

Guide for the sampling of gases and of oil from oil-filled electrical equipment and for the analysis of free and dissolved gases

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Contents

	Page
Cooperating organizations	Inside front cover
National foreword	ii
Foreword	2
Text of EN 60567	3
National annex NA (informative) Committees responsible	Inside back cover
National annex NB (informative) Cross-references	Inside back cover

National foreword

This British Standard has been prepared under the direction of the Cables and Insulation Standards Policy Committee and is the English language version of EN 60567:1992 *Guide for the sampling of gases and of oil from oil-filled electrical equipment and for the analysis of free and dissolved gases*, published by the European Committee for Electrotechnical Standardization (CENELEC). It is identical with IEC 567:1992 published by the International Electrotechnical Commission (IEC).

The guide 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.

BS 5574:1978 is identical with IEC 567:1977. On publication of this standard, BS 5574 will be withdrawn.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 40, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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English version

Guide for the sampling of gases and of oil from oil-filled electrical equipment and for the analysis of free and dissolved gases

(IEC 567:1992)

Guide d'échantillonnage de gaz et d'huile dans les matériels électriques immergés, pour l'analyse des gaz libres et dissous
(CEI 567:1992)

Anleitung für die Probenahme von Gasen und von Öl aus ölfüllten elektrischen Betriebsmitteln und für die Analyse freier und gelöster Gase
(IEC 567:1992)

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CENELEC

European Committee for Electrotechnical Standardization
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Foreword

The text of document 10(CO)260, as prepared by IEC Technical Committee N° 10 “Fluids for electrotechnical applications”, was submitted to the IEC-CENELEC parallel vote in September 1991.

The reference document was approved by CENELEC as EN 60567 on 16 June 1992.

The following dates were fixed:

- latest date of publication of an identical national standard (dop) 1993-07-01
- latest date of withdrawal of conflicting national standards (dow) 1993-07-01

Annexes designated “normative” are part of the body of the standard. In this standard, Annex ZA is normative.

Contents

	Page
Foreword	2
Introduction	3
1 Scope	3
2 Normative references	4
3 Sampling of gases from gas-collecting (Buchholz) relays	4
4 Sampling of oil from oil-filled equipment	5
5 Labelling of samples	7
6 Preparation of gas-in-oil standards	7
7 Extraction of gases from oil	9
8 Gas analysis by gas-solid chromatography	13
9 Sensitivity and precision requirements	15
Annex A (informative) Correction for incomplete gas extraction in partial degassing method by calculation	38
Annex ZA (normative) Other international publications quoted in this standard with the references of the relevant European publications	39
Figure 1 — Sampling of gas by syringe	19
Figure 2 — Sampling of free gases by oil displacement	20
Figure 3 — Sampling of free gases by vacuum	21
Figure 4 — Sampling of oil by syringe	22
Figure 5 — Sampling of oil by sampling tube	23
Figure 6 — Sampling of oil by bottle	24
Figure 7 — First method of preparing gas-in-oil standards	25
Figure 8 — Second method for preparing gas-in-oil standards	27
Figure 9 — Example of a Toepler pump extraction apparatus	28
Figure 10 — Types of glass strippers	31
Figure 11 — Stainless steel stripper	33
Figure 12 — Schematic arrangement for connecting an oil stripper to a gas chromatograph	35
Figure 13 — Schematic arrangement for gas chromatography	37
Table 1 — Examples of suitable column assembly	18

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 599.

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 guide 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 guide 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.

1 Scope

This guide 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. Two basic methods are described, one using extraction by vacuum and the other by stripping (displacing) the dissolved gases by bubbling the carrier gas through the oil sample. 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 component to be analysed and diluted by nitrogen or by the carrier gas.

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 unpressurised air freight and where considerable differences in ambient temperature may exist between the plant and the examining laboratory.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

IEC 296:1982, *Specification for unused mineral insulating oils for transformers and switchgear*.

IEC 599:1978, *Interpretation of the analysis of gases in transformers and other oil-filled electrical equipment in service*.

ISO 5725:1986, *Precision of test methods. Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

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

3.1 General remarks

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 reabsorption 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 (e.g. 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 frequently preferred.

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 (e.g. some sealed transformers).

3.2 Sampling of free gases by syringe

3.2.1 Sampling equipment (see Figure 1)

1) Impermeable oil-resistant plastic or rubber tubing (3) provided with a connector to fit on to a suitable sampling connection of the gas-collecting relay.

2) 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 2 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.

3) Transport containers which should be designed to hold the syringe firmly in place during transport but allow the syringe plunger freedom to move.

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 prelubricated 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 on to 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 (5) 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 the 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

WARNING: When sampling oil precautions should be taken to deal with any sudden release of oil.

Of the three methods described below, the method of sampling by syringe is the most adequate method of oil sampling and is suitable irrespective of the mode of transport of samples.

Sampling into glass sampling tubes is also suitable provided they are fitted with sufficient lengths of rubber tubings 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 litre to 2,5 litre 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 (e.g. 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.

4.2 Sampling of oil by syringe

4.2.1 Sampling equipment

1) 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.

2) Graduated gas-tight syringes of 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 2) 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.

3) Transport containers which should be designed to hold the syringe firmly in place during transport but allow the syringe plunger freedom to move.

4.2.2 Sampling procedure (see Figure 4)

1) 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 4 a), and the equipment sampling valve (5) opened.

2) The three-way cock (4) is adjusted (position A) to allow 1 litre to 2 litres of oil to flow to waste (7) (see note).

3) The three-way cock (4) is then turned (position B) to allow oil to enter the syringe slowly [Figure 4 b)]. The plunger should not be withdrawn but allowed to move back under the pressure of the oil.

4) 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 4 c). Confirm that the inner surfaces of the syringe and plunger are completely oiled.

5) 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 4 d)].

6) The cock (2) on the syringe and the sampling valve (5) are then closed.

7) The three-way stopcock (4) is turned to position C and the syringe disconnected [Figure 4 e)].

Label carefully the sample (see clause 5).

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

4.3 Sampling of oil by sampling tube

4.3.1 Sampling equipment

1) 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 connection.

2) Glass or metal sampling tube, typically of volume 250 ml to 1 litre. 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.

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

4.3.2 Sampling procedure (see Figure 5)

1) 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.

2) 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).

3) After the sampling tube (28) has been completely filled with oil, about 1 litre to 2 litres are allowed to flow to waste (7) (see note 2).

4) 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).

5) 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 that this has been done on the label.

NOTE 2 In the case of small oil-volume equipment, the procedure described in step 3 of this subclause is not applicable. Reference should be made to the instructions of the equipment manufacturer.

4.4 Sampling of oil by bottle

4.4.1 Sampling equipment

1) 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 connection.

2) Glass or metal bottles capable of being sealed gas-tight, typically of volume 0,5 litre to 2,5 litres. Suitable bottles have, for example, screwed plastic caps holding a conical polyethylene seal [see Figure 6 b)]. A bottle and seal design is acceptable if it permits losses of hydrogen of less than 2,5 % each week.

3) Transport containers, designed to protect the bottle during transport.

4.4.2 Sampling procedure [see Figure 6 a)]

1) 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.

2) Connect the oil-proof plastic or rubber tubing (3) to the equipment.

3) The sampling valve (5) is carefully opened and about 1 | to 2 | 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 sub-clause).

4) 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.

5) Allow about one bottle volume to overflow to waste (7), then withdraw the tubing (3) slowly with the oil still flowing.

6) Close the sampling valve (5) and disconnect the tubing (3).

7) 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 3 and 5 of this subclause is not applicable. Reference should be made to the instructions of the equipment manufacturer.

5 Labelling of samples

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

The following information is necessary:

- customer or plant;
- identification of equipment;
- volume of oil in equipment;
- reason for sampling;
- date and time of sampling;
- temperature of sample when drawn (in the case of oil only);
- point where sample is taken.

The following additional information is desirable:

- make of oil (if known);
- load at time of sampling;
- other pertinent data: e.g. reading of winding temperature indicator, type of tapchanger (if fitted) and mode of its communication with main tank, oil preservation system (conservator, nitrogen blanket, etc), operation of pumps, 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.

6.1 First method (see Figure 7)

6.1.1 Equipment

1) A suitable design of apparatus consists of [see Figure 7 a), Figure 7 b), Figure 7 c)]:

- a magnetic stirrer (12);
- a 5 litre oil vessel (13) equipped with three side arm outlets, two of which are fitted with high-vacuum stopcocks (1) and (2) and one with a rubber septum (16);
- a 6 litre 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.

2) The gas-injection system [Figure 7 e) to Figure 7 g)] 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 which incorporate 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.

3) The oil sampling system [Figure 7 h) to Figure 7 k)] 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 Figure 7 a) to Figure 7 k). The positions A to D of the three-way stopcocks (7), (8) and (9) are illustrated in Figure 7 d).

6.1.2.1 Degassing the oil [Figure 7 a) to Figure 7 c)]

- 1) Thoroughly clean all the glassware before assembling.
- 2) Secure the rubber septum and the flexible connections with pieces of twisted metal wire.
- 3) Clean, if necessary, approximately 1 litre of mercury with pentane, and filter through a finely pierced filter paper.

4) Adapt the oil reservoir (14), the splash bulb (15) and stopcock (4) to the 5 litre vessel (13). Connect the system to the vacuum pump (V) and evacuate the splash bulb (15) and the 5 litre oil vessel (13) by opening stopcocks (2) and (4).

5) Fill the oil reservoir (14) with mineral insulating oil complying with IEC 296 and allow oil to flow slowly through stopcock (3) into the 5 litre vessel (13) until it is full. Then close stopcock (2), and remove the oil reservoir (14) and the splash bulb (15).

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

6) Turn the oil vessel to bring it into the position shown in Figure 7 c). 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.

7) 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 7 c)] 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 [Figure 7 c) to Figure 7 g)]

1) 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 7 e)]. 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).

2) 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.

3) 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 (12).

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 [Figure 7 h) to Figure 7 k)]

- 1) To remove oil samples from the 5 litre vessel (13), attach two three-way stopcocks (8) and (9) turned in position B, according to Figure 7 h), 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 7 i)].
- 2) Attach a syringe (19) and turn both stopcocks (8) and (9) to position A, and draw out a suitable volume of oil [see Figure 7 j)]. Then turn stopcock (8) to position C and stopcock (9) to position B, and remove the syringe together with stopcock (8) [see Figure 7 k)].

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:

$$C_i = \frac{V_i}{V} \times 10^6$$

where:

- C_i is the concentration of gas “i”, in microlitres per litre
- V_i is the volume of gas “i” injected, corrected to 20 °C and 101,3 kPa in millilitres
- V is the exact volume of oil in the 5 litre oil flask, in millilitres

6.2 Second method [see Figure 8 a) to Figure 8 g)]

6.2.1 Equipment

- 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);
- a water bubbler (21);
- a length of silicone rubber tubing (22).

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

- 1) Attach to the glass syringe (1) a three-way stopcock (3) and fill the syringe with degassed mineral insulating oil complying with IEC 296, then turn stopcock (3) to position A [see Figure 8 b)].
 - 2) Connect a length of silicone rubber tubing (22) to the gas pressure-reducer valve (20) and to the leak to water bubbler (21) [Figure 8 a)].
 - 3) Open the gas-cylinder valve carefully and flush the silicone rubber tubing carefully (22) by bubbling through the water.
 - 4) Attach a gas-injecting needle (4) to a precision gas-tight syringe (2). Then push the needle through the rubber silicone tubing [see Figure 8 a)] and rinse the gas syringe (2) carefully by pushing the plunger up and down several times while still purging the silicone rubber tubing with gas.
 - 5) Withdraw the gas syringe with its needle and introduce it through the opened three-way stopcock (3), turned in position B. Inject a known amount of calibration gas [Figure 8 c)].
- Record the atmospheric pressure and temperature.

Also refer to the note 1 in 6.1.2.2.

- 6) Turn stopcock (3) to position A and shake the syringe (1) until the calibration gas bubbles are dissolved [Figure 8 d)].

Homogeneity is best achieved by attaching a second oil-filled syringe to stopcock (3) [Figure 8 e)] and moving the oil between the two syringes to mix the oil.

- 7) 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 8 f)].

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.

- 8) Calculate the dissolved gas concentration from oil volume, injected calibration gas volume, temperature and atmospheric pressure, as in 6.1.3.

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 or by stripping.

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

The single-cycle vacuum extraction method is described in 7.3.

The stripping method is described in 7.4.

7.2 Multi-cycle vacuum extraction using Toepler pump apparatus

In this method, an operating procedure is used which attempts to remove as much as possible of the dissolved gas from the oil. 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 assurance using gas-in-oil standards takes account of incomplete extraction.

7.2.1 Toepler pump extraction apparatus (see Figure 9)

An example of a suitable design is shown in Figure 9 a). Note that Figure 9 b) 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 ⑧ which has previously been filled with oil [see Figure 9].

The Toepler pump extraction apparatus shall:

- 1) be capable of subjecting the liquid to a vacuum less than 10 Pa;
- 2) 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 2, 3 and 4 of 7.2.2. Omit step 5. Continue with steps 6, 7 and 8 as if oil were present. After compression of gas to atmospheric pressure in step 8, the amount of gas should be less than 0,1 ml.

- 3) permit the measurement of extracted gas to be made to the nearest 0,05 ml or better, at normal temperature and pressure.

In addition:

- 4) all tubing connecting the degassing flask ③ to the gas collection flask ② [in Figure 9 a)] shall be of large bore, at least 5 mm internal diameter and as short as practicable;
- 5) 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. A Pirani gauge is suitable.

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

Since the detailed design of this apparatus is not standardised, 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:

Use the extraction procedure as detailed in 7.2.2, but instead of degassing an unknown oil sample, substitute a gas-in-oil standard of the volume normally used containing all the gases listed in 8.1.

Degas successive standards until a number of cycles of the Toepler pump, together with a degassing time on each stroke, has been established such that the area or height of each peak on the chromatogram of the extracted gases is within 95 % of the area or height of that peak on the chromatogram of the same quantities of gases injected directly into the chromatograph by means of a standard gas mixture (see 8.6). A degassing time on each cycle of 1 min to 3 min is frequently used; the shorter the time the more degassing cycles are likely to be required.

The above procedure serves to establish a routine of operation, which will apply until any major changes are made to the equipment and will also apply to other equipments of the same design. It is recommended that the overall calibration of the complete equipment (degassing equipment plus chromatograph) is checked periodically (for example, every six months) using gas-in-oil standards to provide correction factors to be applied to areas or heights of chromatogram peaks.

7.2.2 Extraction procedure

The following is a typical extraction procedure used when a sample is in a syringe. It is described by reference to the apparatus shown in Figure 9 a) and Figure 9 b); the circled numbers refer to those figures. Modifications in the procedure may be needed for apparatuses of other designs.

- 1) Weigh the syringe ⑤ containing the oil sample and connect it to the degassing flask ③.

When a bottle is used as a container [Figure 9] the mass of oil degassed is determined by weighing the degassing flask before and after introducing the oil.