm 1/4$ in.). Instead of one piece of 80 mm (3-1/8 in.) length, two pieces of 40 mm ± 2.5 mm (1-9/16 $\pm 1/8$ in.) can be substituted. These pieces shall be analyzed separately and the individual results combined to represent one of the four required results. The lengths of the weld tabs may be arbitrarily chosen such that the lengths of the weld bead on the starting weld tab and on the run-off weld tab comply with the requirement of 5.2.

3.2 Test Assembly Degassing. The pieces for a weld test assembly shall be heat-treated for one hour minimum in air, vacuum, or inert gas at 400° to 650°C (750° to 1200°F) to remove any hydrogen present in the material. In cases of dispute, the referee heat-treatment temperature shall be 625°C ± 25 °C (1150°F ± 50 °F). The rate of heating and cooling is unimportant.

3.3 Test Assembly Cleaning and Weighing. If the pieces were machined to size prior to heat treatment such that no scaled surface remained, and if the heat-treatment atmosphere was such that no surface scale formed, then no subsequent descaling shall be required. If the surface is scaled after heat treatment, the surface shall be completely descaled by dry shot blasting, dry grit blasting, dry belt sanding, or dry power wire brushing. (*Caution: Contamination from this point on may lead to false high values for the test. The exhaust from compressed air tools may contain lubricants which could cause contamination.*)

After heat treatment and descaling, the test specimen identification shall be marked by stamping or engraving any surface which will not be welded. If an upset is formed by this operation which interferes with contact with the copper clamping fixture, the upset shall be removed by dry sanding. Use of paints or other such markers shall not be permitted.

After marking of the test specimen and upset removal, the test specimen shall be weighed to the nearest 0.1 g or less. This weight shall be recorded and becomes part of the analysis in 6.5.

Immediately prior to welding, the marked specimen and the weld tabs shall be degreased in a non-oil bearing, residue-free fluid such as alcohol or acetone. From this point until completion of welding, the test specimen and weld tabs shall be handled only with clean tongs, clean lint-free gloves, or other contaminant-free means. Follow safe handling practices with alcohol and acetone and keep them away from the welding arc. They are flammable.

If gloves are used during the final degreasing of the test pieces, the chemical resistance of the gloves to the cleaning solvent must be ensured to preclude contamination of the specimens.

4. Welding Fixture

A copper clamping fixture shall be used during welding to hold the weld test assembly in alignment, with the test specimen and weld tabs in firm contact, and to serve as a heat sink.

The copper clamping fixture shall accept clamping pressure from the sides of the weld test assembly. It shall also permit rapid release and removal of the weld test assembly for quenching with a minimum of delay. Suggested copper clamping fixtures are shown in Figures 1A and 1B, with a weld test assembly in place. Also shown in Figure 1A is use of soft, clean copper foil (2 mm [.080 in.] maximum thickness) between the weld test assembly and the fixture. The use of copper foil is optional. It can serve to protect the copper clamping fixture from errant arc strikes and can be used to contain flux during submerged arc welding. Clamping pressure shall be applied during welding to hold the pieces of the weld test assembly firmly in contact with one another and with the copper clamping fixture (possibly through the copper foil). Quick-release devices such as pneumatic or spring-loaded clamps and standard vises have been successfully used with clamping fixtures, such as shown in Figure 1B.

The copper clamping fixture sketched in Figure 1A is adequate for shielded metal arc welding, as well as for gas metal arc or flux cored arc welding with small diameter electrodes. For submerged arc welding and for flux cored or gas metal arc welding with larger diameter electrodes (e.g., 2.4 mm [3/32 in.] and larger), a longer fixture may be necessary to accommodate a longer run-off weld tab so that the weld crater can comply with the requirements of 5.2. It is recommended that the weld tabs extend a short distance beyond the ends of the copper clamping fixture to facilitate rapid removal of the completed weld test assembly.

The copper clamping fixture shall be cool enough to be held with a bare hand before each weld test assembly is begun. Water cooling of the fixture by immersion after each test weld is acceptable, or water may be piped through the fixture to cool it continuously. In either case, care shall be taken that the fixture is free of condensation or moisture when welding. Immersion of the fixture in conjunction with the specimen water quench shall not be permitted, i.e., the specimen shall first be removed and quenched in its iced water bath, then the fixture may be cooled separately.

5. Welding and Preparation for Analysis

5.1 Preliminary Preparation. Prior to welding a set of four weld test assemblies, all welding parameter settings shall be adjusted to the desired levels for the test. The four weld test assemblies shall be welded with no alteration of parameter settings from the first through the fourth weld test assembly. These parameter settings shall be recorded and shall become a part of the record of the test, along with measurements made during and after the test. A data sheet suitable for recording the data is shown in Figure 3.

For classification of electrodes, fluxes, and gases according to diffusible hydrogen content of the deposited metal, the welding conditions (current, voltage, etc.) shall duplicate those used for preparing the weld test assembly used for the classification tensile test specimen required by the applicable AWS filler metal specification. However, a stringer bead technique shall be used (no weave) with weld travel speed appropriate to the stringer bead technique, even if a weave was used in preparing the weld test assembly for the tensile test specimen. For purposes other than classification, selection of welding parameters shall be as agreed among the parties concerned with the test results.

5.2 Welding the Test Assemblies. The arc shall be initiated on the starting weld tab at a point such that the starting edge of the deposit shall be $25 \text{ mm} \pm 6 \text{ mm}$ (1 in. $\pm 1/4$ in.) from the edge of the test specimen (see Figure 2). Welding shall proceed in a uniform, uninterrupted manner along the weld test assembly to the run-off weld tab, and shall be terminated on the run-off weld tab at a position such that the back edge of the crater is on the run-off weld tab, but within 25 mm (1 in.) of the edge of the test specimen. See Figure 2 for clarity. Decrease of travel speed for crater filling or other purposes shall not be permitted.

For submerged arc weld tests, flux which was used to cover one weld test assembly shall not be used in preparation of another, without regard as to whether that flux was fused during welding or not.

The weld test assembly shall be released from the copper clamping fixture and plunged into iced water within 5 seconds of extinguishing the arc. The weld test assembly shall be agitated vigorously in the iced water for 20 to 30 seconds and then quickly transferred to a low-temperature liquid bath (-60°C [-76°F] or colder) for storage. Ice shall remain in the iced water bath when the weld test assembly is withdrawn. Water may be removed from the weld test assembly prior to placing into the low-temperature liquid bath, provided that the water removal method does not reheat the weld test assembly, and provided that the weld test assembly

enters the low-temperature liquid bath within 60 seconds of extinguishing the arc.

All four weld test assemblies shall be welded within 60 minutes of beginning the first weld test assembly in the set. Each shall be stored in the low-temperature liquid bath before proceeding further.

5.3 Cleaning and Preparation for Analysis. After a test assembly has been in the low-temperature liquid bath for at least two minutes, that weld test assembly may be removed from the low-temperature bath for further processing. The maximum time out of the bath for any given weld test assembly shall be one minute, followed by at least two minutes back in the bath, which cycle may be repeated as necessary until processing is completed. During processing, the test specimen shall not be allowed to exceed 0°C (32°F).

Processing of the weld test assemblies to ready them for analysis shall include removal of any adherent slag (including slag islands on gas metal arc weld beads), power wire brushing all surfaces of the test specimen with a clean carbon steel brush to remove oxide, measurement and recording of the starting bead length and run-off bead length (see Figure 2), and breaking off the weld tabs. After the weld tabs have been broken off and the weld bead lengths on them recorded, the pieces may be discarded. If the starting or run-off bead length fails to comply with the requirements of Figure 2, the test specimen shall be discarded and a replacement weld test assembly shall be prepared. If this cannot be completed within 60 minutes after beginning welding the first test assembly in the set, all weld test assemblies for that set shall be discarded and the test shall be repeated.

Once processing is complete, the test specimen can be loaded into the analytical apparatus or can be returned to the low-temperature liquid bath for storage or shipment to the analytical apparatus. The storage time for a test specimen shall be no longer than 72 hours (3 days) at -60°C (-76°F), nor 500 hours (21 days) at -196°C (-320°F), before loading into the analytical apparatus.

6. Diffusible Hydrogen Analysis

6.1 Analytical Apparatus Requirements. The procedures outlined in Sections 3, 4, and 5 will result in a standard test specimen size prepared under standard conditions, and therefore delivering to the analytical apparatus a certain quantity of diffusible hydrogen, albeit with a certain degree of statistical scatter. It shall be the requirement of the analytical apparatus and analytical procedure that it collect and measure at least 90 percent of the total diffusible hydrogen within a test specimen delivered to the apparatus. In particular, this requirement precludes the use of analytical apparatus employing

WELDING DATA SHEET

Test identification number _____ Date of welding _____

Investigators _____

Welding process _____ Electrode classification _____

Electrode brand _____ Lot, control or heat No. _____

Electrode treatment after opening package _____

Electrode diameter _____ Coating moisture _____

Flux or gas shielding _____

Flux or gas lot, control, or heat No. _____

Treatment of flux or gas _____

Flux particle size _____ Flux depth _____

Gas dew point _____ Gas flow rate _____

Dry bulb temperature at time of welding _____

Wet bulb temperature at time of welding _____

Welding current _____ Welding voltage _____

Electrode polarity _____ Electrode extension _____

Welding travel speed _____ Wire feed speed _____

Welding heat input _____

Fixture water cooled _____ Copper foil used _____

Test specimen number				
Weight after welding, g				
Weight before welding, g				
Deposit weight, g				
Arc time, sec.				
Electrode stub length, mm				
Starting bead length, mm				
Run-off bead length ^(a) , mm				
Total bead length ^(a) , mm				

^(a) Excludes crater, see Figure 2.

Remarks:

Figure 3 — Welding Data Record Sheet

displacement of glycerin, paraffin, or silicone oil because of the degree of hydrogen solubility in these fluids. The degassing must take place in an inert atmosphere or environment in order to prevent loss or gain of hydrogen through reaction.

Two types of analytical apparatus are known which meet the above requirement. One is a mercury-filled eudiometer, and the other is a gas chromatograph. Both types of apparatus require loading of the test specimen into an isolation chamber and allowing the hydrogen to diffuse out of the specimen, usually at a temperature somewhat above room temperature, into the isolation chamber where it recombines into molecular hydrogen. After evolution of diffusible hydrogen is virtually complete, the volume of hydrogen collected is measured. Whichever analytical apparatus is employed, it shall be accurately readable to 0.02 ml of hydrogen gas or less.

In general, the isolation chamber used for collection must be gas-tight and fabricated of materials which preclude loss of hydrogen collected via diffusion during the time at test temperature.

6.2 Loading the Test Specimen into the Analytical Apparatus. If the hydrogen analysis results are not affected by moisture or liquid from the low-temperature bath adhering to the test specimen, the test specimen may be loaded directly from the bath into the analytical apparatus. Some gas chromatographs can be used in this way. It is the responsibility of the user to determine from the instrument manufacturer if contaminants on the test specimen at this point affect the results. Specimens shall not be loaded if covered with heavy frost.

For mercury displacement and other apparatus from which results could be affected by moisture or contamination from the low-temperature liquid bath, the specimen shall be cleaned of moisture and other contaminants by agitating in cool water until the resulting ice skin melts. This raises the test specimen temperature to slightly above 0°C (32°F). A quick rinse in a residue-free solvent shall be employed if the low-temperature bath liquid is not water soluble, and may be employed if it is water soluble. In either case, the specimen shall then be wiped dry or blown dry with cool air and immediately loaded into the isolation chamber.

If the analytical apparatus is a gas chromatograph, the required purge gas shall then be vented into the isolation chamber. If the analytical apparatus is a mercury eudiometer, the mercury shall then be drawn up into the eudiometer over the test specimen so that mercury entirely fills the eudiometer to the valve at the top, which is then closed, as described in 7.2. The time from the test specimen reaching 0°C (32°F) to sealing of the purged isolation chamber or eudiometer shall not exceed 150 seconds. See Figure 4 for a sketch of a suitable eudiometer for mercury displacement measurements.

6.3 Hydrogen Evolution and Analysis. The test specimen confined within its isolation chamber shall then be held at the hydrogen evolution temperature for a period of time necessary to assure diffusion of at least 90 percent of the diffusible hydrogen out of the test specimen. Table 1 lists minimum times at corresponding temperatures which have been determined to meet this diffusion requirement.

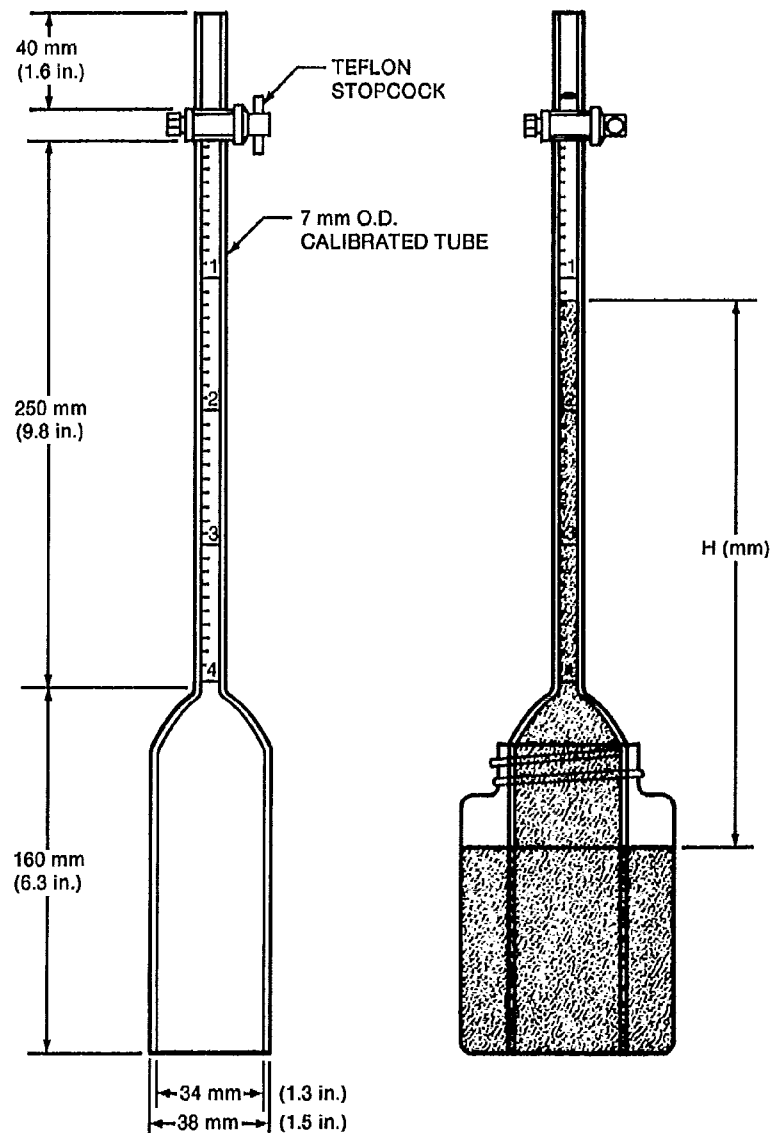
After the required hydrogen evolution period determined from Table 1, the hydrogen volume evolved from each test specimen individually shall be measured. In the case of analysis by mercury displacement, the procedure for analysis will, of necessity, depend upon the specifics of the glassware and mercury handling. One suitable glassware apparatus and procedure for test specimen handling and analysis are detailed in Section 7, Standard Mercury Displacement Method. For analysis by gas chromatography, see Section 8, Standard Gas Chromatography Method.

6.4 Variants of Hydrogen Evolution. If a hydrogen evolution time/temperature combination other than those given in Table 1 is used, it shall be the responsibility of the user to verify that no hydrogen is being lost, at least 90 percent of the diffusible hydrogen initially present in the test specimen is being measured, and nondiffusible hydrogen is not being released. This can only be done by comparing results with those obtained using the mercury displacement method detailed in Section 7 or the gas chromatography method of Section 8, using one of the standard hydrogen evolution time/temperature combinations given in Table 1. The comparison shall be made at two diffusible hydrogen levels: less than 5 ml/100 g, and more than 7 but less than 15 ml/100 g. Each comparison shall consist of eight test specimens prepared at one time under identical conditions.

Four test specimens, randomly selected, shall be analyzed by gas chromatography or by the Standard Mercury Displacement Method detailed in Section 7, using a time/temperature combination from Table 1, and the remainder shall be analyzed by gas chromatography or mercury displacement using the variant time/temperature combination. For each comparison, the averaged results of the two sets of test specimens shall agree within 1 ml/100 g.

Table 1
Minimum Hydrogen Diffusion Times
at Acceptable Temperatures

Diffusion temperature, °C (°F)		
± 3°C	(± 5°F)	Minimum Diffusion Time, hrs.
45	(113)	72
150	(302)	6



Sample Calculation (see 7.3): Suppose the barometric pressure P is 745 mm Hg at time of measurement of $V = 1.20$ ml of gas in the eudiometer with a head of mercury $H = 242$ mm Hg and the temperature of gas column T is 24°C . Then the volume of hydrogen, corrected to Standard Temperature and Pressure is:

$$\frac{273}{(273 + 24)} \times \frac{(745 - 242)}{760} \times 1.20 = 0.73 \text{ ml}$$

Figure 4 — Eudiometer Tube and Assembly for Standard Mercury Displacement Method

6.5 Reporting of Results. Whichever analytical procedure is used, the hydrogen volume collected shall be converted to Standard Temperature and Pressure (0°C [32°F] and 760 mm Hg [14.7 psia]). After hydrogen analysis, the test specimen shall be cleaned of any mercury or other displacement material. Evidence of significant levels of rust on the test specimen shall be cause for considering it a "no test" and that result discarded. It shall then be weighed to the nearest 0.1 g or less. The individual results shall be recorded on an analysis data sheet, such as is shown in Figure 5, along with the details of the analytical method and conditions.

The test specimen weight increase, compared to that before welding, shall be taken as the weight of the deposited metal. Then the hydrogen volume, converted to Standard Temperature and Pressure, shall be multiplied by 100 and divided by the deposit weight to obtain the diffusible hydrogen value in ml/100 g of deposited metal. Care must be taken to obtain an accurate measure of ambient atmospheric pressure to be used in these calculations. The average of four nominally identical test specimens shall then be calculated, and this value, rounded to the nearest 0.1 ml/100 g, shall be reported as

the diffusible hydrogen value for the welding electrode, flux, or shielding gas under test. In the event of a clearly evident leaking eudiometer or isolation chamber at this point or evidence of significant rust on a specimen one (and only one) result in either analytical system may be discarded and three values averaged to arrive at the diffusible hydrogen value.

6.6 Reference Atmospheric Condition. The use of a reference atmospheric condition during welding is necessitated because the arc is imperfectly shielded. Moisture from the air, distinct from that in the consumable, can enter the arc and dissociate, contributing to the hydrogen content of the arc atmosphere and the resulting observed diffusible hydrogen.

The reference atmospheric condition during welding which is recommended for specifying limits (unless otherwise noted) is 1.43 grams of water per kilogram of dry air (10 grains of water per pound of dry air). This corresponds to 21°C (70°F) and 10% relative humidity (RH) on a standard psychrometric chart at 760 mm (29.92 in.) Hg barometric pressure.

HYDROGEN ANALYSIS DATA

Test identification number _____		Date of analysis _____	
Analytical equipment operator _____			
Specimen storage time before loading analytical apparatus _____			
Specimen storage temperature _____		Specimen loaded from bath or warmed to 0°C _____	
Temperature of mercury during loading _____			
Hydrogen evolution time _____		Temperature _____	
Method of analysis _____			
Barometric pressure at time of analysis (mm Hg) _____		Ambient temperature at time of analysis (°C) _____	
Sample number	_____	_____	_____
Hydrogen volume collected, ml	_____	_____	_____
Head of mercury in tube, mm	_____	_____	_____
Temperature of gas, °C	_____	_____	_____
Hydrogen corrected to STP, ml	_____	_____	_____
Deposit weight, g	_____	_____	_____
Diffusible hydrogen, ml/100 g	_____	_____	_____
Average diffusible hydrogen, ml/100 g _____		Standard deviation of average, ml/100 g _____	

Figure 5 — Hydrogen Analysis Data Sheet

The recommended reference condition of 1.43 g/kg (10 grains/lb) for classification and lot conformance represents a minimal atmospheric contribution to the test results. Consequently, diffusible hydrogen requirements specified with this reference condition are indicative of the consumable contribution under a given set of welding conditions.

Actual atmospheric conditions shall be measured with any suitable instrument (calibrated in accordance with the manufacturer's procedure and the appropriate quality assurance requirements) at the time and location that test specimens are welded. The referee method shall be use of a psychrometer. Since the atmospheric moisture can only add to the contribution from the consumable, any tests conducted under conditions which equal or exceed the reference condition are considered valid tests. If the diffusible hydrogen results meet the specified requirements under such conditions, then the results are considered acceptable as demonstrating compliance with the requirement.

7. Standard Mercury Displacement Method

7.1 Safety Precautions. Metallic mercury and mercury vapors are hazardous and can be absorbed into the body by inhalation, ingestion, or contact with the skin. All precautions involving the handling of mercury should be observed, which include but are not limited to the following:

7.1.1 The diffusible hydrogen test apparatus should be located under a fume hood, and any steps involving the handling of mercury should be performed under a fume hood.

7.1.2 The apparatus should be located on a tray or counter with a raised lip to contain any possible spills.

7.1.3 Plastic or rubber gloves should be used at all times while handling mercury or mercury-contaminated samples and equipment.

7.1.4 Any mercury spill should be cleaned up immediately.

7.1.5 When not in use, the heating bath should be turned off, and the mercury in the plastic reservoir bottles should be capped.

7.1.6 Air sampling for mercury vapor should be done when the apparatus is first installed and periodically thereafter to determine if the legally mandated air standards are being met. See *OSHA Safety and Health Stan-*

dards, 29 CFR 1910, available from the U.S. Department of Labor, Washington, D.C. 20210.

7.2 Collection of Diffusible Hydrogen. Prior to the test, plastic bottles containing mercury shall be placed in a bath, and the bath and mercury shall be brought to a temperature of $45^{\circ}\text{C} \pm 3^{\circ}\text{C}$ ($113^{\circ}\text{F} \pm 5^{\circ}\text{F}$). The bath shall be maintained at this temperature throughout the entire test.

The weld test specimen shall be inserted into the eudiometer tube and, by a magnet or other means, held vertical while sliding the eudiometer tube and the specimen into the mercury. A vacuum shall be slowly and carefully drawn through the stopcock and the mercury allowed to be pulled up and around the test specimen in the eudiometer tube. (*Note: The stopcock shall have been ascertained to be leak-tight.*) The stopcock shall be closed when the mercury is about halfway up the calibrated portion of the eudiometer tube and the sample and tube slightly bounced against the bottom of the plastic jar to remove any entrapped air bubbles. (*Caution: Failure to remove air bubbles at this point may lead to a false high reading later.*) The stopcock shall be slowly opened again, and the mercury allowed to completely fill the tube and just pass through the stopcock opening. The stopcock shall be closed and the vacuum line removed. See Figure 6 for clarity.

The hydrogen will evolve from the weld test specimen and displace the mercury. The test period shall be 72 hours minimum. Before any readings are taken, the tube shall be bounced lightly against the bottom of the plastic bottle again to release any entrapped bubbles of hydrogen. The hydrogen volume shall be read to the nearest 0.02 ml or less. The head of mercury present, dimension H in Figure 4, shall be measured from the surface of the mercury in the bottle to the top of the mercury column in the eudiometer tube to the nearest 2 mm (0.079 in.). See Figure 4. The vacuum shall be released by slowly opening the stopcock so that the mercury runs back into the plastic bottle, and the specimen then removed from the mercury. The specimen shall be rinsed free of any residual mercury with running water or alcohol, while holding over a large beaker to collect any mercury in the rinsings. This residual mercury should be disposed of or recycled in an environmentally acceptable manner. The specimen shall be dried and weighed to the nearest 0.1 g or less.

7.3 Calculations. The hydrogen content shall be expressed in ml/100 g of deposited metal at Standard Temperature and Pressure. The volume of hydrogen gas is calculated as:

$$VH = \frac{273}{(273 + T)} \times \frac{(P - H) V}{(760)} \quad (\text{Eq. 1})$$

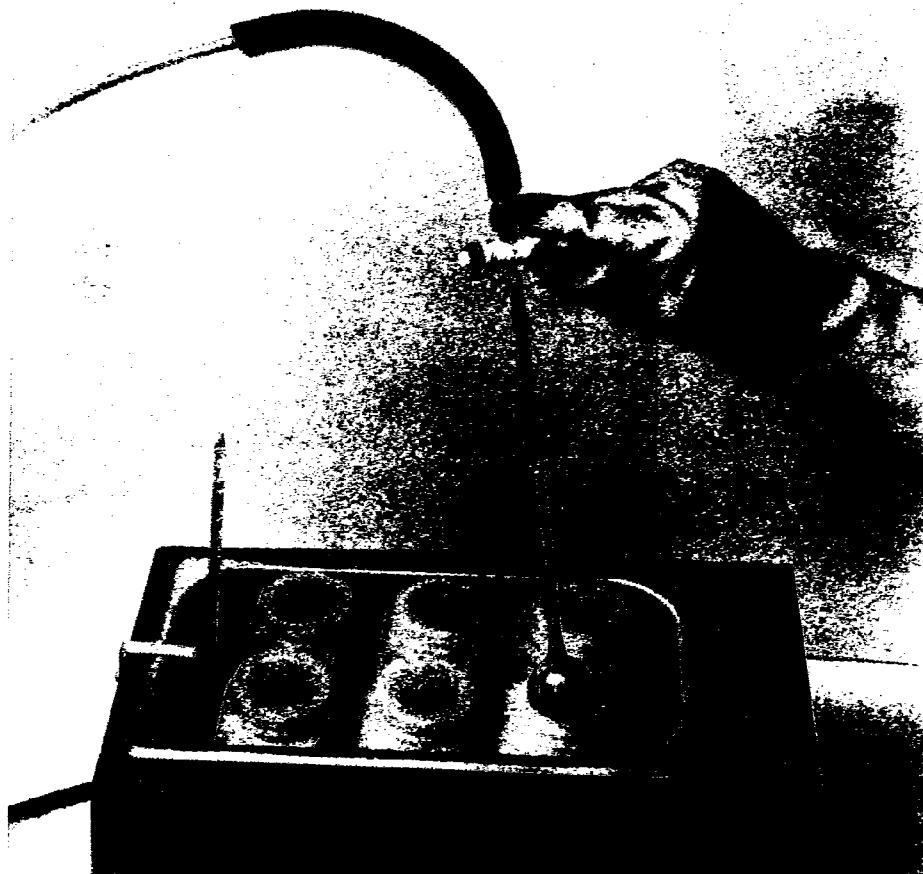


Figure 6 — Use of a Vacuum Line to Evacuate Ediometer Tubes Over a Heated Oil Bath

where

- T = gas column temperature ($^{\circ}\text{C}$) at time of measurement (note that the gas column temperature will be essentially the air temperature above the bath, not the heated bath temperature)
- P = barometric pressure (mm Hg) at time of measurement
- V = measured volume of gas in the eudiometer (ml)
- H = head of mercury (mm) at time of measurement (see Figure 4 for definition of head and a sample calculation)
- VH = volume of hydrogen gas converted to Standard Temperature and Pressure (ml)

This volume is then converted to ml/100 g as described in 6.5.

8. Standard Gas Chromatography Method

Specific requirements for satisfactory operation vary from instrument to instrument. Once the specimen is degassed in its isolation chamber, the quantity of hydrogen collected is determined by gas chromatography. Determinations shall be in accordance with ASTM E260, *Standard Practice for General Gas Chromatography Procedures*, and E355 *Standard Practice for Gas Chromatography Terms and Relationships*.¹ Calibration of a gas chromatograph may be affected by the volume of

1. ASTM standards can be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

total gas in the specimen isolation chamber being sampled. See the instrument manufacturer's instructions and calibration procedure.

Determine the volume of hydrogen contained in each isolation chamber by gas chromatography. If the hydrogen volume measured is not at standard temperature and pressure, conversion is made as follows:

$$VH = \frac{(273)}{(273 + T)} \times \frac{(P) V}{(760)} \quad (\text{Eq. 2})$$

where

T = temperature ($^{\circ}\text{C}$) of measured hydrogen volume

P = pressure (mm Hg) of measured hydrogen volume

V = measured hydrogen volume (ml)

VH = volume of hydrogen gas at Standard Temperature and Pressure (ml)

For purposes of converting to Standard Temperature and Pressure (STP), the temperature and pressure to be used are those conditions prevailing at the time that the standard used for calibration is prepared or analyzed, or both.

Remove specimens from the isolation chambers and weigh to the nearest 0.1 g or less. The hydrogen content shall be expressed in ml/100 g of deposited weld metal at STP as described in 6.5.

Appendix

Guide to Standard Methods for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic, and Ferritic Steel Weld Metal Produced by Arc Welding

(This Appendix is not a part of ANSI/AWS A4.3-93, *Standard Methods for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic, and Ferritic Steel Weld Metal Produced by Arc Welding*, but is supplied for information only.)

The following information is considered to be helpful to understanding the significance of test results obtained using this standard. The information is based upon published literature and the experience of the A5 Task Group to Develop a Ferritic Weld Metal Diffusible Hydrogen Standard. The data and relationships presented do not constitute a mandatory part of this standard.

A1. Introduction

During arc welding, monatomic hydrogen is produced in the arc by the decomposition of hydrogenous compounds which enter the arc. There are at least three sources of these hydrogenous compounds. The welding consumables (electrode, flux, or shielding gas) may contribute water, lubricants, or elemental hydrogen. The base metal may contribute water, lubricants, elemental hydrogen, paints, or other coatings. Aspiration air, with its own moisture, may enter the welding arc to a certain extent.

Monatomic hydrogen is extensively soluble in the weld pool and is retained in solution when the pool freezes. In solid martensitic, bainitic, and ferritic materials, hydrogen diffuses rapidly even at temperatures as low as room temperature. This rate of diffusion is reduced as the temperature is reduced.

There is a well established interaction in martensitic, bainitic, and ferritic materials among stress (such as weld residual stresses), high yield strength material, and diffusible hydrogen that can result in weld metal or heat-affected-zone cracking if the three factors are present in sufficient amounts. This cracking is often, but not always, delayed for some hours after welding, because of the time needed for hydrogen to diffuse to the crack site.

Measurements of weld hydrogen levels therefore provide the means of deciding the degree to which a given electrode, flux, or gas is introducing hydrogen into the weld pool. They may thus help to determine the sources of hydrogen and classify welding electrodes, fluxes, and gases. In addition, such measurements provide a starting point for calculating preheating temperatures and temperatures of heat treatment to remove hydrogen after welding.

Hydrogen, unlike other elements in weld metal, diffuses rapidly at normal room temperatures, and some of it may escape before an analysis can be made. This, coupled with the fact that the concentrations to be measured are usually at the parts per million level, means that special sampling and analysis procedures are needed. In order that results may be compared and used between different laboratories, standardization of these sampling and analysis methods is necessary.

After completion of diffusible hydrogen removal, some residual hydrogen will remain indefinitely "trapped" within the weld test specimen. Experience has shown that temperatures as high as 650°C (1200°F), or more, are necessary to remove it. This residual hydrogen appears to have no role in the incidence of weldment cracking except insofar as it may be converted back into diffusible hydrogen by re-entering the arc during a subsequent weld pass. Consideration of residual hydrogen is outside of the scope of this standard.

A2. Correlations of Diffusible Hydrogen with Covered Electrode Coating Moisture

It has been the practice for many years to assess the low hydrogen character of covered electrodes classified according to ANSI/AWS A5.5, *Specification for Low Alloy Steel Covered Arc Welding Electrodes*, by their coating moisture content. More recently, this test was added to ANSI/AWS A5.1, *Specification for Carbon Steel Electrodes for Shielded Metal Arc Welding*, as well. This approach has been a generally successful means of classifying electrodes.

Nevertheless, coating moisture is at best only an indirect assessment of the likelihood of hydrogen cracking from an electrode. This is especially true when the electrode departs from the as-manufactured condition by rehydration of the covering caused by exposure to the atmosphere prior to welding. It is also important to consider that during welding, the atmosphere itself, apart from any rehydration of the electrode, supplies moisture directly into the arc area which is always imperfectly shielded. The atmosphere infiltrating the arc is greatly affected by the welding technique used, in particular by the arc length held.

The literature contains a detailed study of these effects (IIW Document II-929-80). Although the hydrogen determination method employed in that study differed in certain respects from the methods herein, the trends are meaningful. In that study, effects of as-manufactured coating moisture, rehydrated coating moisture and atmospheric moisture during welding were considered. The

general trends for an AWS E7018 electrode are depicted in Figures A1 and A2. They agree with trends observed by AWS Task Groups.

Figures A1 and A2 illustrate the following two points:

(1) The diffusible hydrogen from a very dry electrode is affected by the atmospheric moisture in the welding environment. In particular, electrodes giving low hydrogen values when tested in a dry environment in January can be expected to give somewhat higher hydrogen values when tested in a humid environment in August.

(2) Rehydrated moisture is far less effective in introducing diffusible hydrogen into the weld than as-manufactured coating moisture.

While the numerical values obtained are likely to vary from one electrode formulation to another, the trends illustrated by the above data can be expected to be followed.

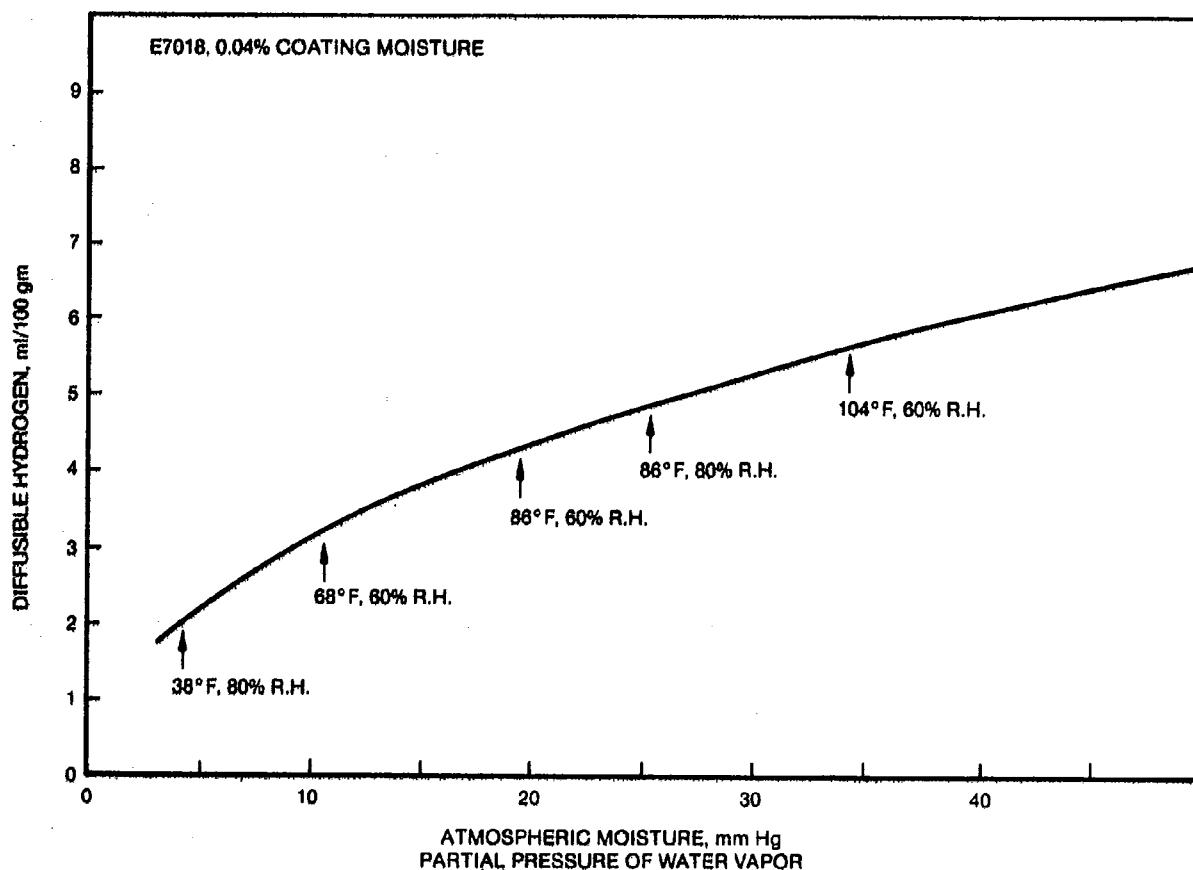
A3. Correlations of Diffusible Hydrogen Obtained by the Methods of this Standard with Values Obtained from Other Methods

This standard uses a larger test specimen than the IIW draft standard method or that of ISO 3690. Recent tests conducted both by laboratories in the United States and Europe have indicated that within the accepted inter-laboratory variation, the methods yield a 1:1 correlation with this AWS method.

As noted in the Foreword of this standard, laboratory-to-laboratory reproducibility of hydrogen determination using measurement by glycerin displacement in the AWS Task Group round robin studies was very poor. This is at least in part attributable to absorption of oxygen, nitrogen, other gases, and water by the glycerin (see Quintana, M. A. "A critical evaluation of the glycerin test." *Welding Journal* 63(5):141s-149s, May 1984). Therefore, any correlation of glycerin test results with results obtained using the methods of this standard, or of the IIW/ISO standards, must be viewed with skepticism.

Nevertheless, testing for diffusible hydrogen using glycerin displacement had been a part of earlier versions of MIL-E-24403² and the *Guide Specifications for Fracture Critical Non-Redundant Steel Bridge Members* of

2. United States of America Military Specification available from Commander, Naval Publications and Forms Center, 5801 Tabor Avenue, Philadelphia, PA 19120-5094.



Note: Specific partial pressures of water corresponding to certain atmospheric combinations of temperature and relative humidity are indicated by arrows.

Figure A1 — Approximate Effect of Atmospheric Moisture at Time of Welding on Diffusible Hydrogen with a Very Dry Covered Electrode

AASHTO³ for some time. The users of these two documents may wish to have at least an indication of how the results they have obtained in the past correlate with results obtained using this standard or the IIW/ISO methods.

Correlation comparisons have been made by the Japanese of the glycerin method of the Japanese Standard JIS Z3113⁴ with the IIW mercury method of ISO 3690 for covered electrodes. The Japanese standard is virtually

identical to the glycerin method of earlier versions of MIL-E-24403 and AASHTO procedures, while it has been shown that ISO 3690 for covered electrodes gives identical results to the IIW draft standard. And, as noted above, this standard appears to give results equal to those of the IIW draft standard.

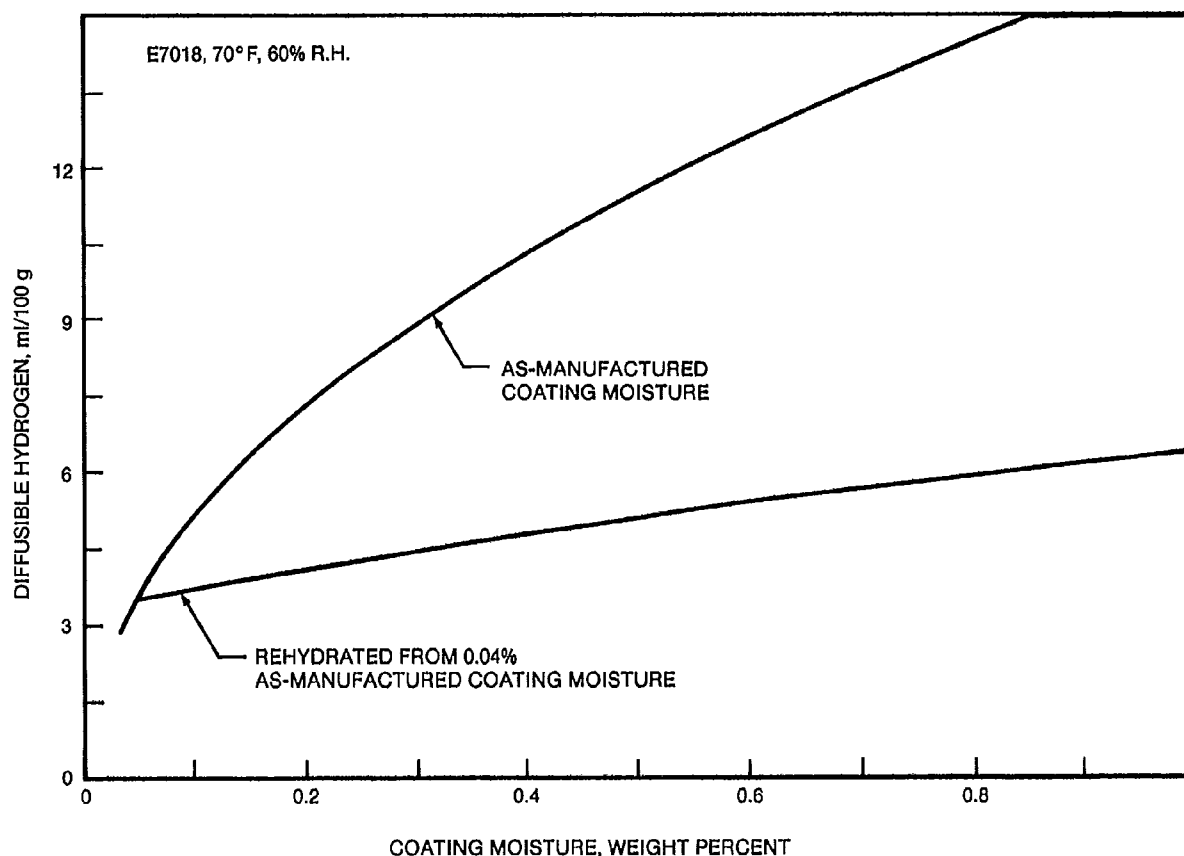
It has been the practice in the glycerin based standards to report hydrogen in units of ml/g of deposited metal, while this standard and ISO 3690 report hydrogen in ml/100 g of deposited metal. So the results obtained using the glycerin based standards need first to be multiplied by 100 to convert them to ml/100 g, which is also used in the JIS standard, if direct comparisons are to be made.

The earliest correlation reported in units of ml/100 g is given in IIW Document II-A-305-72 as:

$$H_{JIS} = 0.64 H_{IIW} - 0.93 \quad (\text{Eq. 1})$$

3. American Association of State Highway and Transportation Officials, 444 N. Capitol Street, N.W., Suite 225, Washington, DC 20001.

4. Japanese Industrial Standard, published by the Japanese Standards Association, 1-24, Akasaka 4 Chome, Minato-ku, Tokyo, 107 Japan.



Note: For a given total moisture level, as-manufactured moisture contributes more to the diffusible hydrogen than does rehydrated moisture.

Figure A2 — Approximate Effect of As-Manufactured Coating Moisture and Rehydration of a Very Dry Covered Electrode on Diffusible Hydrogen

This work was based on quenching the JIS specimen 30 sec after the arc was extinguished. Later, the Japanese moved to quenching within 5 sec after the arc was extinguished. Then additional testing was done, and a new correlation relation was recommended in IIW Document II-698-74 as:

$$H_{JIS} = 0.67 H_{IIW} - 0.8 \quad (\text{Eq. 2})$$

If this latter relationship is used to compare glycerin tests to both the IIW draft standard results and results obtained using this standard, then the approximate correlations of Table A1 are obtained.

Bearing in mind that the A5 Task Group found severe lab-to-lab variation in glycerin testing, and that there is appreciable statistical scatter in using the methods of this standard and of the IIW draft standard, one

can use Table A1 for only a rough correlation among the test methods.

A4. Welding Process Comparisons

Welding process comparisons have, thus far, been outside of the scope of AWS Task Group activities. The user of this standard may wish guidance in making such comparisons, and for this reason the following information has been extracted from IIW documents. At the present time, the AWS Task Group can make no judgments based upon actual experience regarding the adequacy of the following approach.

Welding process comparisons, due to variations in dilution, may require values of hydrogen concentration in terms of fused metal rather than of deposited metal for

Table A1
Diffusible Hydrogen
Measurement Correlations

Diffusible hydrogen determined by this standard, the IIW draft standard, or ISO 3690 (ml/100g)	Approximate diffusible hydrogen determined by glycerin based standards* (ml/100g)
1.0	0.0
2.0	0.5
3.0	1.2
4.0	1.9
5.0	2.6
6.0	3.2
7.0	3.9
8.0	4.6
9.0	5.2
10.0	5.9
11.0	6.6
12.0	7.2
13.0	7.9
14.0	8.6
15.0	9.3
16.0	9.9

*See Appendix, Section A3 for a discussion of the inaccuracy of hydrogen determinations by glycerin based standards.

valid comparison. It may also be desirable to evaluate autogenous welding processes, such as gas tungsten-arc welding, where calculation of diffusible hydrogen based upon deposited metal would not be possible, so that evaluation of diffusible hydrogen must be based upon fused metal.

If the hydrogen content is required in terms of its concentration in the fused metal, it is necessary to derive a figure for the mass of fused metal (WF , g). The cross-sectional area of fused metal can be measured on the fractured ends of the test specimen using an image analyzing microscope, an enlarged tracing, or an enlarged photograph. If filler metal was used and the weight of deposited metal obtained, then the area of deposited metal can be obtained also from the fractured ends of the test specimen as the area of the weld reinforcement. In this case, the weight of fused metal is obtained by multiplying the mass of deposited metal by the fused metal area divided by the deposited metal area.

If no filler metal was used, then the weight of fused metal can be calculated from the fused metal area multiplied by the test specimen length and multiplied by the density of the weld metal. For most steel weld metal, the

density can be estimated closely as the density of mild steel, 7.86 g/cc.

In either case, the averaged areas from the two fractured ends of the test specimen should be used in the computations.

To distinguish hydrogen expressed on the basis of fused metal (HF , ppm) from that on the basis of deposited metal, it is appropriate to express hydrogen based on fused metal in terms of parts per million (ppm). This is obtained from the weight of fused metal (WF , g) and the volume of hydrogen gas collected (V , ml) at standard temperature and pressure as:

$$HF = 90 V / WF \quad (\text{Eq. 3})$$

A5. Significance of Differences in Results Among Laboratories

In the process of developing this standard, the A5 Task Group conducted extensive round-robin testing to evaluate reproducibility of results among laboratories. Under the most carefully controlled conditions, with more than ten laboratories participating in the work, the Task Group observed variation in results of approximately ± 1 ml/100 g about the inter-laboratory mean when the inter-laboratory mean was less than 10 ml/100 g.

In order to put this variation into perspective, it is useful to consider that ± 1 ml/100 g is very nearly equal to ± 1 part per million concentration in the deposited weld metal. Such a level of variability in results would also be expected in analyzing other trace elements such as boron, nitrogen, sulfur or oxygen in weld metal. A similar level of inter-laboratory variation has also been observed when following the IIW draft standard (IIW documents II-A-576-82 and II-1019-84).

The source of this variability is not fully understood, but it is considered by the A5 Task Group that the greatest part of this variation arises at the time of preparing the test specimens, not at the time of analysis. Certainly the wet bulb temperature of the arc atmosphere at the time of welding will influence the results obtained. Variation in coating moisture of individual covered electrodes within a given lot has been observed by the Task Group. The quantity of drawing lubricant remaining on a wire at the time of welding may vary from point to point within a heat. The efficiency of a bake cycle for moisture removal may vary from point to point within a lot of submerged arc flux or electrodes.

Since the inter-laboratory variation appears to be about ± 1 ml/100 g when the inter-laboratory mean is 10 ml/100 g or less, it is the opinion of the A5 Task Group that no significance should be attached to a difference in individual test results between two laboratories that is smaller than 2 ml/100 g.

AWS Filler Metal Specifications and Related Documents

AWS Designation	Title
FMC	Filler Metal Comparison Charts
A4.2	Standard Procedures for Calibrating Magnetic Instruments to Measure the Delta Ferrite Content of Austenitic and Duplex Austenitic-Ferritic Stainless Steel Weld Metal
A4.3	Standard Methods for Determination of the Diffusible Hydrogen Content of Martensitic, Bainitic, and Ferritic Steel Weld Metal Produced by Arc Welding
A5.01	Filler Metal Procurement Guidelines
A5.1	Specification for Carbon Steel Electrodes for Shielded Metal Arc Welding
A5.2	Specification for Carbon and Low Alloy Steel Rods for Oxyfuel Gas Welding
A5.3	Specification for Aluminum and Aluminum Alloy Electrodes for Shielded Metal Arc Welding
A5.4	Specification for Stainless Steel Welding Electrodes for Shielded Metal Arc Welding
A5.5	Specification for Low Alloy Steel Covered Arc Welding Electrodes
A5.6	Specification for Covered Copper and Copper Alloy Arc Welding Electrodes
A5.7	Specification for Copper and Copper Alloy Bare Welding Rods and Electrodes
A5.8	Specification for Filler Metals for Brazing and Braze Welding
A5.9	Specification for Bare Stainless Steel Welding Electrodes and Rods
A5.10	Specification for Bare Aluminum and Aluminum Alloy Welding Electrodes and Rods
A5.11	Specification for Nickel and Nickel Alloy Welding Electrodes for Shielded Metal Arc Welding
A5.12	Specification for Tungsten and Tungsten Alloy Electrodes for Arc Welding and Cutting
A5.13	Specification for Solid Surfacing Welding Rods and Electrodes
A5.14	Specification for Nickel and Nickel Alloy Bare Welding Electrodes and Rods
A5.15	Specification for Welding Electrodes and Rods for Cast Iron
A5.16	Specification for Titanium and Titanium Alloy Welding Electrodes and Rods
A5.17	Specification for Carbon Steel Electrodes and Fluxes for Submerged Arc Welding
A5.18	Specification for Carbon Steel Filler Metals for Gas Shielded Arc Welding
A5.19	Specification for Magnesium Alloy Welding Electrodes and Rods
A5.20	Specification for Carbon Steel Electrodes for Flux Cored Arc Welding
A5.21	Specification for Composite Surfacing Welding Rods and Electrodes
A5.22	Specification for Flux Cored Corrosion-Resisting Chromium and Chromium-Nickel Steel Electrodes
A5.23	Specification for Low Alloy Steel Electrodes and Fluxes for Submerged Arc Welding
A5.24	Specification for Zirconium and Zirconium Alloy Welding Electrodes and Rods
A5.25	Specification for Carbon and Low Alloy Steel Electrodes and Fluxes for Electroslag Welding
A5.26	Specification for Carbon and Low Alloy Steel Electrodes for Electrogas Welding
A5.27	Specification for Copper and Copper Alloy Rods for Oxyfuel Gas Welding
A5.28	Specification for Low Alloy Steel Filler Metals for Gas Shielded Arc Welding
A5.29	Specification for Low Alloy Steel Electrodes for Flux Cored Arc Welding
A5.30	Specification for Consumable Inserts
A5.31	Specification for Fluxes for Brazing and Braze Welding

For ordering information, contact the Order Department, American Welding Society, 550 N.W. LeJeune Road, P.O. Box 351040, Miami, Florida 33135. Phone: 1-800-334-9353.