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Revision marker in margin of paragraph 5.1.2 was removed.



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1 SAMPLING

1.1 General

Good sampling is essential if the laboratory is to produce meaningful test measurement data. It should be planned and documented with as much care as the analysis itself. A sampling protocol should specify which aspects (e.g. population, stability, parameters to be measured, accuracy) should be considered and should specify which procedures are to be used (e.g. how to collect, reduction, preservation, etc.)

Representative samples are a prerequisite for the evaluation of any product or process stream. The directions for obtaining representative samples cannot be made explicit to cover all cases and must be supplemented by judgement, skill and sampling experience. Long sampling lines should be avoided.

1.2 Precautions

Refer to the section on safety aspects and consult ISO 3165 "Sampling of Chemical Products for Industrial Use - Safety in Sampling".

1.3 Material safety data sheets

Material safety data sheets are provided in the process design report.

1.4 Sampling techniques

Take EO/water samples in stainless steel cylinders. To avoid pressure build-up the sampling system should be designed such, that the cylinders sent for analysis are not liquid full.

Gaseous samples should be taken in metal pressure cylinders, unless at atmospheric pressure where glass sample tubes may be used (SMS 380). Most of the glycol streams are hot and sample coolers should be provided to allow sampling as a liquid at ambient temperature.

It is recommended that the appropriate sections of the following methods be employed:

ASTM D 3370 "Practices for Sampling Water from Closed Conduits"

ASTM E 300 "Practice for Sampling Industrial Chemicals"

SMS 380 "Sampling of Gases and Liquefied Gases"

If appropriate, special sampling procedures are given in the sampling sections of the respective test methods.

2 GUIDELINES FOR LABORATORY SAFETY

2.1 Introduction

Laboratories are generally recognized as places where a high potential for accidents exists. This is primarily due to the nature of the materials handled, the equipment used and the wide range of activities undertaken. However, due to an awareness of the potential hazards amongst laboratory workers, the frequency of accidents is usually relatively low, but they do nevertheless occur and can have serious consequences.

To ensure that such accidents are prevented it is essential that all locations prepare comprehensive written safety instructions and procedures covering foreseeable risks and potential hazards which could arise in the laboratory. These instructions and procedures should be placed in the context of the Enhanced Safety Management Programme.

The objective of these guidelines is to provide advice to managers responsible for laboratories on setting up safety management systems and assembling the information needed. This will enable laboratory staff to carry out their tasks in a safe and efficient manner, both inside and outside the department. Furthermore it will ensure that visitors will be adequately controlled and protected whilst they are present in the laboratory.

These guidelines are designed for use by all chemical manufacturing laboratories, irrespective of size, and notwithstanding the diverse nature of the work carried out in different locations. However, it is clear that the approach will depend on the size of the laboratory.

This document is therefore general in nature. It is designed to help individual locations to provide the necessary detailed instructions and procedures appropriate to their situation. To facilitate this, references are made to relevant open and in-house literature.

In those countries where statutory regulations or national codes of practice concerning laboratory operations are in place, these must be observed.

2.2 Enhanced safety management for laboratories

The principles of Enhanced Safety Management are fully applicable to laboratories.

The essential features of an effective safety programme are identified as:

- Visible management commitment to safety
- Sound safety policy
- Safety to be a line management responsibility
- Competent safety advisers
- High, well understood, safety standards
- Techniques to measure safety performance
- Realistic safety targets and objectives
- Audits of safety standards and practices
- Risk assessment of all activities.
- Effective safety training
- Thorough investigation and follow-up of injuries and incidents
- Effective motivation and communication

2.3 Hazards

A laboratory is an area of potential hazard because of the materials being handled and the techniques, which are performed. The hazards are increased because of the diversity of tasks undertaken. Known and routine reactions or tests should be carried out observing the prescribed precautions. These can normally be found in the relevant methodology documents such as SMS, ASTM and IP.

Non-routine reactions or tests should not be carried out without prior consultation with, and the agreement of, the laboratory supervision, and thereafter great care should be exercised.

Supervisors should ensure a standard, clearly-written procedure is worked out for each new routine task.

For convenience, these hazards have been divided into three categories, viz. chemical hazards (taken to mean reagents and samples), physical hazards and hazardous techniques. This categorisation follows that adopted by the Royal Society of Chemistry (RSC) in their Guide to Safe Practices in Chemical Laboratories.

Useful sources of general information on laboratory hazards are: Hazards in the Chemical Laboratory (ref. 2) and Handbook of Laboratory Safety (ref. 3).

These guidelines do not deal with biological hazards, nor those arising specifically in medical testing laboratories.

2.3.1 Chemical hazards

All laboratory reagents and samples must be considered as potentially hazardous to health and as posing a threat to safety.

In order to recognise the risks, laboratory staff should therefore be aware of any hazardous properties of the materials being used. This may be done by means of safety data sheets from the supplier or by reference books (refs. 2, 3, 7, 8, 9, 10, 11).

The more usual types of hazard to be found are:

- **Flammability:** flammable substances constitute the most frequently found hazard in the majority of laboratories. Special attention must be given to substances with a flash point below room temperature and it should be noted that auto-ignition temperatures of some materials are extremely low. Only the minimum amount of flammable liquids should be kept in laboratories and any operation which involves the production of non-contained flammable vapours should be carried out in a properly designed fume cupboard.

When large quantities of flammable solvents are dispensed it is essential to ensure that all vessels used are of metal and are electronically bonded and earthed. Corrosion resistant metal containers should be used wherever practical.

Vapours from flammable liquids are usually denser than air and may spread along the ground and reach distant sources of ignition.

Larger quantities of flammable liquids should be stored, preferably in metal cans, in storage rooms separate from the laboratory.

Solids such as alkali metals, metallic hydrides and some organometallic compounds are also flammable and frequently used in laboratories.

- **Corrosive and Irritant Substances:** for these chemicals it is especially important to avoid ingestion and skin or eye contact. Strict attention must be given to the use of the correct Personal Protection Equipment and eye-wash stations and emergency showers must be

available at the appropriate points. They should be regularly tested and a record kept of these tests.

- **Toxic or Harmful Substances:** these substances may cause illness, injury or even death, by inhalation, ingestion or skin contact. The use of the correct Personal Protection equipment is vital.

Toxic effects can result either from short-term (acute) exposure or long-term (chronic) exposure.

- **Highly Reactive Substances:** these substances may react vigorously because of their strongly oxidising or reducing properties. An authoritative source of information in this field is the "Handbook of Reactive Chemical Hazards" (ref. 7).

2.3.1.1 Spillage

Every laboratory worker should understand that if a hazardous chemical is spilled the situation must be dealt with both rapidly and effectively because of increased exposure problems. The method employed will depend upon the nature and quantity of the material spilt and the surface on which the spill has occurred.

Details for specific chemicals and the appropriate personal protective equipment necessary will be found on the Material Safety Data Sheet or by reference to one of the specialist books containing sections on clean-up procedures (ref. 2). It is important that appropriate clean-up materials are available in the laboratory and their mode of use is known and understood. Special attention must be paid to removing all traces of the spillage since additional injury can occur where ineffective or inappropriate measures are taken. If appropriate the environment should be tested to confirm the effectiveness of decontamination procedures.

The removal of spills of non-toxic material can be equally important in the prevention of accidents and all spills of oil, coffee, tea, etc. should immediately be cleaned up.

2.3.1.2 Storage

Chemicals must always be stored in a dedicated area that has been properly designed for the purpose. The following points of organisation must be borne in mind in storage spaces.

- **Inventory:** An inventory list should be maintained of all chemicals stored. The system should identify the consumption rate and age of chemicals so that the question of retention/disposal or replacement can be addressed from time to time. Quantities of chemicals stored should be kept to the minimum practicable.
- **Reactive Hazards:** Chemicals which react together must not be stored in close proximity (ref. 7).
- **Labelling:** All containers should be correctly labelled, indicating identification, warning symbols and expire date.
- **Large Liquid Containers:** These should always be kept at floor level.
- **Spillage Trays:** These must be used to contain spills of all chemicals. They should be made of appropriate material according to the chemical that may be spilt in them.
- **Shelving:** Shelving should always be maintained in sound and secure condition, and should be constructed of an appropriate material.

2.3.1.3 Waste disposal

Uncontrolled disposal can be hazardous and detrimental to the environment. Therefore care should be taken in the disposal of all laboratory chemicals. Procedures should be in place for the disposal of routinely handled chemicals, e.g. large quantities of solvents. In other cases the literature should be consulted (refs. 2, 3, 10 and 11).

Waste samples, reagents, etc., must never be returned to bottles and containers. It is good practice to purchase reagents and chemicals in containers and bottles of such size that the contents will be completely consumed within a few months to reduce any possible deterioration in quality.

Fixed disposal points should be clearly indicated and suitable covered containers should be provided in the laboratory, or close to it, for the collection and segregation of hazardous waste and the containers should be labelled. Care should be taken not to produce in these containers reactive mixtures that will in themselves create fresh hazards (ref. 7).

It should be remembered that accumulation of toxic material in the domestic sewage system, to which many existing laboratory drains are connected, can be hazardous to maintenance personnel. Furthermore the proper biological balance in the sewage treatment works can be seriously affected by changes in pH resulting from acid or alkaline effluent. The presence of heavy metallic and other toxic elements is also detrimental. Strict limits on the concentrations of these substances must be observed. Disposal of acids and alkalis can be achieved by extensive dilution with water or, if the quantities are substantial, by prior neutralisation.

Both the facilities provided and the procedures in force must take into account environmental as well as safety requirements.

It should be kept in mind that the responsibility for waste may not cease on disposal and that disposal records should be kept. Gases and volatile liquids should be disposed of via a fume cupboard in a controlled manner and their container should be earthed.

The risk of creating a nuisance must be borne in mind and, accordingly, volatile liquids with unpleasant smells should not be allowed to evaporate to the atmosphere.

2.3.2 Physical hazards

In a laboratory, hazards may arise from physical factors, such as extremes of pressure and temperature, and all forms of radiation.

Special hazards include:

- Cryogenic Liquids: There are special hazards associated with cryogenic liquids and all personnel involved with their usage and handling must be appropriately trained and aware of the dangers.
- Radiation Hazards:
 - ❖ Ionizing. Careful protective measures must be taken to restrict exposure of individuals to this type of radiation. The use of exposure monitoring badges may be necessary.
 - ❖ Non-Ionizing. The exposure of individuals to non-ionizing radiation should be permitted only within safe limits. Particular attention should be paid to the use of lasers.
- Noise. Noise levels should be restricted as per the SHC guide (ref. 4).

- Thermal Stress. Expert advice should be sought when individuals are required to work under conditions of extremes of temperature.

2.3.3 Hazardous techniques

Apart from the hazards, which arise from the nature and state of the substances handled, hazards can also be generated by chemical or physical reactions involved in laboratory operations. Particular attention should be paid to hazards involving:

- Oxidative experiments and other reactions which are highly exothermic and/ or involve explosion risks, or the evolution of toxic fumes.
- Reactions at elevated pressures.
- Operations under vacuum.
- The handling of glass.
- The use of centrifuges.

2.4 Design

2.4.1 General

The following important aspects of design should be addressed for the design of the laboratory:

- location and layout
- piping
- heating, ventilation and air conditioning architectural design
- electrical installation
- fixed furniture and special exhausts and drains
- fire fighting facilities

In addition to the above, the following specific requirements must be carefully considered.

2.4.2 Means of escape

Signs giving directions for escape should be visible at night and after failure of normal power supply.

The distance of travel from within the laboratory room to the nearest final exit or exit serving a storey should be no more than 15 m.

There should normally not be less than two escape routes available from a storey or room with doors constructed to open outwards in the line of the exit route.

Every exit should be wide enough to enable easy passage out of the building.

Gangways within the laboratory should be of adequate width to afford rapid egress to the exit doors.

Lifts should never be used in emergency evacuation. Special arrangements should be made for disabled people.

2.4.3 Emergency alarm

A means of giving warning, in case of fire or other emergency, must be provided in all laboratories. The control panel for the system should be at a safe location and give an indication of the area from which the alarm originated. The alarm should also be automatically relayed to the fire fighting services if at all possible. The wiring of the alarm system should be exclusive to that system and must be tested on a regular basis.

2.4.4 Fixed fire extinguishing system

Some laboratory operations, e.g. stores, can be protected best by fixed fire extinguishing systems, which may be activated automatically or manually. Professional advice should be sought in regard to the choice and use of fixed installations.

2.4.5 Gas cylinders

It is recommended that as far as is possible individual gases are kept securely in separate bays outside the laboratory. Gas cylinders should be used inside the laboratory only under exceptional circumstances, because they present a real danger in the event of a fire, whether they are empty or full. The size of cylinders temporarily used in the laboratory should be kept to a minimum, preferably 5 L or less. Cylinders should be colour-coded and bear an unambiguous description of their contents. They should be regularly inspected and tested.

Pipework between the supply cylinders and the point of usage should have a minimum of couplings and connections, but should be provided with easily accessible and clearly marked shut-off valves at strategic points, both inside and outside the building.

2.4.6 Electrical installation

All electrical installations should be carried out in accordance with the legislative requirements pertaining to the country in which the laboratory is situated. Consideration should be given to the use of automatic or remote circuit breakers as a means of isolation of the electrical circuit.

Switches, flexible leads, fuse ratings, insulation and earth resistance of plugged appliances should be regularly inspected. The above checks must all be recorded. The earth resistance of socket outlets should also be checked (more frequently when atmospheric corrosion of the contacts is likely).

2.4.7 Fume cupboards

Particular attention must be given to the siting and design of fume cupboards (ref. 14).

The fume cupboards installed in a laboratory should meet the most stringent foreseen general requirement of that laboratory and ventilation performance should be checked at least annually. In all cases separate vents to atmosphere from each fume cupboard should be installed. The effect of fume cupboard extraction capacity should be taken into account in the design of the air conditioning equipment.

2.4.8 Solvent store

Bulk stocks of laboratory solvents, chemicals and fuels should be stored in a separate building, constructed of fire-proof material, to local or national standards, be adequately ventilated and provide fire containment.

The store should be 15 m from other buildings. If fuels and/or solvents are supplied to the laboratory buildings by fixed pumps and pipework, a system must be provided such that the supply is automatically cut off in the event of fire. It may be appropriate for this store room to be protected by a fixed fire fighting system, which may be activated either automatically or manually. Professional advice must be sought in regard to the choice and use of such fixed installations

2.4.9 Retained sample store

Retained samples should be stored in a separate building at least 15 m from other buildings. Shelves/racks for storing these samples should be made of metal and be of sound construction.

As above it may be appropriate to protect this building with a fixed fire fighting system, which may be activated either automatically or manually. Again, professional advice must be obtained before installation of such a system.

2.4.10 Laboratory chemical store

Laboratory chemical stores will usually be an integral part of the main laboratory building and in view of the relatively large quantities of chemicals stored will constitute an area of high risk.

It is essential that due consideration is given to all hazards that may arise. Thus the store should be ventilated, have spillage containment, the floor should be of non-absorbent, easily cleanable material and have properly sited doors with easy access to firefighting appliances. It should be separated from the main laboratory by a fire-proof wall. Shelving should also be non-absorbent. Access to the chemical store should be restricted to authorised personnel.

2.5 Emergency procedures

2.5.1 General

Procedures should be established for all foreseeable emergency situations, and should be practiced regularly and recorded. Procedures should include how to inform emergency services, how to alert other staff, and indicate the appropriate initial response expected from staff in the immediate vicinity. Plans should be established for safe evacuation of buildings and these need to include isolating and shutting down of equipment, re-entry to the building and check in a head count.

A person should be appointed for each building who will be responsible for liaison with attendant emergency services to give them factual information on any special hazards, e.g. dangerous chemicals within a building on fire and on head count. To facilitate head counting it is recommended that a formal system of recording staff presence in, or absence from, the laboratory is established, e.g. a tag board. The system adopted should enable the laboratory supervision or emergency services to determine rapidly the total staff to be accounted for in any emergency situation.

A plan should be prepared showing the location and nature of flammable and dangerous chemicals which can be consulted by the emergency services.

The above should be formulated following due consultation with laboratory staff and professional emergency services where applicable.

2.5.2 Occupational health

Laboratory management should ensure that proper attention is given to the following key elements:

- health hazard identification:
identify and make inventory of potential physical, chemical, biological, ergonomic and psychological health hazards;
- health risk assessment:
evaluate the risk to health associated with exposure to health hazards and decide on remedial action;
- control measures:
introduce measures to prevent or control health risks
- health surveillance:
monitor health where risk to health cannot be excluded;
- record keeping:
keep record of exposure and surveillance to identify trends and areas of action, to satisfy legal requirements and safeguard the company against unjustified claims.

2.5.3 Personal protection equipment

2.5.3.1 General

The selection and use of personal protective equipment in the laboratory should be considered in conjunction with other methods of controlling hazards. As a means of controlling exposure to hazards it should be seen as the last resort, with other measures being implemented first.

2.5.3.2 Eye protection

The wearing of eye protection should be mandatory at all times in laboratory rooms where operations are carried out. Safety spectacles conforming to an appropriate standard (e.g. BS 2092) would be a minimum requirement. The protection provided by ordinary glasses is insufficient. Although contact lenses do offer some protection against mechanical damage to the eye, they can interfere with eye irrigation if such treatment is required. Safety spectacles should be worn in addition to contact lenses. Under many circumstances, however, safety spectacles do not provide adequate protection, for example where there is an increased risk of splashing or where very corrosive or highly irritant materials are being handled. These risks should be minimized by the design of chemical dispensing systems and the selection of types of packaging. Reference 7 should be consulted for selection of eye protection equipment.

Transparent protective screens of plastic or gauze placed in front of or over working equipment, particularly glassware, are a good first line of defence. Gauze covers should also be provided for glassware, such as vacuum filter vessels, working under reduced pressure.

2.5.3.3 Gloves

There should be a ready supply of gloves selected for protection against heat, cold, corrosive or toxic chemicals. For toxic and solvent chemicals particularly there is a wide range of available materials each with different resistance to permeation. Specialist advice should be sought when glove performance is a critical issue.

2.5.3.4 Coats, overalls and footwear

Staff must wear suitable laboratory coats or overalls in the working area. There should be a ready supply of these and a system for laundering. Normal footwear should be non-absorbent and cover the whole foot (not sandals). Safety shoes should be worn where appropriate. For more hazardous operations involving liquids impervious aprons, suits with appropriate cuffs, hoods, anklets and rubber boots should be selected. A suitable method for decontamination/disposal should be provided.

2.5.3.5 Protective equipment for special situations

When emergency procedures are formulated, the laboratory management should consider the extent to which the laboratory staff, rather than the specialist services, will be required to intervene and protective equipment should be supplied accordingly, e.g. emergency breathing apparatus should be provided in particularly hazardous areas as a means of facilitating escape or for re-entry in emergency. Staff must receive training in the use of such equipment.

2.5.3.6 Warning signs

At the entrance to all areas in the laboratory where personal protective equipment is required, clear, concise warning signs are mandatory.

2.5.3.7 Visitors

All visitors to laboratories need to be informed of, and must comply with, mandatory protective requirements and be accompanied by laboratory staff at all times.

2.5.3.8 Escape routes

Escape routes and doors must be kept free of obstructions and regularly checked with a written record kept.

2.5.3.9 First aid

An adequate number of staff should be trained in First Aid and appropriate materials should be available and regularly checked.

2.5.3.10 Work permits

A permit to work system must be written and enforced covering:

- laboratory staff working outside their normal department,
- non-lab staff (including contractors) working in the laboratory.

If a work permit system exists, then the laboratory procedures above must be fully integrated into this. Documentation as is commonly in place with Operating Companies may be adopted in a laboratory. However, as this has usually been designed for general use within a site it is invariably inadequate when confronted with the special hazards that may occur in a

laboratory. Hence, documentation may need to be specially designed to cover individual laboratory requirements. In connection with the above it is most important to ensure that the permit to work system includes reporting arrival and departure from the work areas. The access to all laboratories by non-lab personnel must be strictly controlled.

2.5.4 Sampling and samples

2.5.4.1 Sampling

Laboratory personnel will sometimes be involved in taking samples outside the laboratory, e.g. from plant streams and finished product tankage.

Procedures including use of appropriate protective equipment should be developed with the cooperation of operations personnel. Sampling points should be clearly identified.

2.5.4.2 Samples

Samples must be clearly and correctly labelled at all times. This applies to:

- the initial sample
- divided samples (aliquoting)
- samples for dispatch
- retained/stored samples
- samples for disposal.

The regulations governing sample movement are dependent upon quantity, destination and mode of transportation. Correct labelling is most important to impart the relevant information both to the transporter and to the recipient. This applies equally to samples moved within a laboratory complex as to an outside location, although the requirements may be different.

Correct packaging, for external dispatch, is the responsibility of the dispatcher.

Samples are frequently stored for prolonged periods prior to disposal.

Suitably designed storage and disposal facilities must exist at all sites.

Disposal must conform to local and national regulations.

2.5.4.3 Contractors

All hired staff and contractors must be brought within the safety regime of the whole site. Therefore all their work must be covered by the laboratory safety procedures and permit to work system as appropriate. In the case of

contractors who are required to carry out laboratory tasks, it will be essential that they are given all necessary training to enable them to carry out their work in a safe manner.

2.5.4.4 Safety training and auditing

The importance of effective safety training is stressed in the guide to Enhanced Safety Management, as is the value of audits of Safety Standards and Practices.

2.6 References

This list is current at the time of writing this report and may become outdated as new editions are published. Care should be taken to ensure that the latest edition is consulted.

1. deleted
2. Hazards in the Chemical Laboratory (Royal Society of Chemistry, London, 4th Edition, 1986)
3. Handbook of Laboratory Safety (Chemical Rubber Company) 2nd Edition 1980 Ed. N.V. Steere
4. Noise Guide (Shell Safety Committee - 1981 and Addendum - 1984)
5. deleted
6. deleted
7. Handbook of Reactive Chemical Hazards (L. Bretherick), 6th Edition 1999, Butterworth-Heineman
8. Sax's Dangerous Properties of Industrial Materials (R.J. Lewis, John Wiley & Sons, Chichester, UK, 8th Edition 1992)
9. Condensed Chemical Dictionary (R.J. Lewis, John Wiley & Sons, Chichester, UK, 13th Edition 1997)
10. Rapid Guide to Hazardous Chemicals in the Workplace (R.J. Lewis, John Wiley & Sons, Chichester, UK, 1997)
11. Handling Chemicals Safely (Dutch Safety Institute, Amsterdam, 1980)
12. deleted
13. deleted
14. Laboratory Fume Cupboards, British Standard Institution DD.80
15. deleted

3 SPECIFICATIONS

The column test method shows the test method agreed for determining product quality (MEG, DEG, TEG and high purity EO). The column preferred test method is a more modern equivalent method. For determination of feedstock quality only the Shell preferred method is given.

3.1 Feedstock specification

3.1.1 Ethylene

	<u>Specification</u>	<u>Test Method</u>
Ethylene	min 99.95 % mol	ASTM D 2505 ⁽¹⁾
Ethane+methane	max 500 ppm mol	ASTM D 2505
Propylene and heavier hydrocarbons	max 10 ppm mol	ASTM D 2505
Acetylene	max 1 ppm mol	ASTM D 2505
Hydrogen	max 5 ppm mol	ASTM D 2504
Carbon monoxide	max 0.1 ppm mol	ASTM D 2504
Carbon dioxide	max 0.1 ppm mol	ASTM D 2505
Oxygen	Max 0.1 ppm mol	ASTM D 2505 ASTM D 4468 or ASTM D 3246
Total sulphur	max 0.5 mg/kg	
Total sulphur after sulphur guard bed	max 0.01 mg/kg	ASTM D 4468
Methanol	max 0.5 mg/kg	SMS 2047
Oxygenated compounds	max 0.5 mg/kg	SMS 2047
Total combined nitrogen	max 0.2 ppm mol	SMS 1735 SMS 1553, ASTM D1142
Water	max 0.1 ppm mol	
COS	max 0.02 ppm mol	
Mercaptans	max 0.3 ppm mol	
Others	zero	

⁽¹⁾ 100% - impurities

3.1.2 Oxygen

	<u>Specification</u>	<u>Test Method</u>
Oxygen	min 99.8 % mol	SMS 1735 ⁽¹⁾
Nitrogen + Argon ⁽²⁾	max 2000 ppm mol	SMS 1735
Trichloroethylene +trichloroethane	max 0.001 ppm mol	SMS 2412
Others	zero	

⁽¹⁾ 100% - impurities

⁽²⁾ Nitrogen and argon should be determined separately

3.1.3 Methane

	<u>Specification</u>	<u>Test Method</u>
Methane	min 93.0 % mol ¹⁾	ASTM D 1946
Ethane	max 0.05 % mol	ASTM D 1946
Propylene and heavier hydrocarbons	max 15 ppm mol	ASTM D 2505
Hydrogen	max 3.0 % mol	ASTM D 1946
Carbon monoxide/dioxide	max 0.5 % mol	ASTM D 1946
Acetylene	max 5 ppm mol	SMS 2505
Sulphur	max 2 mg/kg	ASTM D 4468 / ASTM D 3246
Sulphur (after guard bed)	max 0.01 mg/kg	ASTM D 4468
Methanol and other organic oxygenates	max 10 mg/kg	SMS 2047 SMS 1553,
Water	zero	ASTM D1142
Nitrogen	Max 0.84 % mol	SMS 1735
Others	zero	

¹⁾ Spec specified according to KOM.

Methane from natural gas supply will be upgraded in OBL purification facilities (MES scope). Organic chlorides spec in CO₂ vent of max 10 ppm vol may not be met in case ethane content of max 0.05 % mole in methane is exceeded due to higher moderator injection.

The simulations have been based on a methane feed stream composition of 99.11 % mole of methane, 0.05 % mole of ethane and 0.84 % mole of nitrogen.

3.1.4 Ethylchloride

	<u>Specification</u>	<u>Test Method</u>
Appearance	clear and free from foreign material	Visual
Purity	min 99.8 % wt	SMS 2910
Acidity (as HCl)	max 10 mg/kg	ASTM D 2989
Water	max 100 mg/kg	ASTM D 3401
Trichloroethylene + trichloroethane	max 50 mg/kg	SMS 2910
Non-volatile residue	Max 5 mg/100ml	ASTM D 2109

3.2 Product specification

3.2.1 MEG

	<u>Specification</u>	<u>Test Method</u>	<u>Preferred Method</u>
Appearance	Colourless, transparent	ASTM D 4176, Visual	Visual
Colour (Pt-Co)	max 5	ASTM D 1209	ASTM D 1209
Purity	min 99.9 % wt	SMS 2886	SMS 2886
DEG	max 0.08 % wt	SMS 2886	SMS 2886
Water	max 0.08 % wt	ASTM E 1064 / ASTM E 203	ASTM E 1064
Aldehydes (as formaldehyde)	max 8 mg/kg	SMS 1996	SMS 1996
Acidity (as acetic acid)	max 10 mg/kg	ASTM D 1613	ASTM D 1613
Iron (as Fe)	max 0.1 mg/kg	ASTM E 394	ASTM E 394
Inorganic chlorides (as Cl)	max 0.05 mg/kg	SMS 2901 ⁽¹⁾ / SMS 1677 ⁽¹⁾	SMS 1677
Ash	max 50 mg/kg	ASTM D 482	ASTM D 482
Specific gravity, 20/20 °C	1.1151-1.1156	ASTM D 4052	ASTM D 4052
UV Transmittance %		SMS 1997	SMS 1997
- 220 nm	min 80 %		
- 275 nm	min 95 %		
- 350 nm	min 99 %		
Distillation range at 0.1013 MPa		ASTM D 1078	ASTM D 1078
5 % vol	min 196°C		
95 % vol	max 199°C		

⁽¹⁾ Measured as total halogens

3.2.2 DEG

	<u>Specification</u>	<u>Test Method</u>	<u>Preferred Method</u>
Appearance	Colourless, clear	ASTM D 4176, Visual	Visual
Colour (Pt-Co)	max 10	ASTM D 1209	ASTM D 1209
Purity	min 99.8 % wt	SMS 2886	100 – impurities
MEG	max 0.05 % wt	SMS 2886	SMS 2886
TEG	max 0.05 % wt	SMS 2886	SMS 2886
Water	max 0.05 % wt	ASTM E 1064 / ASTM E 203	ASTM E 1064
Acidity (as acetic acid)	max 50 mg/kg	ASTM D 1613	ASTM 1613
Ash	max 50 mg/kg	ASTM D 482	ASTM D 482
Specific gravity, 20/20 °C	1.1175-1.1195	ASTM D 4052	ASTM D 4052
Distillation range at 0.1013 MPa		ASTM D 1078	ASTM D 1078
5 % vol	min 242°C		
95 % vol	max 250°C		

3.2.3 TEG

	<u>Specification</u>	<u>Test Method</u>	<u>Preferred Method</u>
Appearance	Colourless, clear	ASTM D 4176, Visual	Visual
Colour (Pt-Co)	max 25	ASTM D 1209	ASTM D 1209
Purity	min 99.5 % wt	SMS 2886	100 – impurities
DEG	max 0.35 % wt	SMS 2886	SMS 2886
PEG	max 0.1 % wt	SMS 2886	SMS 2886
Water	max 0.05 % wt	ASTM E 1064 / ASTM E 203	ASTM E 1064
Ash	max 100 mg/kg	ASTM D 482	ASTM D 482
Specific gravity, 20/20 °C	1.124-1.126	ASTM D 4052	ASTM D 4052
Distillation range at 0.1013 MPa		ASTM D 1078	ASTM D 1078
5 % vol	min 280°C		
95 % vol	max 295°C		

3.2.4 CO₂

	<u>Specification</u>	<u>Test Method</u>	<u>Preferred Method</u>
Organic chlorides	Max 10 ppm vol		
Sulphur	zero	ASTM D 4468	ASTM D 4468
EO	max 20 mg/m ³	SMS 2669	
Hydrocarbons (CH ₄ , C ₂ H ₄)	max 0.5 % vol	ASTM 2593 ASTM 2192 /	ASTM 2593 ASTM 2192 /
Aldehydes	max 300 ppm vol	ASTM 2194	ASTM 2194
EG	max 5 ppm vol		

4SAMPLE POINTS TEST METHOD

The frequency of the testing strongly depends upon the operating experience.

Sample description	Property	Unit	Test method
Oxygen feed	Oxygen	% (V/V)	SMS 1735
	Nitrogen/Argon	% (V/V)	SMS 1735
	Trichloro ethylene	$\mu\text{g}/\text{m}^3$	SMS 2412
Ethylene feed	Acetylene	ppm (mol)	ASTM D 2505
	Propylene and heavier hydrocarbons	ppm (mol)	ASTM D 2505
	Methanol	ml/m^3	SMS 2047
	Ethane + Methane	mol %	ASTM D 2505
	H ₂ /CO	ppm (mol)	ASTM D 2504
	Total sulphur	mg/kg	ASTM D 4468 / D3246
Methane feed	Acetylene	ppm (mol)	ASTM D 2505
	Composition	mol %	ASTM D 1946
	Total sulphur	mg/kg	ASTM D 4468 / D3246
EC moderator	Purity	% (m/m)	SMS 2910
	Acidity, as HCl	mg/kg	ASTM D2989
	Water	mg/kg	ASTM D3401
Ethylene feed after guard bed	Total sulphur	mg/kg	ASTM D 4468
	Acetylene	ppm (mol)	ASTM D 2505

Sample description	Property	Unit	Test method
Reactor feed	Check on process analysers		
	Total chlorine	ml/m ³	SMS 2296
	C ₁ ;CO ₂	% (V/V)	SMS 2655
	C ₂ ^o ;C ₂ ^{''}	% (V/V)	SMS 2655
	O ₂ ; Ar	% (V/V)	SMS 1735
	N ₂	% (V/V)	SMS 1735
Reactor product	EO; O ₂ ; etc.	% mol	process analysers
	Calibrate by laboratory analysed standard gas mixtures: EO: SMS 2669; C ₁ , C ₂ ^o , C ₂ ^{''} , CO ₂ : SMS 2655; O ₂ , Ar, N ₂ : SMS 1735		
Demineralised water	Conductivity	μS/cm	Meter
	Chloride ion	mg/L	ASTM D 512
Boiler feed water	Appearance		visual
	Electr. Conduct.	μS/cm	ASTM D 1125
	Silica (SiO ₂)	mg/kg	ASTM D 859B
	pH at 15 °C		ASTM D 1293
	Dissolved oxygen	mg/kg	ASTM D 888A
	Iron	mg/kg	ASTM D 1068B
	Copper	mg/kg	ASTM D 1688B
	Total CO ₂	mg/kg	ASTM D 513
	Hardness	mg CaCO ₃ /l	ASTM D 1126
	KMnO ₄ consumption	mg/kg	ISO 8467
	Oil	mg/kg	ASTM D 3921, IP 426
Coolant circulation	As for reactor make-up feed water.		
CO2 removal section			SMS 2909

Sample description	Property	Unit	Test method
EO absorber overhead gas	EO	% (V/V)	SMS 2669
Quench bleed	pH		Meter
	EO	% (m/m)	SMS 2908
	MEG	% (m/m)	SMS 2001
Lean absorbent to EO absorber	EO	mg/kg	SMS 2567
	MEG	% (m/m)	SMS 2001
	foaming	cm	ASTM D 3601
	pH		Meter
EO absorber fat absorbent	EO	% (m/m)	SMS 2908
Quench bleed residue	MEG	% (m/m)	SMS 2001
	pH		Meter
Glycol bleed	MEG	% (m/m)	SMS 2907
	DEG	% (m/m)	SMS 2907
	Total glycols	% (m/m)	SMS 2907
	pH		Meter
LE column btms	EO	% (m/m)	SMS 2908
	Aldehydes	mg/kg	SMS 2786
	Water	% (m/m)	ASTM E 1064 or E 203
	CO ₂	mg/kg	SMS 1968

Sample description	Property	Unit	Test method
Recycle water	MEG	% (m/m)	SMS 2001
	pH		meter in lab
Glycol reactor feed	EO	% (m/m)	SMS 2908
Glycol reactor product	MEG	% (m/m)	SMS 2001
	EO	mg/kg	SMS 2567
Glycol conc. 1 btms	Total glycols	% (m/m)	SMS 2907
Glycol conc. 2 btms	Total glycols	% (m/m)	SMS 2907
Glycol conc. 3 btms	Water	% (m/m)	ASTM E 1064 or E 203
Dehydrator btms	MEG/DEG/TEG	% (m/m)	SMS 2907
	Water	% (m/m)	ASTM E 203 or E 1064
Dehydrator ovhd	MEG	mg/kg	SMS 2886
	COD	mg/kg	ASTM D 1252
Glycol residue	MEG/DEG	% (m/m)	SMS 2907
Condensed process steam	pH		meter
MEG column btms	MEG/DEG/TEG	% (m/m)	SMS 2907
MEG-product	Colour	Pt-Co	ASTM D 1209
	Water	% (m/m)	ASTM E 203 or ASTM E 1064
	DEG	% (m/m)	SMS 2886
	UV-transmittance	%	SMS 1997
MEG-product	Specification properties		See section 3.2.1
MEG past. cut	Water	% (m/m)	ASTM E 203 or ASTM E 1064
	Acidity, as HAc	% (m/m)	ASTM D 1613
	Aldehydes	% (m/m)	SMS 1996
	UV-transmittance	%	SMS 1997
Recycle MEG	DEG	% (m/m)	SMS 2907

Sample description	Property	Unit	Test method
MEG rec. col. btms	MEG	% (m/m)	SMS 2001
DEG-product	Colour	Pt-Co	ASTM D 1209
	Water	% (m/m)	ASTM E 203 or ASTM E 1064
	MEG/TEG	% (m/m)	SMS 2886
DEG-product	Specification properties		See section 3.2.2
DEG column bottom	TEG	% (m/m)	SMS 2907
TEG-product	Colour	Pt-Co	ASTM D 1209
	Water	% (m/m)	ASTM E 203 or ASTM E 1064
TEG-product	Specification properties		See section 3.2.3
Heavy glycols	TEG/TTEG	% (m/m)	SMS 2907

5 ANALYTICAL METHODS

5.1 Non-Shell methods - Including supporting and supplemental methods

5.1.1 ASTM

D 482	Ash from Petroleum Products
D 512	Chloride Ion in Water and Waste Water
D 513	Carbon Dioxide and Bicarbonate and Carbonate Ions in Water
D 859	Silica in Water and Waste Water
D 888	Dissolved Oxygen in Water
D 891	Specific Gravity of Liquid Industrial Chemicals
D 1066	Sampling Steam
D 1068	Iron in water
D 1078	Distillation Range of Volatile Organic Liquids
D 1125	Electrical Conductivity and Resistivity of Water
D 1126	Hardness in Water
D 1129	Definition of Terms Relating to Water
D 1192	Specifications for equipment for Sampling Water and Steam
D 1193	Specifications for Reagent Water
D 1209	Colour of Clear Liquids (Platinum-Cobalt Scale)
D 1250	Petroleum Measurement Tables
D 1252	Chemical Oxygen Demand of Water
D 1293	pH of Water
D 1613	Acidity in Volatile Solvents and Chemical Intermediates
D 1688	Copper in Water
D 1744	Water in Liquid Petroleum Products by Karl Fischer Reagent
D 1946	Reformed Gas Analysis by Gas Chromatography
D 2106	Amine Acid Acceptance of Halogenated Organic Solvents
D 2108	Colour of Halogenated Organic Solvents (Platinum-Cobalt Scale)
D 2186	Deposit-Forming Impurities in Steam
D 2504	Non condensable Gases in C3 and Lighter Hydrocarbons by Gas Chromatography
D 2505	Ethylene, Other Hydrocarbon, and Carbon Dioxide in High-Purity Ethylene by Gas Chromatography
D 2777	Precision and Bias of Methods of Committee D-19 on Water
D 2989	Acidity-alkalinity of Halogenated Organic Solvents
D 3223	Total Mercury in Water

D 3246	Sulphur in Petroleum Gas by Oxidative Microcoulometry
D 3370	Practices for Sampling Water
D 3401	Water in Halogenated Organic Solvents
D 3601	Foam in Aqueous Media (Bottle Test)
D 3694	Practices for Preparation of Sample Containers and Preservation of Organic Constituents
D 3919	Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
D 3921	Oil and Grease and Petroleum Hydrocarbons in Water
D 4051	Practise for Preparation of Low Pressure Gas Blends
D 4052	Density and relative density of liquids by digital density methods
D 4057	Practise for Manual Sampling of Petroleum and Petroleum Products
D 4127	Terminology Used with Ion Selective Electrodes
D 4177	Practise for Automatic Sampling of Petroleum and Petroleum
D 4453	Practise for Handling of Ultra-pure Water Samples
D 4468	Total Sulphur in Gaseous Fuels by Hydrogenolysis and Rateometric Colorimetry
D 4519	On-line Determination of Anions and Carbon Dioxide in High Purity Water by Cation Exchange and Degassed Cation Conductivity
D 4691	Practise for measuring Elements in Water by Flame Atomic Absorption Spectrophotometry
D 4841	Practices for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
D 5391	Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample
D 5540	Practise for Flow Control and Temperature Control for On-line Water Sampling and Analysis
D 5542	Trace Anions in High Purity Water by Ion Chromatography
E 1	Specifications for ASTM Thermometers
E 60	Photometric Methods for Chemical Analysis of Metals
E 70	pH of Aqueous Solutions with the Glass Electrode
E 100	Specifications for ASTM Thermometers
E 131	Def. of Terms and Symbols of Molecular Spectroscopy
E 133	Specifications for Distillation Equipment
E 169	Ultraviolet Quantitative Analysis
E 180	Precision Data on ASTM Methods for Analysis and Testing of Industrial Chemicals

E 200	Preparation, Standardisation, and Storage of Standard Solutions for Chemical Analysis
E 201	Calc. of Volume and Weight of Industrial Chemical Liquids
E 202	Analysis of Ethylene Glycols and Propylene Glycols
E 203	Test for Water Using Karl Fischer Reagent
E 260	General Gas Chromatography Procedures
E 275	Describing and measuring Performance of Spectrophotometers
E 299	Trace Peroxides in Organic Solvents
E 300	Sampling Industrial Chemicals
E 394	Iron in Trace Quantities Using the 1,10-Phenanthroline Method
E 1064	Water in Organic Liquids by Coulometric Karl Fischer Titration
E 1547	Terminology Relating to Industrial Chemicals
F 307	Practice for Sampling Pressurized Gas for Gas Analysis

5.1.2 ISO

760	Determination of Water - Karl Fischer Method (General Method)
2210	Liquid Halogenated Hydrocarbons for Industrial use - Determination of Residue on Evaporation
3165	Sampling of Chemical Products - Safety in Sampling
3170	Petroleum Liquids - Manual Sampling (equivalent to ASTM D 4057)
3171	Petroleum Liquids - Automatic Pipeline Sampling (equivalent to ASTM D 4177)
8467	Determination of Permanganate Index

ASTM-methods are published by the American Society for Testing and Materials.

ASTM European Office, 27/29 Knowl Piece, Wilbury Way, Herts SG4 OSX, United Kingdom

ISO-methods are published by the International Organisation for Standardisation.

ISO, 1 Rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland.

5.2 Shell methods - Including supporting and supplemental methods

5.2.1 SMS

CT6	Tables for Density of Water
CT7	Table for Vapour Pressure of Water
51	Water in Liquid and Solids

380	Sampling of Gases and Low-Boiling Liquids
1000	Accuracy (Precision and Trueness) of SMS Methods
1001	Guidelines for Verification and Calibration of Viscosity Tubes
1002	Guidelines for Verification and Calibration of Liquid-in-Glass Thermometers
1003	Guidelines for Verification and Calibration of Glass Hydrometers
1004	Guidelines for Verification of Capacity of Volumetric Glassware
1005	Guidelines for Statistical Evaluation of the Linear Calibration Function as Used in Analytical Methods
1006	Guidelines for Statistical Control of Test Methods
1008	Guidelines for Statistical Control of Test Methods
1010	Guidelines for Verification and Calibration of Soapfilm Flowmeters
1011	Guidelines for Verification and Calibration of Rotameters
1012	Guidelines for Verification of Wet and Dry Total Gas Volume Meters
1013	Guidelines for Verification of Liquid Flowmeters
1014	Guidelines for Verification of (Analytical) Balances
1015	Guidelines for Verification of Capacities of Automatic Pipettes
1016	Verification of Capacity and Check of Linearity of Delivery of Automatic Burettes
1017	Calibration and Verification of Thermocouples/Display and Digital Thermometers
1019	Calibration of Control Frequency of Equipment
1553	Water in Gases
1720	Hydrogen Sulphide in Gases
1735	Oxygen, Argon, Nitrogen, Methane and Carbon Monoxide in Gases
1968	Acidity and Carbon Dioxide in Ethylene Oxide and Propylene Oxide
1996	Aldehydes in Monoethylene Glycol
1997	Ultraviolet Transmittance of Ethylene Glycol
2001	Monoethylene Glycol in Mixtures
2047	Acetone, Methanol and Isopropyl Alcohol in Ethylene and Propylene
2296	Halogens in Gases and Organic Liquids
2327	Non-volatile Matter in Ethylene Oxide
2412	Trichloroethene and 1,1,1-Trichloroethane in Oxygen
2423	Peroxides Present in Ethers, Solvents and Hydrocarbons
2500	Gas Chromatographic Analysis : Supporting Method
2567	Ethylene Oxide in Polyethylene Glycols and Detergent Ethoxylates
2654	Impurities in Dichloroethane
2655	Methane, Carbon Dioxide and C2 through C7 hydrocarbons in gases

2669	Ethylene Oxide in Nitrogen
2691	Total Sulphur in Gases
2786	Aldehydes in Ethylene Oxide and Propylene Oxide
2886	Glycol Impurities in Mono-, Di- and Triethylene Glycols
2907	Composition of Ethylene Glycols
2908	Ethylene Oxide in Aqueous Solutions
2909	Analysis of Plant Solution from the Carbon Dioxide Removal Unit of the Ethylene Oxide/Ethylene Glycol Process
2910	1,1,1-Trichloroethane, Trichloroethylene Content and Purity of Ethyl chloride

Shell Methods are being provided to licensees in Bench Copy Books

Requests for further Shell methods should be directed to:

EO/EG Department in Shell Global Solutions International BV
Research and Technology Centre Amsterdam
P.O. Box 38000, 1030 BN Amsterdam, The Netherlands.

with a copy to:

Analytical Department in Shell Global Solutions International BV
Shell Research and Technology Centre Amsterdam
P.O. Box 38000, 1030 BN Amsterdam, The Netherlands.

6 LABORATORY EQUIPMENT

6.1 Special laboratory equipment

Microcoulometer for the analysis of sulphur/chlorides	1
Karl Fischer titration apparatus	1
Spectro-photometer UV/Vis light	1
Spectro-photometer AAS	1
pH meter	1
Potentiometric titration equipment	1
Gas chromatograph for gas analysis, isothermal, flame ionisation detection (FID)	2
Gas chromatograph for gas analysis, isothermal, thermal conductivity detection (TCD)	3
Gas chromatograph for gas analysis, temperature programme, TCD	2
Conductivity meter	1
Gas chromatograph for liquid analysis, temperature programme, dual FID	1
Gas chromatograph for liquid analysis, isothermal, FID	1
Gas chromatograph for gas analysis, isothermal, dual FID	1
Stainless steel sampling bombs, high pressure (ethylene)	4
Stainless steel sampling bombs, medium pressure (EO)	6
Air sampling equipment (set of charcoal tubes)	1

6.2 Standard laboratory equipment

Laboratory glassware (burettes, pipettes, flasks, bottles, condensers, etcetera), balances, hot plates, heating mantles, steam baths, thermostat baths, thermometers, thermocouples, hydrometers, Dewar flasks, gas flow meters, syringes, sieves, distillation equipment, ovens, furnace, colour comparators etcetera.

7 GLOSSARY OF ABBREVIATIONS

AAS	Atomic Absorption Spectrometry
ASTM	American Society for Testing and Materials
btms	Bottoms
bp	Boiling Point
B.S.	British Standards
B.V.	Besloten Vennootschap
C-	Column
cm	Centimetre
c/o	Care of
COD	Chemical Oxygen Demand
col.	Column
conc.	Concentrator
DEG	Diethylene Glycol
disch.	Discharge
E-	Exchanger
EC	Ethylene Chloride
e.g.	Exempli Gratia (for example)
EO	Ethylene Oxide
eq.	Equivalent
et al.	Et Alli, et alia (and others)
HAc	Acetic Acid
IP	Institute of Petroleum
ISO	International Organisation for Standardisation
K-	Compressor
kg	Kilogram
kJ	Kilojoule
L	Litre
liq.	Liquid
Ltd	Limited
m	Metre
m ³	Cubic Metre
max.	Maximum
MEG	Monoethylene Glycol
mg	Milligram
μg	Microgram
min.	Minimum

mL	Millilitre
μL	Microlitre
m/m	Mass/Mass
mol	Mole
μS	Micro Siemens
nm	Nanometre
ovhd	Overheads
P-	Pump
Pa.	Pennsylvania
P.O.	Post Office
ppm	Parts Per Million
Pt-Co	Platinum-Cobalt (colour scale)
R-	Reactor
ref.	Reference
SSC	Shell Safety Committee
SMS	Shell Method Series
TEG	Triethylene Glycol
TLV	Threshold Limit Value
TTEG	Tetra Ethylene Glycol
UV	Ultraviolet
V-	Vessel
V/V	Volume/Volume
WPH	Water Purity High