

Oil-filled electrical equipment — Sampling of gases and of oil for analysis of free and dissolved gases — Guidance

The European Standard EN 60567:2005 has the status of a
British Standard

ICS 17.220.99; 29.040.10; 29.035.99

National foreword

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The UK participation in its preparation was entrusted to Technical Committee GEL/10, Fluids for electrotechnical applications, which has the responsibility to:

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**Oil-filled electrical equipment –
Sampling of gases and of oil for analysis of
free and dissolved gases –
Guidance
(IEC 60567:2005)**

Matériels électriques immergés –
Echantillonnage de gaz et d'huile pour
analyse des gaz libres et dissous –
Lignes directrices
(CEI 60567:2005)

Ölgefüllte elektrische Betriebsmittel –
Probennahme von Gasen und von Öl
für die Analyse freier und gelöster Gase –
Anleitung
(IEC 60567:2005)

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CENELEC

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Foreword

The text of document 10/621/FDIS, future edition 3 of IEC 60567, prepared by IEC TC 10, Fluids for electrotechnical applications, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 60567 on 2005-09-01.

This European Standard supersedes EN 60567:1992.

The main changes with respect to EN 60567:1992 are listed below.

Since the publication of EN 60567:1992, a number of new gas extraction methods have been developed and are commercially available, such as mercury-free versions of the standard Toepler and partial degassing methods, which are referenced to in Annex C of this new edition. The head space method, based on a new concept for the extraction of gases from oil is introduced as a full method, and reference is made to a simplified version of it also in Annex C (shake test method). More sensitive chromatographic techniques have also been developed and are presented in this new edition of the standard.

The following dates were fixed:

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Annex ZA has been added by CENELEC.

Endorsement notice

The text of the International Standard IEC 60567:2005 was approved by CENELEC as a European Standard without any modification.

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INTRODUCTION

Gases may be formed in oil-filled electrical equipment due to natural ageing but also, to a much greater extent, as a result of faults.

Operation with a fault may seriously damage the equipment, and it is valuable to be able to detect the fault at an early stage of development.

Where a fault is not severe, the gases formed will normally dissolve in the oil, with a small proportion eventually diffusing from the liquid into any gas phase above it. Extracting dissolved gas from a sample of the oil and determining the amount and composition of this gas is a means of detecting such faults, and the type and severity of any fault may often be inferred from the composition of the gas and the rate at which it is formed.

In the case of a sufficiently severe fault, free gas will pass through the oil and collect in the gas-collecting (Buchholz) relay if fitted; if necessary, this gas may be analysed to assist in determining the type of fault that has generated it. The composition of gases within the bubbles changes as they move through the oil towards the gas-collecting relay.

This can be put to good use, as information on the rate of gas production may often be inferred by comparing the composition of the free gases collected with the concentrations remaining dissolved in the liquid.

The interpretation of the gas analyses is the subject of IEC 60599.

These techniques are valuable at all stages in the life of oil-filled equipment. During acceptance tests on transformers in the factory, comparison of gas-in-oil analyses before, during and after a heat run test can show if any hot-spots are present, and similarly analysis after dielectric testing can add to information regarding the presence of partial discharges or sparking. During operation in the field, the periodic removal of an oil sample and analysis of the gas content serve to monitor the condition of transformers and other oil-filled equipment.

The importance of these techniques has led to the preparation of this standard to the procedures to be used for the sampling, from oil-filled electrical equipment, of gases and oils containing gases, and for subsequent analysis.

NOTE Methods described in this standard apply to mineral insulating oils, since experience to date has been almost entirely with such oils. The methods may also be applied to other insulating liquids, in some cases with modifications.

General caution, health, safety and environmental protection

This International Standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of the standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

The mineral insulating oils which are the subject of this standard should be handled with due regard to personal hygiene. Direct contact with the eyes may cause irritation. In the case of eye contact, irrigation with copious quantities of clean running water should be carried out and medical advice sought. Some of the tests specified in this standard involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

Mercury presents an environmental and health hazard. Any spillage should immediately be removed and be properly disposed of. Consult local regulations for mercury use and handling. Mercury-free methods may be requested in some countries.

Environment

This standard is applicable to mineral oils, chemicals and used sample containers.

Attention is drawn to the fact that, at the time of writing of this standard, many mineral oils in service are known to be contaminated to some degree by PCBs. As this is the case, safety countermeasures must be taken to avoid risks to workers, the public and the environment during the life of the equipment, by strictly controlling spills and emissions. Disposal or decontamination of these oils must be carried out strictly according to local regulations. Every precaution should be taken to prevent release of mineral oil into the environment.

OIL-FILLED ELECTRICAL EQUIPMENT – SAMPLING OF GASES AND OF OIL FOR ANALYSIS OF FREE AND DISSOLVED GASES – GUIDANCE

1 Scope

This International Standard deals with the techniques for sampling free gases from gas-collecting relays and for sampling oil from oil-filled equipment such as power and instrument transformers, reactors, bushings, oil-filled cables and oil-filled tank-type capacitors. Three methods of sampling free gases and three methods of sampling oil are described; the choice between the methods often depends on the apparatus available and on the quantity of oil needed for analysis.

Before analysing the gases dissolved in oil, they must first be extracted from the oil. Three basic methods are described, one using extraction by vacuum (Toepler and partial degassing), another by displacement of the dissolved gases by bubbling the carrier gas through the oil sample (stripping), and the last one by partition of gases between the oil sample and a small volume of the carrier gas (head space). The gases are analysed quantitatively after extraction by gas chromatography; a method of analysis is described. Free gases from gas-collecting relays are analysed without preliminary treatment.

The preferred method for assuring the performance of the gas extraction and analysis equipment, considered together as a single system, is to degas samples of oil prepared in the laboratory and containing known concentrations of gases ("gas-in-oil standards") and quantitatively analyse the gases extracted. Two methods of preparing gas-in-oil standards are described.

For daily calibration checks of the chromatograph, it is convenient to use a standard gas mixture containing a suitable known amount of each of the gas components to be in a similar ratio to the common ratios of the gases extracted from transformer oils.

The techniques described take account, on the one hand, of the problems peculiar to analyses associated with acceptance testing in the factory, where gas contents of oil are generally very low and, on the other hand, of the problems imposed by monitoring equipment in the field, where transport of samples may be by un-pressurized air freight and where considerable differences in ambient temperature may exist between the plant and the examining laboratory.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60296, *Fluids for electrotechnical applications – Unused mineral insulating oils for transformers and switchgear*

IEC 60599, *Mineral oil-impregnated electrical equipment in service – Guide to the interpretation of dissolved and free gases analysis*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 5725 (all parts), *Accuracy, trueness and precision of measurement methods and results*

3 Sampling of gases from gas-collecting (Buchholz) relays

3.1 General remarks

It is important to bear in mind that receiving a qualitative and a representative sample is crucial for obtaining a reliable diagnosis of the electrical equipment. Even the most sophisticated extraction or diagnosis methods cannot overcome faulty samples.

Gas samples from relays should be taken from the equipment with the minimum delay after gas accumulation has been signalled. Changes in composition caused by the selective re-absorption of components may occur if free gases are left in contact with oil.

Certain precautions are necessary when taking gas samples. The connection between the sampling device and the sampling vessel must avoid the ingress of air. Temporary connections should be as short as possible. Any rubber or plastic tubing used should have been proved to be impermeable to gases.

Gas samples should be properly labelled (see Clause 5) and analysed without undue delay to minimize hydrogen loss (for example, within a maximum period of one week).

Oxygen, if present in the gas, may react with any oil drawn out with the sample. Reaction is delayed by excluding light from the sample, for example, by wrapping the vessel in aluminium foil or suitable opaque material.

Of the three methods described below, the syringe method is recommended. The other two methods are alternatives to be used exclusively in case of serious hindrance.

Sampling into a sampling tube by liquid displacement using transformer oil as a sealing liquid is simple, but the different solubilities of the gas components may need to be taken into account if the gas quantity is such that some oil remains in the tube.

The vacuum method requires skill to avoid contaminating the sample by leakage of air into the system. It is particularly true where the gas to be sampled may be at less than atmospheric pressure (for example, some sealed transformers).

3.2 Sampling of free gases by syringe

3.2.1 Sampling equipment

See Figure 1.

- a) Impermeable oil-resistant plastic or rubber tubing (3) provided with a connector to fit onto a suitable sampling connection of the gas-collecting relay. To avoid cross-contamination, the tubing should be used only once.

- b) Gas-tight syringes of suitable volume (1) (25 ml to 250 ml). Medical or veterinary quality glass syringes with ground-in plungers may be suitable; alternatively, syringes with oil-proof seals may be used. The syringe should be fitted with a cock enabling it to be sealed. It is often convenient to use the same syringes for both gas sampling and for oil sampling (see item b) of 4.2.1). The gas tightness of a syringe may be tested by storing an oil sample containing a measurable quantity of hydrogen for at least two weeks and analysing aliquots for hydrogen at the beginning and end of the period. An acceptable syringe will permit losses of hydrogen of less than 2,5 % per week. General experience suggests that all-glass syringes leak less than those using plastic seals. Improvement of the gas tightness may be obtained by the use of a lubricant such as a light grease or transformer oil.

It is a good practice to test the integrity of syringes and stopcock system before the sampling. A recommended procedure appears in Annex A.

- c) Transport containers which should be designed to hold the syringe firmly in place during transport but allow the syringe plunger freedom to move, and prevent its tip from contacting the container whatever its position during transportation.

3.2.2 Sampling procedure

The apparatus is connected as shown in Figure 1. The connections should be as short as possible and filled with oil at the start of sampling.

Sampling valve (5) is opened. If sampling from a gas-collecting relay on a transformer fitted with a conservator, a positive pressure will exist; the three-way cock (4) is carefully turned to position A and the oil in the connecting tubing (3) allowed to flow to waste (7). When gas reaches the three-way cock (4), the latter is turned to position B to connect the pre-lubricated syringe (1). Cock (2) is then opened and the syringe allowed to fill under the hydrostatic pressure, taking care that its plunger is not expelled. When a sufficient sample has been taken, cock (2) and sampling valve (5) are closed and the apparatus is disconnected.

The oil in the syringe is expelled by inverting the syringe and applying gentle pressure to the plunger.

Label carefully the sample (see Clause 5).

3.3 Sampling of free gases by displacement of oil

This method is reliable only where the gas sample is at or above atmospheric pressure. The apparatus is shown in Figure 2.

The sampling tube (28), typically of 100 ml capacity, is preferably of glass since the operator can then see how much oil remains in it during gas sampling. The sampling tube is filled with oil from the transformer on site. Before being used as described below, the connecting tube (3) should also be filled with oil.

The open end of the connecting tube (3) is fitted onto the gas sampling valve (5). The sampling valve and inlet cock of the sampling tube are opened. The sampling tube is inclined so that its closed end is the lowest point. The outlet cock on the sampling tube is then opened, allowing oil to run out to waste (7), drawing first any oil from the connection between relay and sampling valve, and the gas from the relay, into the sampling tube.

Sampling is complete when the gas collecting relay is completely filled with oil or when nearly all oil has gone from the sampling tube.

Both cocks (2) on the sampling tube and the sampling valve (5) are closed and then the connections removed.

3.4 Sampling of free gases by vacuum

The apparatus is connected as shown in Figure 3. With the equipment sampling valve closed, cocks (1), (2) and (10) open, and the three-way cock (4) turned to position A, the vacuum pump (12) is allowed to evacuate the connecting tubing, the trap and the sampling vessel.

A satisfactory vacuum will be below 100 Pa. The system should be checked for leaks by closing the pump suction cock (10) and observing that no appreciable change in vacuum occurs. Over a time equal to that which will be taken for sampling, the pressure should not increase by more than 100 Pa. Similarly, the stopcock (1) on the sampling tube should be vacuum tight to the same degree over several weeks.

If the connecting tubing between the equipment sampling valve (5) and the gas-collecting relay is filled with oil, the three-way cock (4) is turned to position (B). The equipment sampling valve (5) is carefully opened and oil allowed to flow into the trap (9). When the end of the oil stream is observed to reach the three-way cock (4), it is turned to position D to evacuate the oil from it. Thereafter, cock (4) is turned to position C. When sampling is complete, cock (1) is closed first, then the equipment sampling valve (5) closed and the apparatus disconnected.

If the connecting tubing between the equipment and the sampling valve is empty of oil, the procedure for draining oil is omitted and the three-way cock (4) used in position C after evacuating and testing that the apparatus is leak tight.

4 Sampling of oil from oil-filled equipment

4.1 General remarks

It is important to bear in mind that receiving a qualitative and a representative sample is crucial for obtaining a reliable diagnosis of the electrical equipment. Even the most sophisticated extraction or diagnosis methods cannot overcome faulty samples.

Warning: When sampling oil, precautions should be taken to deal with any sudden release of oil and avoid oil spillage.

Of the three methods described below, the method of sampling by syringe is recommended. The other two methods are alternatives to be used in case of difficulties.

Sampling into glass sampling tubes is also suitable provided they are fitted with sufficient lengths of rubber tubing acting as expansion devices.

Stainless steel sampling tubes fitted with valves are very robust and are not affected by large temperature changes and can be used without expansion devices.

Sampling in glass bottles is also adequate provided the bottles are fitted with a suitable cap which allows oil expansion. Sampling into bottles is simple, requires little skill, and is adequate for many purposes such as routine sampling on a large scale from equipment on site. The use of bottles (0,5 l to 2,5 l capacity) may be preferred where comparatively large samples of oil are required. When using the glass bottle sampling method, care should be taken to minimize air contact with the sample.

The methods described are suitable for large oil-volume equipment such as power transformers. With small oil-volume equipment, it is essential to ensure that the total volume of oil drawn off does not endanger the operation of the equipment.

The selection of points from which samples are drawn should be made with care. Normally, the sample should be taken from a point where it is representative of the bulk of the oil in the equipment. It will sometimes be necessary, however, to draw samples deliberately where they are not expected to be representative (for example, in trying to locate the site of a fault).

Samples should be taken with the equipment in its normal condition. This will be important in assessing the rate of gas production.

Some of the dissolved oxygen present in the oil sample may be consumed by oxidation. The reaction can be delayed by exclusion of light (for example, by wrapping a clear glass sampling vessel in an opaque material) but, in any case, the analysis should be carried out as soon as possible after sampling.

NOTE 1 When sampling from bushings, the manufacturer's instructions should be followed carefully. Failure to do so may lead to serious damage and bushing failure. The oil sampling should be carried out on de-energized bushings. When sampling, precautions should be taken to deal with any sudden release of oil. Samples should be taken with the off-load equipment in its normal position in order to assess correctly the bushing condition.

NOTE 2 For transformers with two sampling valves, the following procedure should be used: open the outer valve first, followed by the second one. This is particularly important to avoid entrance of air into the transformers.

4.2 Sampling of oil by syringe

4.2.1 Sampling equipment

- a) Impermeable oil-proof plastic or rubber tubing to connect the equipment to the syringe. This should be as short as possible. A three-way cock should be inserted in the tubing.

The connection between the tubing and the equipment will depend upon the equipment. If a sampling valve suitable for fitting to a tubing has not been provided, it may be necessary to use a drilled flange or a bored oil-proof rubber bung on a drain or filling connection.

NOTE Sampling by syringe is the procedure recommended for bushings by IEC SC36A. In the case of bushings fitted with a sampling point at the mounting flange, the described procedure applies.

In the case of bushings not fitted with a sampling point at mounting flange, it may be possible to take a sample from the top of the bushing. The manufacturer's instructions should be consulted to determine a suitable position. Insert one end of the sampling tube into the bushing, from the top, and connect the other end to the three-way stopcock on the syringe, using plastic coupling, then follow the same procedure.

In the case of bushings pressurized at ambient temperature, the procedure is not applicable, and reference should be made to the instructions of the equipment manufacturer.

- b) Graduated gas-tight syringes of a size suitable for containing an adequate oil sample volume (20 ml to 250 ml) fitted with a cock or an obturator so that it may be sealed. See item b) of 3.2.1 for checking the gas-tightness of the syringe. The size of sample required depends on the likely concentration of gas in the sample, the analytical techniques and the sensitivity required.
- c) Transport containers which should be designed to hold the syringe firmly in place during transport but allow the syringe plunger freedom to move and prevent its tip from contacting the container whatever its position during transportation.

4.2.2 Sampling procedure

See Figure 4.

- a) The blank flange or cover (11) of the sampling valve is removed and the outlet cleaned with a lint-free cloth to remove all visible dirt. The apparatus is then connected as shown in Figure 4a, and the equipment sampling valve (5) opened.
- b) The three-way cock (4) is adjusted (position A) to allow 1 l to 2 l of oil to flow to waste (7) (or less, see note).
- c) The three-way cock (4) is then turned (position B) to allow oil to enter the syringe slowly (Figure 4b). The plunger should not be withdrawn but allowed to move back under the pressure of the oil.
- d) The three-way cock (4) is then turned (position C) to allow the oil in the syringe to flow to waste (7) and the plunger pushed to empty the syringe. To ensure that all air is expelled from the syringe, it should be approximately vertical, nozzle upwards, as shown in Figure 4c. Confirm that the inner surfaces of the syringe and plunger are completely oiled.
- e) The procedure described in steps 3) and 4) of this subclause is then repeated until no gas bubble is present. Then the three-way stopcock (4) is turned to position B and the syringe filled with oil (Figure 4d).
- f) The cock (2) on the syringe and the sampling valve (5) are then closed.
- g) The three-way stop-cock (4) is turned to position C and the syringe disconnected (Figure 4).

Label carefully the sample (see Clause 5).

NOTE 1 In the case of small oil-volume equipment, the procedure described in step b) of this subclause is not applicable, and lower volumes should be removed. Reference should be made to the instructions of the equipment manufacturer.

NOTE 2 It is good practice to avoid contamination of the outer surface of the plunger and inner surfaces of the syringe by dust or sand. Those particles can affect the sealing properties of the syringe. This kind of contamination can be originated by dusty winds or from the handling of the syringe.

NOTE 3 In the case of sealed transformers, if a bubble appears in the syringe directly after sampling, it is recommended to resample.

4.3 Sampling of oil by sampling tube

4.3.1 Sampling equipment

- a) Impermeable oil-proof plastic or rubber tubing to connect the equipment to the sampling tube. This should be as short as possible.

The connection between the tubing and the equipment will depend upon the equipment. If a sampling valve suitable for fitting to a tubing has not been provided, it may be necessary to improvise by using a drilled flange, or a bored oil-proof rubber bung on a drain or filling connections.

- b) Glass or metal sampling tube, typically of volume 250 ml to 1 l. It may be closed either by cocks or pinch cocks on impermeable oil-resistant tubing or by valves.

A sampling tube and its seal design is acceptable if the loss of hydrogen of the sample contained is less than 2,5 % each week.

The size of sample required depends on the likely concentration of gas in the sample, the analytical technique and the sensitivity required.

- c) Transport containers, which should be designed to hold the sampling tubes firmly in place during transport.

4.3.2 Sampling procedure

See Figure 5.

- a) The blank flange or cover (11) of the sampling valve is removed and the outlet cleaned with a lint-free cloth to remove all visible dirt. The device is then connected as shown in Figure 5.
- b) The cocks (2) on the sampling tube (28) are opened and the equipment sampling valve (5) is carefully opened so that oil flows through the sampling tube to waste (7).
- c) After the sampling tube (28) has been completely filled with oil, about 1 l to 2 l are allowed to flow to waste (7) (see Note 2).
- d) The oil flow is then closed by shutting off firstly the outer cock (2), then the inner one (2) and finally the sampling valve (5).
- e) The sampling tube (28) is then disconnected and the sample carefully labelled (see Clause 5).

NOTE 1 If a glass sampling tube with integral glass cocks is used, it is preferable to drain 1 ml or 2 ml of oil from it prior to transporting it back to the laboratory in order to avoid breaking the tube in the event of it being exposed to a rise in ambient temperature. Record on the label that this has been done.

NOTE 2 In the case of small oil-volume equipment, the procedure described in step c) of this subclause is not applicable. Reference should be made to the instructions of the equipment manufacturer.

4.4 Sampling of oil by bottles

4.4.1 Sampling equipment

- a) Impermeable oil-proof plastic or rubber tubing to connect the equipment to the bottle. This should be as short as possible.

The connection between the tubing and the equipment will depend upon the equipment. If a sampling valve suitable for fitting to a tubing has not been provided, it may be necessary to improvise by using a drilled flange, or a bored oil-proof rubber bung on a drain or filling connections.

- b) Glass or metal bottles capable of being sealed gas-tight, typically of volume 0,5 l to 2,5 l. Suitable bottles have, for example, screwed plastic caps holding a conical polyethylene seal (see Figure 6b). A bottle and seal design is acceptable if it permits losses of hydrogen of less than 2,5 % each week.
- c) Transport containers, designed to protect the bottle during transport.

4.4.2 Sampling procedure

See Figure 6a.

- a) The blank flange or cover (11) of the equipment sampling valve is removed and the outlet cleaned with a lint-free cloth to remove all visible dirt.

- b) Connect the oil-proof plastic or rubber tubing (3) to the equipment.
- c) The sampling valve (5) is carefully opened and about 1 l to 2 l of oil allowed to flow to waste (7) through the tubing (3) ensuring that all gas bubbles are eliminated before the oil sample is collected (see the note at the end of this subclause).
- d) Place the end of the tubing (3), with the oil still flowing, at the bottom of the sampling bottle and allow the bottle to fill slowly.
- e) Allow about one bottle volume to overflow to waste (7), then withdraw the tubing (3) slowly with the oil still flowing.
- f) Close the sampling valve (5) and disconnect the tubing.
- g) Tilt the bottle to allow the oil level to fall a few millimetres from the rim so as to leave a small expansion volume. Place the bottle cap securely in position and label the sample (see Clause 5).

NOTE In the case of small oil-volume equipment, the procedure described in steps c) and e) of this subclause is not applicable. Reference should be made to the instructions of the equipment manufacturer.

4.5 Disposal of waste oil

Waste oil shall be disposed of according to local regulations.

5 Labelling of samples

Oil and gas samples should be properly labelled before dispatch to the laboratory.

The following information is necessary (whenever it is known).

Transformer	Sampling
- customer	- sampling date
- location	- sampling point
- transformer number	- sampling person
- manufacturer	- reason for analysis (routine or other)
- general type (power, instrument or industrial)	- transformer non-energized, off-load energized or on-load
- rated MVA	- top oil temperature
- main voltage	- bottom oil temperature
- type of OLTC	Oil
- date of commissioning	- product name
	- volume of oil
	- date of last oil treatment

The following additional information is desirable:

- ambient temperature, reading of winding temperature indicator, reading of MVA or load current or % load, operation of pumps, mode of communication of its tap-changer with the main tank, oil preservation system (conservator, nitrogen blanket, etc), and any changes in operational conditions or any maintenance carried out since last sampling.

6 Preparation of gas-in-oil standards

As noted in Clause 1, the preferred method of assuring the performance of the entire system for gas extraction and analysis is to analyse oils containing known concentrations of gases (gas-in-oil standards).

Two methods of preparing gas-in-oil standards are described below, a general method and a simpler method. The first method has the benefit of producing much larger quantities of oil.

NOTE If gas-in-oil standards are commercially available, they can be used provided they fulfill quality assurance requirements.

6.1 First method: preparation of a large volume of gas-in-oil standard

6.1.1 Equipment

A suitable design of apparatus consists of (see Figures 7a, 7b, 7c):

- a magnetic stirrer (12);
- a 5 l oil vessel (13) equipped with three side-arm outlets, two of which are fitted with high-vacuum stop-cocks (1) and (2) and one with a rubber septum (16);
- a 6 l reservoir (14) equipped with a high-vacuum stopcock (3) connected with a 75 ml splash bulb (15) and a high-vacuum stopcock (4);
- a mercury displacement system (17) consisting of two 500 ml mercury glass bulbs connected with a high-vacuum oil-resistant flexible rubber tubing.

The gas-injection system (Figures 7e to 7g) consists of

- a set of gas tight syringes of appropriate volumes (18);
- a plastic three-way stopcock of standard type (7), modified to minimize dead volume by inserting pieces of stainless steel tubing (approximately 0,4 mm inner diameter and 1,5 mm outer diameter) into the inner branches and connected to a gas-injecting needle (10) (approximately 10 cm long, 0,25 mm outer diameter) to go through the rubber septum into the oil flask (16);
- gas cylinders equipped with a pressure reducer and a valve (20);

NOTE Gas cylinders containing each of the individual gases to be injected can be used. Ready-made mixtures of these gases can also be obtained from some gas suppliers.

- a length of oil-resistant flexible tubing connecting the gas cylinder to the three-way stopcock (7) and incorporating a hypodermic needle (approximately 0,3 mm inner diameter and 0,6 mm outer diameter). The latter acts as a leak to a water bubbler (21) which is used to check the absence of back diffusion.

NOTE Replace the length of tubing connecting the gas cylinder to the three-way stopcock after each gas change.

The oil sampling system (Figures 7h to 7k) consists of glass syringes (19) of suitable capacity equipped with two three-way plastic stopcocks (8) and (9) and an oil-extracting needle (11) (approximately 0,6 mm inner diameter, 1,0 mm outer diameter and 120 mm long).

6.1.2 Procedure

NOTE All the circled numbers refer to Figures 7a to 7k. The positions A to D of the three-way stop-cocks (7, 8 and 9) are illustrated in Figure 7d.

6.1.2.1 Degassing the oil

- a) Thoroughly clean all the glassware before assembling.
- b) Secure the rubber septum and the flexible connections with pieces of twisted metal wire.
- c) Clean, if necessary, approximately 1 l of mercury with pentane and filter through a finely pierced filter paper.
- d) Adapt the oil reservoir (14), the splash bulb (15) and stop-cock (4) to the 5 l vessel (13). Connect the system to the vacuum pump (V) and evacuate the splash bulb (15) and the 5 l oil vessel (13) by opening stopcocks (2) and (4).
- e) Fill the oil reservoir (14) with mineral insulating oil complying with IEC 60296 and allow oil to flow slowly through stopcock (3) into the 5 l vessel (13) until it is full. Then close stopcock (2) and remove the oil reservoir (14) and the splash bulb.

NOTE If the procedure is followed carefully (filling time about 4 h,) the oil in the vessel will be virtually gas-free.

- f) Turn the oil vessel to bring it into the position shown in Figure 7c. Place a wet cloth over it to prevent its temperature rising and attach the mercury displacement system (17) to stopcock (1). Introduce 750 ml of mercury into the system with stopcock (6) closed.
- g) Connect the system to vacuum (V), open stopcock (5) and evacuate the section between stopcocks (1) and (6). Open stopcock (6) to allow mercury to rise up to stopcocks (1) and (5), and then close stopcock (5). Raise the left bulb of the system (see Figure 7c) so that the mercury level is above the oil flask. Open stopcock (1). Remove the wet cloth and switch on the magnetic stirrer (12).

6.1.2.2 Preparation of gas-in-oil standards

See Figures 7c to 7g.

- a) To inject the gases, attach the modified three-way stopcock (7), in position B, to the gas-injecting needle (10). Then push the needle through the rubber septum (16) into the oil. Turn the three-way stopcock to position D to purge the needle and stopcock with oil, then turn it to position B. Attach to stopcock (7) the barrel of a precision calibrated gas-tight syringe (18) of appropriate volume (precision within 1 %) and the gas cylinder connecting tubing (see Figure 7e). Allow a gentle flow of gas to flush the barrel, then slowly push the plunger several times into the barrel, finally down to the volume of gas to be injected, making sure there is continuous bubbling through the needle leak (21).
- b) Switch stopcock (7) to the injection position A, lower the mercury level below the needle tip, and push the plunger to inject the gas volume into the oil. Switch stopcock (7) to position B and raise the left mercury bulb above the oil vessel.
- c) Repeat the same procedure with each of the gases to be dissolved then remove the gas needle and stopcock (7).

When all the gases are dissolved, switch off the magnetic stirrer. Record the atmospheric pressure and temperature.

NOTE Instead of adding individual gases, it is more convenient to use a standard gas mixture, preferably containing all the gases listed in 8.1, in proportions representative of those found in the sampled oil.

6.1.2.3 Sampling of gas-in-oil standards

See Figures 7h to 7k.

- a) To remove oil samples from the 5 l vessel (13), attach two three-way stopcocks (8) and (9) turned in position B, according to Figure 7b, to the liquid-withdrawing needle (11) and push the needle through the rubber septum (16). With the mercury level up, switch stopcocks (8) and (9) to the draw-off position D to purge them with oil (see Figure 7i).
- b) Attach a syringe (19) and turn both stopcocks (8) and (9) to position A, and draw out a suitable volume of oil (see Figure 7j). Then turn stopcock (8) to position C and stopcock (9) to position B and remove the syringe together with stopcock (8) (see Figure 7k).

To remove more oil, additional mercury must be added to the mercury displacement system.

6.1.3 Calculation

Calculate the concentration of each gas “i” dissolved as follows:

$$C_i = V_i/V \times 10^6$$

where

C_i is the concentration of gas “i”, in $\mu\text{l/l}$;

V_i is the volume of gas “i” injected, corrected to 20 °C and 101,3 kPa in ml;

V is the exact volume of oil in the 5 l oil flask, in ml.

6.2 Second method: preparation of gas-in-oil standards in a syringe or a vial

See Figures 8a to 8g.

6.2.1 Equipment

Equipment consists of

- a set of gas-tight syringes of appropriate volume (2);
- a set of glass syringes of appropriate volume (1);
- a set of gas-injecting needles (4);
- three-way plastic stopcocks (3);
- gas cylinders equipped with a pressure reducer and a valve (20).

NOTE Gas cylinders containing each of the individual gases to be injected may be used. Ready-made mixtures of these gases can also be obtained from some gas suppliers.

6.2.2 Procedure

- a) Bubble argon through 1 l of oil for 1 h under agitation, adjusting gas flow to maintain agitation of the oil in order to purge all the other dissolved gases. Degas this argon-saturated oil under a vacuum down to 1 Pa over an 8 μm -filter, in a 2 l vessel (residual contents of 500 $\mu\text{l/l}$ O_2 and 2 000 $\mu\text{l/l}$ N_2 are acceptable). When the oil is completely degassed, break the vacuum with argon up to 20 kPa.

- b) Weigh a 100 ml glass syringe containing 10 ml of glass beads. Connect the syringe to the bottom of the 2 l vessel and purge the syringe twice with 20 ml of oil. Make sure the cylinder and plunger are completely wetted with oil. Fill the syringe with 90 ml of degassed oil.
- c) Connect a gas-tight syringe of the required volume with a needle long enough to introduce a gas mixture in the syringe cylinder part, for instance of 115 mm length, to a cylinder containing a standard gas mixture, and purge 4 times with the standard gas. Fill the syringe with a known volume of standard gas. Insert the needle through the tip of the oil syringe and transfer the volume of standard gas into the oil syringe. Reweigh to determine the actual oil volume. Wrap the oil syringe with aluminium foil to avoid degradation with daylight and attach it to a laboratory 3-D shaker. Shake for 1 h or until all gasses are dissolved.

NOTE 1 Any other procedure that assists dissolution of gases in oil (for example, mixing with magnetic stirrer or other) is suitable and can be used.

- d) Record the atmospheric pressure and temperature, also refer to the note in 6.1.2.2. Calculate the dissolved gas concentration from oil volume, injected calibration gas volume, temperature and atmospheric pressure, as in 6.1.3. Express the quantities of gases injected in μmoles , or in μl converted to normal conditions (20 °C; 101,3 kPa).
- e) Repeat the procedure in order to obtain at least 3 gas-in-oil samples (in the case of headspace only) having concentrations that allow a calibration curve to be drawn covering the values expected in the unknown samples.

NOTE 2 If a total gas-saturated mixture is desired, pull dry CO₂-free air or nitrogen into the syringe to make, with the gases already injected, a total of 10 %, to 8 % of the oil volume according to the gas chosen (see Figure 8f).

Turn the three-way (3) stopcock to position A and shake the syringe again until the air or nitrogen is dissolved. Cooling the syringe in a refrigerator accelerates the dissolution of the gas in the oil.

NOTE 3 A vial may be used instead of a syringe. Fill the vial with the same amount of degassed oil as that needed when the ordinary analysis is performed, with the exact volume of oil measured by weight. Introduce into this vial known quantities of a calibrated gas mixture, so that the concentrations in the oil are in the same range as in the unknown field sample.

7 Extraction of gases from oil

7.1 General remarks

Removal of dissolved gases from oil for analysis may be accomplished either by vacuum extraction, stripping or head space.

The multi-cycle vacuum extraction (Toepler) method is described in 7.2.

The single-cycle vacuum extraction (partial degassing) method is described in 7.3.

The stripping method is described in 7.4.

The head space method is described in 7.5.

7.2 Multi-cycle vacuum extraction using Toepler pump apparatus

In this method, an operating procedure which attempts to remove as much as possible of the dissolved gas from the oil is used. It is normally possible to remove about 97 % of the more soluble gases and even higher percentages of the less soluble gases. Such a small imperfection is rarely significant when considering overall accuracies, but, in any case, the preferred method of calibration using gas-in-oil standards takes account of incomplete extraction.

NOTE To calibrate this method with gas-in-oil standards, the procedure described in 6.1 for preparation of argon-free standards should be used, since the calculation will be affected by the amount of dissolved argon that will be extracted but not detected by the gas chromatograph.

7.2.1 Toepler pump extraction apparatus

An example of a suitable design is shown in Figure 9a. Note that Figure 9b includes recommended volumes. In this design, oil containing gas in solution is injected through a septum (9). Alternatively, after the equipment has been evacuated, oil may be withdrawn from an oil sample bottle via a tube attached to valve (V8) which has previously been filled with oil (see Figure 9c).

The Toepler pump extraction apparatus shall

- a) be capable of subjecting the liquid to a vacuum less than 10 Pa;
- b) be vacuum-tight. The vacuum tightness of the whole system may be verified by carrying out the extraction procedure but without introducing oil, as follows.
Carry out steps b), c) and d) of 7.2.2. Omit step e). Continue with steps f), g) and h) as if oil were present. After compression of gas to atmospheric pressure in step h), the amount of gas should be less than 0,1 ml;
- c) permit the measurement of extracted gas to be made to the nearest 0,05 ml or better, at normal temperature and pressure.

In addition:

- d) all tubing connecting the degassing flask (3) to the gas collection flask (2) (in Figure 9a) shall be of large bore, at least 5 mm internal diameter and as short as practicable;
- e) the vacuum gauge used cannot be of a type that operates at high temperature or uses ionizing radiation (thermocouple, ionization or Penning gauges) since these can produce gases of the types being measured by cracking the oil vapours present in the system. Since it may react with extracted gases, a Pirani gauge is not suitable either. A sensor based on capacitance changes between two chambers is suitable. It is recommended that two sensors be used, one in the vacuum chamber, the second for measuring gas volumes at atmospheric pressure.

Further requirements are noted in 7.3 in which the partial degassing method is described.

Since the detailed design of this apparatus is not standardized, it is necessary to establish an operation which will ensure adequate extraction of all components of the dissolved gas. The main parameters that need to be established are the number of degassing cycles (strokes) of the Toepler pump that should be used and the time for which each degassing cycle should last. This operation is preferably established by degassing a gas-in-oil standard as follows.