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COMMENTED VERSION

INTERNATIONAL STANDARD

colour inside

Method of sampling insulating liquids

IEC 60475:2022-05 CMV(en)

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COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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METHOD OF SAMPLING INSULATING LIQUIDS

FOREWORD

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
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This commented version (CMV) of the official standard IEC 60475:2022 edition 3.0 allows the user to identify the changes made to the previous IEC 60475:2011 edition 2.0. Futhermore, comments from IEC TC 10 experts are provided to explain the reasons of the most relevant changes, or to clarify any part of the content.

A vertical bar appears in the margin wherever a change has been made. Additions are in green text, deletions are in strikethrough red text. Experts' comments are identified by a blue-background number. Mouse over a number to display a pop-up note with the comment.

This publication contains the CMV and the official standard. The full list of comments is available at the end of the CMV.

IEC 60475 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications. It is an International Standard.

This third edition cancels and replaces the second edition published in 2011. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) addition of a new Annex C on sampling of oil from bushings, at the request of IEC subcommittee 36A, in order to transfer to IEC 60475 the corresponding contents of IEC TR 61464 relating to oil sampling from bushings;

b) deletion of NOTE 2 in 4.2.1.2.

The text of this International Standard is based on the following documents:

Draft	Report on voting
10/1163/FDIS	10/1173/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

General caution, health, safety and environmental protection

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

The insulating oils which are the subject of this document should be handled with due regard to personal hygiene. Direct contact with the eyes ~~may~~ can 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 document involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

~~**Environment**~~

This document is applicable to mineral oils and non-mineral oils, chemicals and used sample containers.

Attention is drawn to the fact that some mineral oils in service ~~may~~ can still be contaminated to some degree by PCBs. If this is the case, safety countermeasures should be taken to avoid risks to workers, the public and the environment during the life of the equipment, by strictly controlling spills and emissions. The disposal or decontamination of these oils ~~should be carried out strictly according to local regulations~~ can be subject to regulatory requirements with regard to their impact on the environment. Every precaution should be taken to prevent release of mineral oil and non-mineral oil into the environment.

METHOD OF SAMPLING INSULATING LIQUIDS

1 Scope

This document is applicable to the sampling procedure ~~to be~~ used for insulating liquids in delivery containers and in electrical equipment such as power and instrument transformers, reactors, bushings, oil-filled cables, oil-filled tank-type capacitors, switchgear and load tap changers (LTCs).

This document applies to liquids the viscosity of which at the sampling temperature is less than 1 500 mm^2/s (or cSt). It applies to mineral oils and non-mineral oils (such as synthetic esters, natural esters, vegetable oils or silicones).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 60567:2011, *Oil-filled electrical equipment – Sampling of gases and analysis of free and dissolved gases – Guidance*

IEC 60970, *Insulating liquids – Methods for counting and sizing particles*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
delivery container
container used to store, transport and deliver batches of oil

EXAMPLE Drum, rail tanker, road tanker, flexible plastic bag.

3.2
electrical equipment
equipment filled with insulating oil

EXAMPLE Power and instrument transformers, reactors, bushings, oil-filled cables, oil-filled tank-type capacitors, switchgear and load tap changers (LTCs).

3.3
sampling equipment
equipment used for sampling oil from delivery containers (e.g. sampling probes, such as dippers or siphons) and from electrical equipment (e.g. connecting tubing and drain valve adapters)

Note 1 to entry: Sampling equipment also includes sample containers, waste oil containers and other accessories.

3.4
sample container
container or other device used to store and transport samples of oil for analysis

EXAMPLE Syringe, bottle, ampoule.

Note 1 to entry: Sample containers include accessories such as valves, tubing or caps attached to the container.

4 General principles for the sampling of insulating liquids

4.1 New insulating liquids in delivery containers

4.1.1 Place of sampling

The sample shall be taken from the part of the delivery container where the insulating liquid is likely to be most heavily contaminated. To evaluate the quality of a consignment, two types of samples may be normally taken:

a) composite sample: mixture of samples taken at the same level in several containers;

b) individual sample: sample or mixture of samples taken at the same level in one container.

From a delivery, individual samples of 1 l may be taken from different containers for the electric strength test. Further tests may be carried out on these samples and a complete examination on the mixture of these (composite sample).

In certain cases, it ~~may~~ can be useful to constitute an average sample within the container. An average sample is a mixture of samples taken at different levels in one container.

1) tankers: samples should be taken from each tanker as described in 4.1.4.2 below;

2) drums: samples should be taken as described in 4.1.4.3 below.

In the case of a single drum, this shall be sampled.

In case there is more than one drum of a lot of oil, sampling procedures should be negotiated between supplier and user. For example, samples can be taken from 10 % of drums or at least two drums, whichever the largest.

4.1.2 Quantity of sample to be taken

This depends on the tests to be performed and the procedures used.

Typically, 2 l are taken.

4.1.3 Sampling equipment

4.1.3.1 General

Since the results of the tests included in IEC requirements for insulating liquids can greatly depend on the impurities in the sample, it is essential to observe the following precautions:

– separate sampling equipment shall be reserved exclusively for each type of liquid. All seals and tubing used should be compatible with the insulating liquid to be sampled;

– the equipment shall be clean and dry, following the cleaning procedures described in 4.2.1.6. Particular care should be taken to ensure the absence of any traces of solid impurities, such as dust, fibres~~, etc~~. The use of rags for cleaning is not permitted.

4.1.3.2 Sampling probes

As examples, four types of sampling probes are described below. Other probes may also be used, provided they do not introduce any contamination. Stainless steel and aluminium are suitable.

a) Sampling from tankers

The thief dipper shown in Figure 1 is suitable for taking samples at the bottom of the container. This is a dipper constructed of stainless steel or aluminium tubes and castings, machine-finished all over. It shall be sufficiently heavy to sink in the liquid. It should always be suspended by means of a metal wire or chain. String or other fibrous materials shall not be used.

The cream dipper is used for taking top samples of insulating liquids. This probe shall be constructed as shown in Figure 2 and shall be of stainless steel.

Dimensions in millimetres

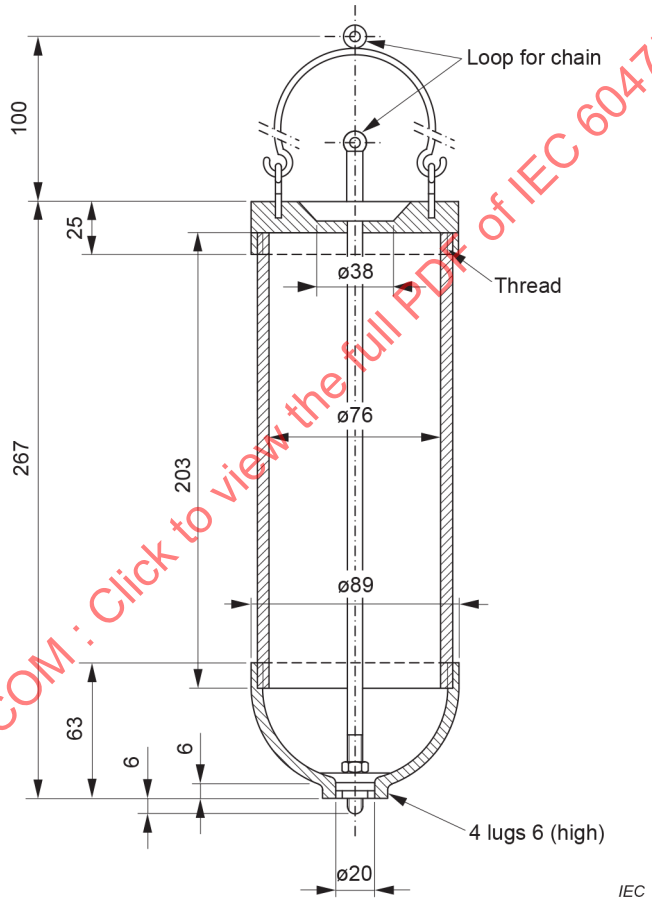


Figure 1 – Thief dipper

Dimensions in millimetres

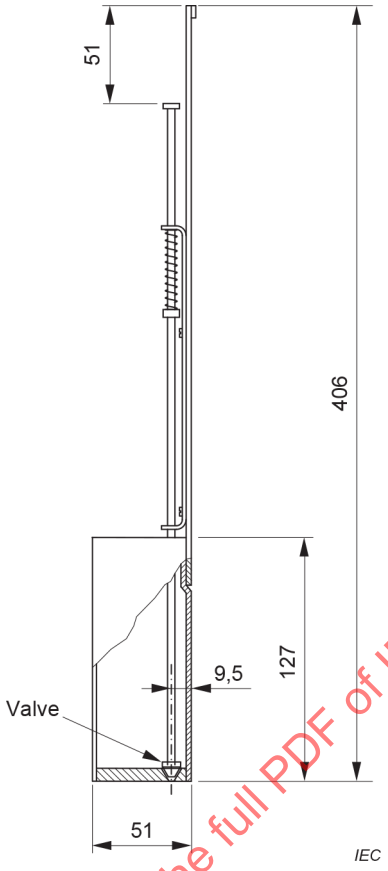


Figure 2 – Cream dipper

b) Sampling from drums

The pipette shown in Figure 3 enables samples to be taken at the bottom of drums. This pipette has a capacity of about 500 ml.

Another probe to take samples at the bottom is shown in Figure 4; it is a siphon with a glass, stainless steel or aluminium tube having an internal diameter of about 13 mm for taking off the sample liquid, and a metal tube (internal diameter 5 mm) for applying pressure. Both tubes are set in an oil-resistant bung whose dimensions correspond to the diameter of the bung hole in the drum. Commercial versions of this equipment are available. When possible, glass is preferable for probes illustrated in Figure 3 and Figure 4.

The cream dipper (Figure 2) may be used for taking top samples.

Dimensions in millimetres

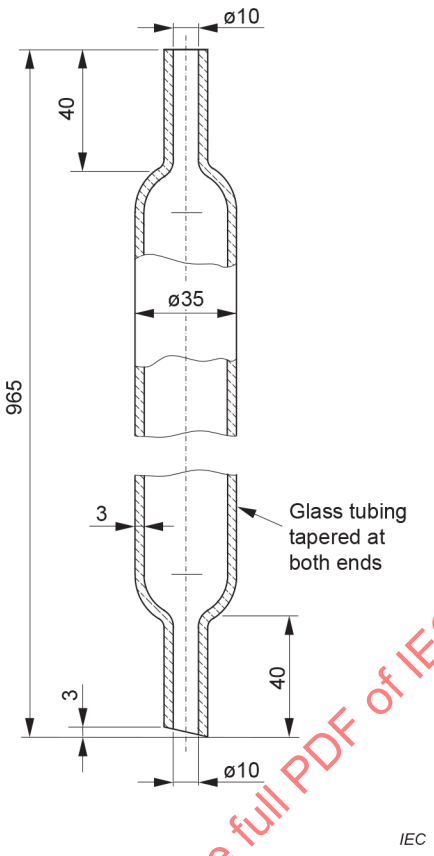


Figure 3 – Pipette

Dimensions in millimetres

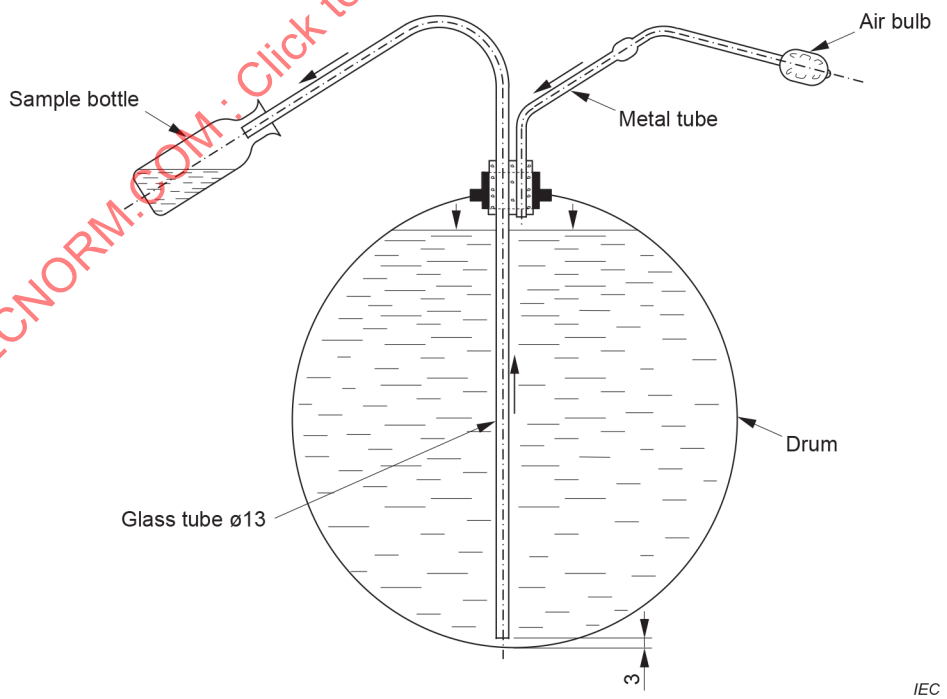


Figure 4 – Siphon

4.1.3.3 Sample containers

For storing and transporting samples, depending on the oil test to be performed, sample containers of appropriate volume shall be used. Different types of sample containers are indicated in 4.2.1.5.

For the mixing of different samples, a special sample container made of glass with a capacity of at least 6 l shall be used. These special sample containers shall be closed in a manner that allows them to be sealed, by means of oil-resistant plastic or compatible rubber tubing or screw caps equipped with a polytetrafluoroethylene (PTFE) lining. Natural rubber tubing and/or seals are not permitted. PTFE and polypropylene (PP) seals are acceptable.

Each sample container shall have a label on which are marked all the indications necessary to identify the contents, i.e. the markings of the drums or tanks, date of sampling and the name of the recipient.

4.1.3.4 Cleaning of sampling equipment

Sampling equipment shall be cleaned following the procedures described in 4.2.1.6.

4.1.4 Sampling procedure

4.1.4.1 General remarks

According to general principles for sampling (see 4.1.1), samples of new insulating liquid shall be taken from the bottom of the delivery container, where the contamination is likely to be the greatest. But in certain cases, an average sample is also of interest.

NOTE 1 To obtain an average sample, samples are taken at intermediate levels in tanks or drums. Examples of procedures are given in Annex A. A procedure is indicated in the NOTE in 4.1.4.2 a) for obtaining the equivalent of an average sample.

In Table 1 different cases are considered:

Table 1 – Types of samples of new insulating liquids

Type of delivery	Recommended sampling	Equipment to be used	Procedure	Recommended quantity
Drums	Composite	Pipette (Figure 3) or Siphon (Figure 4)	4.1.4.2	3 × 2 l
	Individual		4.1.4.2	1 l or 2 l
Tankers	Individual	Thief dipper (Figure 1) or None (valve)	4.1.4.1 b) 4.1.4.1 a)	3 × 2 l
Drums	Average	Pipette or Siphon (Figure 3 and Figure 4)	Annex A	3 × 2 l
Tankers	Average	Thief dipper (Figure 1) or None (valve)	4.1.4.1 a)	3 × 2 l

NOTE 2 Before sampling from tankers, sufficient oil ~~should be~~ is pumped from the end of the delivery pipe, as ~~required by~~ indicated in 4.1.4.2.

Every precaution shall be taken during sampling in order to avoid contamination of the insulating liquids. Outdoor sampling of insulating liquids in rain, fog or high wind is only permitted if all precautions have been taken to avoid contamination of the liquid. In this special case, the use of a cover is necessary.

Condensation shall be avoided by warming the sampling equipment so as to be above the ambient air temperature. Before use, the equipment shall be rinsed with the liquid being sampled. The operator shall be warned not to ~~permit his~~ let their hands ~~to~~ come into contact with the surfaces of sampling equipment subsequently in contact with the oil. The insulating liquids shall be protected against light irradiation during transportation and storage.

On arrival at the laboratory, the sampling container shall not be opened immediately. It is necessary to wait until the temperature of the sample is the same as the room temperature.

4.1.4.2 Sampling from tankers

Insulating liquids may be sampled either through the tank outlet or by a thief dipper or by a cream dipper.

a) Sampling through the tank outlet

By this procedure, it is possible to obtain a sample representative of the bottom of the tank after the tank has been allowed to stand for at least 1 h after the vehicle has arrived.

NOTE It may be possible, by this procedure, to obtain the equivalent of an average sample, if the sampling is done directly after the vehicle has arrived.

In this case, the sampling procedure shall be as follows:

– remove the outlet valve shield, if fitted;
– remove all visible dirt and dust from the valve by means of lint-free clean cloths or oil-resistant synthetic sponges;
– the outlet system (pump, delivery pipe), if incorporated, shall be started or opened as appropriate in order to get a sample;
– open the valve and allow to flow, slowly, at least 10 l of insulating liquid into a waste oil container. In any case, discard at least an amount of oil equivalent to the volume of the pipe;
– rinse sampling bottles with the insulating liquid;
– fill sampling bottles at constant flow to avoid turbulence.

b) Sampling with a thief dipper or a cream dipper

This sampling should be carried out after the tank has been allowed to stand for at least 1 h after the vehicle has arrived.

1) Procedure with the thief dipper (Figure 1) (bottom samples)

For taking bottom samples (i.e. within 1 cm to 2 cm from the bottom of the tank) the dipper is lowered until the projecting stem of the valve rod strikes the bottom of the tank. The dipper then fills. Filling is complete when no more air bubbles escape. The dipper is then withdrawn and its contents poured into the sample container (in the case of an individual sample) or into the special glass sample container for collecting and mixing the various samples taken (in the case of a composite sample). In this latter case, the sample container(s) is (are) filled with the mixture so obtained. During pouring of the liquid, avoid forming air bubbles by pouring too quickly.

2) Procedure with the cream dipper (Figure 2) (top samples)

With the valve closed, fill the cream dipper by slowly immersing it in the liquid to be sampled until the rim is just below the surface of the liquid so that it will flow slowly into the dipper. Discard the first filling. Refill the dipper as above and transfer the sample to the sample container by allowing it to flow from the bottom orifice against the side of the sample container and not in a stream into the bottom of the sample container. Repeat the operation until sufficient liquid is obtained to fill the sample container (individual sample) or the special glass sample container used for mixing samples depending on the type of sample to be obtained.

4.1.4.3 Sampling from drums

Samples should be taken after the drums have been allowed to stand for at least 8 h with the bung uppermost, protected against rain and rainwater. For sampling the bottom (i.e. 3 mm up), the pipette (Figure 3) or the siphon pressure thief (Figure 4) may be used.

For taking a sample from the surface layer of the liquid, the cream dipper (Figure 2) may be used.

Examples of procedures:

a) Use of pipette (Figure 3) (bottom samples)
   - block the upper orifice of the pipette with the thumb, and then immerse the pipette in the liquid to the bottom of the drum;
   - remove the thumb to allow liquid to enter the pipette;
   - again close the upper end of the pipette with the thumb and withdraw the pipette;
   - the first filling is used for rinsing the pipette; transfer the next fillings into either a sample container (individual sample) or the special glass sample container for mixing samples (composite sample) (see 4.1.4.2 b)) taking care not to form air bubbles during pouring the liquid.

b) Use of siphon (Figure 4) (bottom samples)
   - fit the bung in which are set the riser and pressure tubes into the bung hole of the drum and ensure that this seal is airtight;
   - dip the lower end of the riser tube to about 3 mm from the bottom of the drum;
   - raise the pressure inside the drum by means of the air bulb;
   - run off enough liquid to rinse the tube and then run off the required quantity directly into the sample container (individual sample) or the special glass sample container for mixing samples (composite sample) (see 4.1.4.2 b)) taking care not to form air bubbles during pouring of the liquid.

c) Use of cream dipper device (Figure 2) (top samples)

See 4.1.4.2 b).

4.1.4.4 Sampling report

The sampling report shall give all the information necessary for identifying the sample as well as any details or special information likely to be of help to those entrusted with the tests. The type of sample (i.e. composite, individual or average sample) shall be specified. A copy of the report shall accompany each sample. The distribution of samples shall be in accordance with the agreed procedure, for example as given in the sales contract.

4.2 Sampling of oil from oil-filled equipment

4.2.1 General remarks

4.2.1.1 Safety and quality of sampling

The manufacturer's instructions for taking oil samples from the electrical equipment shall be followed. Particular attention shall be paid to the safety precautions to be taken.

Ensure that the oil in the energized electrical equipment is not under a negative pressure when taking an oil sample, since this could introduce air bubbles in the oil, induce electrical short-circuits in the equipment and put the sampling personnel at risk.

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

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

In all cases, oil sampling should be performed by properly and specifically trained personnel, especially for low volume equipment (e.g. instrument transformers).

4.2.1.2 Place of sampling

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 (for example, from the bottom oil drain valve or the oil sampling valve). 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, such as from the tap changer, selector switch or gas relay).

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.

~~NOTE 1~~ 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.

~~NOTE 2 When sampling from bushings or from instrument transformers or cables, the manufacturer's instructions should be followed carefully. Failure to do so may lead to serious damage and equipment failure. The oil sampling should be carried out on de-energized equipment. 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 equipment condition.~~

~~Sampling by syringe is the procedure recommended for bushings by IEC subcommittee 36A. 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.~~

4.2.1.3 Cleaning of sampling point

Cleaning of the sampling equipment and flushing of the sampling point shall be done to prevent contamination of oil samples.

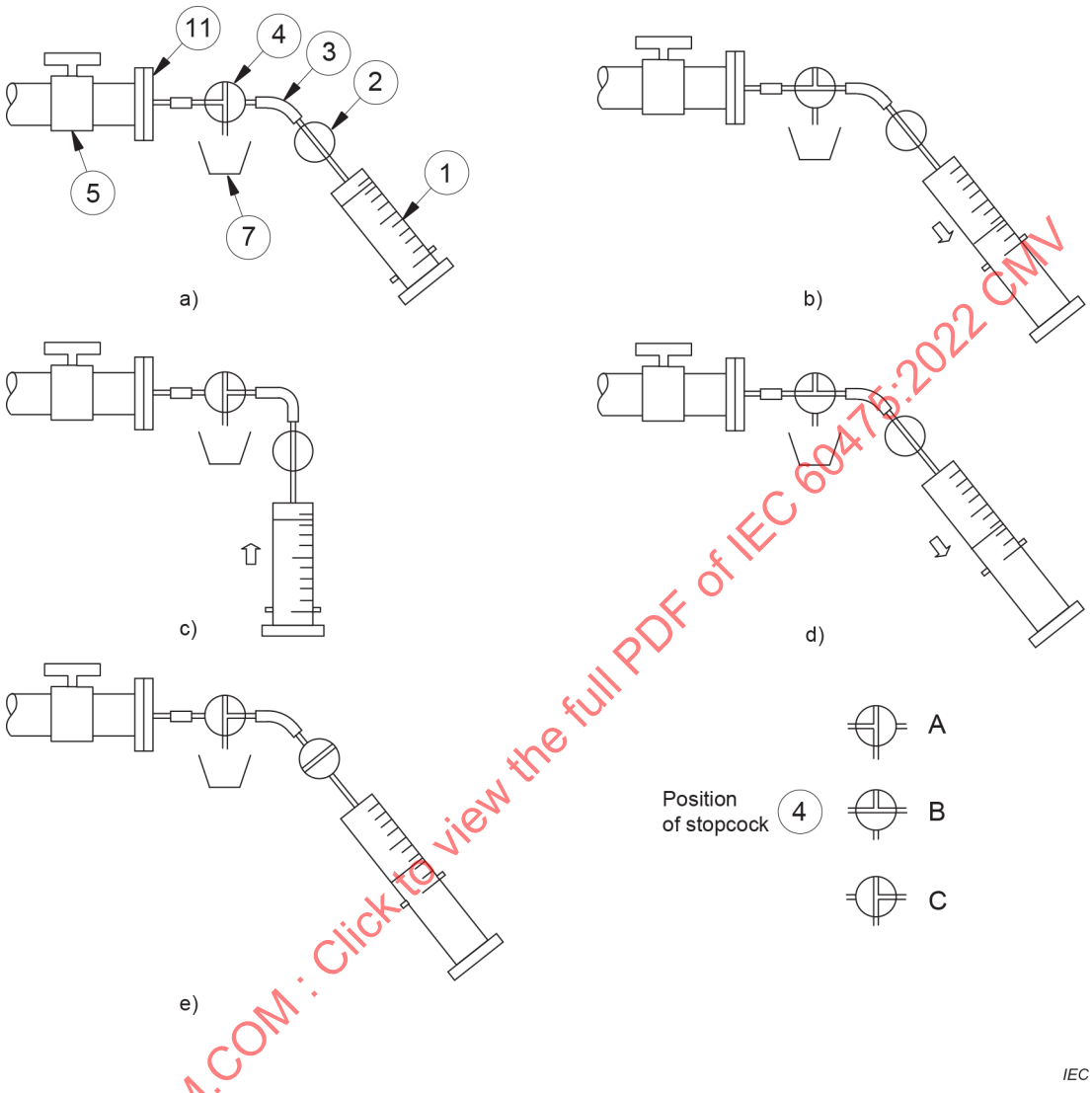
The blank flange or cover (11) of the sampling valve in Figure 5, Figure 6 and Figure 7 a) is removed and the outlet cleaned with a lint-free cloth or oil-resistant synthetic sponge to remove all visible dirt.

The drain valve is flushed with a sufficient quantity of oil (typically, 2 l to 5 l), under a turbulent flow, to eliminate any contaminants (water and particles) that might have accumulated in the drain valve and at its orifice.

Use protection gloves, preferably made of nitrile rubber, and a bucket for waste oil. The sampling point shall be cleaned each time a new sample of oil is taken.

For measuring water content in oil, sampling shall be carried out ~~preferably~~ during days when the humidity of the air is as low as possible, to prevent moisture condensation on sampling equipment and contamination of the oil sample.

The temperature of oil at sampling point shall be measured with a thermometer placed in the flow of oil and indicated on the sample, to ensure the calculation of the relative humidity of oil, also recording whether or not the fans and pumps are running. In both cases, the method used to measure the temperature shall be indicated.

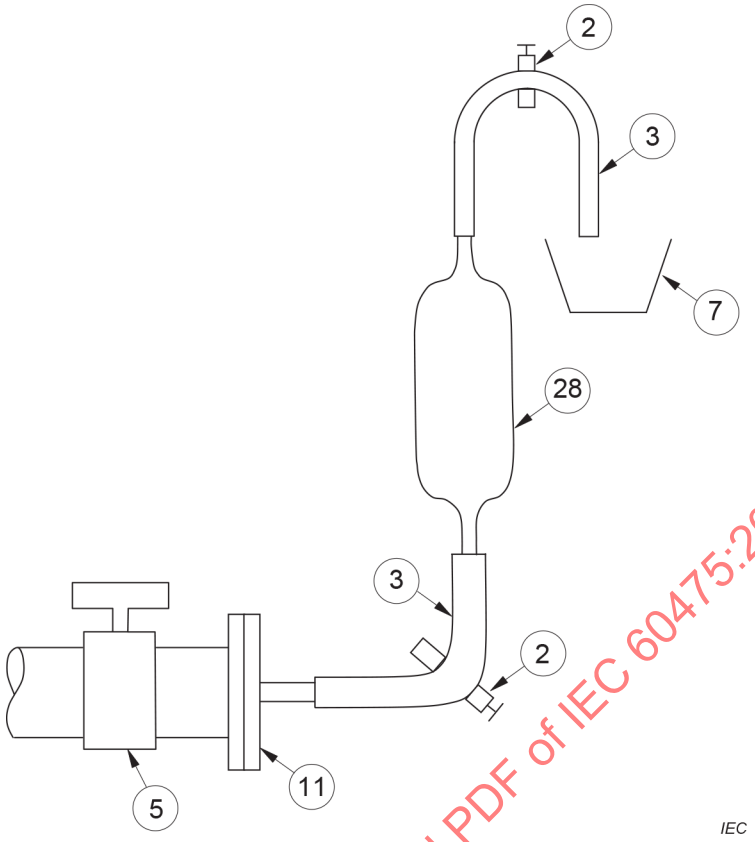


Key

a flushing position
b wetting and flushing of syringe
c emptying of syringe
d taking of sample
e disconnecting of syringe

1 syringe
2 stopcock
3 flexible connecting tubing
4 three-way valve
5 equipment sampling valve
7 waste vessel
11 blank flange

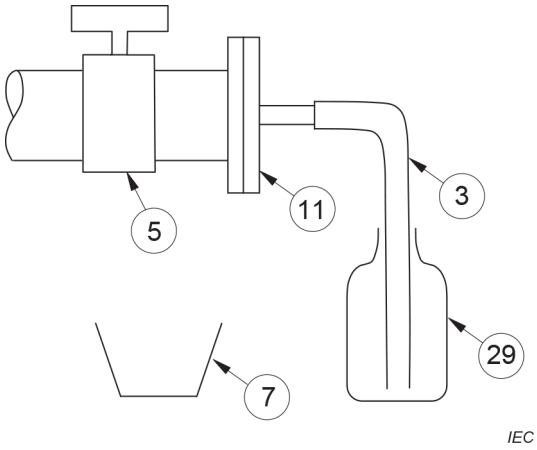
Figure 5 – Sampling of oil by syringe



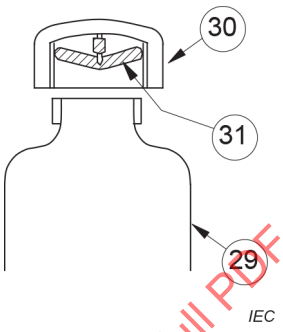
Key

2	stopcock	7	waste vessel
3	flexible connecting tubing	11	blank flange
5	equipment sampling valve	28	sampling tube

Figure 6 – Sampling of oil by ampoule



a) Example of sampling by bottle



b) Example of seal cap for bottle

Key

3	flexible connecting tubing	29	bottle
5	equipment sampling valve	30	hard plastic screw cap
7	waste vessel	31	conical soft polyethylene seal
11	blank flange		

Figure 7 – Sampling of oil by bottle

4.2.1.4 Connection between sampling point and sampling device

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

Attach a piece of oil-compatible plastic or rubber tubing to connect the sampling point to the sampling device. This tubing should be as short as possible. To avoid contamination by the previous oil sample, use a new piece of tubing, or flush the tube well and wash its outer surface with the next oil to be sampled.

Suitable tubing should be made for example of perfluorinated material (e.g. Viton®[1], Tygon®), PTFE or metal, not PVC.

[1] Viton® and Tygon® are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of these products.

4.2.1.5 Choice of sample container

Table 2 indicates the different types of sample containers that can be used depending on the oil analysis to be made.

Table 2 – Sample containers for oil tests

Sample container	Syringe	Flexible bottle	Bottle	Flexible bottle	Ampoule	Ampoule	Oil volume
Material	Glass	Metal	Glass	Plastic	Glass	Metal	ml
Oil test							
Dissolved gases	Y	Y	Y		Y	Y	25 to 100
Water	Y	Y	Y				20
Dielectric dissipation factor	Y	Y	Y	Y			200
Particles	Y	Y	Y	Y			100
Breakdown voltage		Y	Y				500 to 1 000
Other chemical and physical tests		Y	Y	Y			250
All tests							1 000 to 2 000
Volume (ml)	25 to 250	125 to 2 500			125	25 to 250	
Key Y = Yes							

Metal or plastic containers may be preferred where adequate protection of glass containers is not available for the transportation of oil samples.

For dissolved gas analysis (DGA), to minimize losses of the low solubility gases (H_2 and CO) and pick-up of air at low total gas contents, it is critical to strictly follow the sampling procedures of 4.2.2 to 4.2.5, particularly with bottles and ampoules. In addition, only the materials recommended for caps, gaskets, valves and tubing of sample containers should be used by well-trained and experienced personnel familiar with those containers.

When using bottles for DGA, water and breakdown voltage, care should be taken to minimize air contact with the oil sample during sampling and analysis.

The use of plastic bottles is not permitted for DGA, water and dielectric strength, since ambient air contamination and gas losses ~~may~~ can occur by diffusion through the plastic. For the other tests, plastic bottles should be made of a compatible plastic (such as high-density polyethylene (HDPE), polypropylene or polycarbonate), which does not contaminate the oil with additives contained in the plastic. Each new type of plastic (and glass) bottle should be tested for compatibility with oil.

The recommendations of 4.2.1.3 and 4.2.1.6 (cleaning of sampling point and sampling equipment) should be followed strictly for water content, dielectric strength, dielectric dissipation factor, interfacial tension and particles content.

The other physical and chemical tests (viscosity, density, acidity, DBPC, furans and PCB contents, etc.) are less affected by the sample containers and sampling procedures used.

4.2.1.6 Cleaning of sampling equipment

4.2.1.6.1 Use of disposable sample containers

Use of disposable, pre-cleaned metal, plastic and glass bottles having a known level of cleanliness for dust and humidity has been found by several users more convenient than cleaning them. Such sampling devices are relatively inexpensive and available from several vendors of labware or veterinarian equipment. To verify that the cleanliness of disposable bottles is acceptable, a few un-cleaned and cleaned bottles can be tested in parallel.

4.2.1.6.2 Cleaning procedures

Non-disposable sampling devices may be cleaned in a dishwasher using a detergent, and rinsed with tap water (without detergent in the rinse aid compartment of the dishwasher). A last, optional rinse with de-ionized water may be used.

Sampling equipment and containers may also be cleaned with normal heptane.

After cleaning, the sampling devices are dried in an oven at typically 100 °C until fully dried, then allowed to cool in the oven or a dry box.

After drying, they shall be immediately closed to protect from contamination and not opened until just before use.

Appropriate cleaning of sample containers is critical for DDF and interfacial tension, which are particularly sensitive to contamination. They should not be cleaned with solvents.

Dedicated sampling containers shall be cleaned according to IEC 60970 ~~are recommended~~ for measuring particles content in oil.

4.2.2 Sampling of oil by syringe

4.2.2.1 Sampling equipment

The following sample equipment shall be used:

a) Graduated gas-tight syringes of a size suitable for containing adequate oil sample volume (20 ml to 250 ml), and equipped preferably with a three-way plastic valve made of nylon body and polypropylene (PP) barrel, or with a three-way stainless steel valve. The use of syringes with matched piston and barrel is preferred when sampling for DGA in order to allow the piston to flow freely with oil volume variations, and to avoid pressure and vacuum build-up in the syringe and breakage during handling. Plastic syringes should not be used.

   For plastic three-way valves, a new valve should be used each time an oil sample is taken and not recycled, because it ~~may~~ can be contaminated with the previous oil sample and lose its gas tightness when used several times. For added protection during transportation, a stainless steel Luer-Lock cap may be placed on top of the three-way valve. This cap may be recycled after use.

   NOTE Priming the piston with clean, degassed oil has been found useful to avoid the formation of bubbles along the piston when introducing the oil sample for DGA analysis. The use of a low viscosity water-soluble lubricant has also been found useful for DGA.

   The size of sample required depends on the likely concentration of gas in the sample, the analytical techniques and the sensitivity required. For DGA after factory tests, a 250 ml syringe has been found convenient.

   See Annex B.

b) Transport containers, designed to hold the syringe firmly in place during transport but which allow the syringe plunger freedom to move and prevent its tip from contacting the container whatever its position during transportation. Cardboard boxes with removable inner cardboard flaps that hold the barrel in place have been found convenient for that purpose. Metal or plastic cylinders with inside foam packing have also been found appropriate for transportation. When sampling for DGA, the syringe should preferably be transported in the vertical position, piston upwards, to avoid the formation of bubbles in oil.

4.2.2.2 Sampling procedure

See Figure 5.

a) The electrical equipment is connected as shown in Figure 5 a), and its sampling valve (5) opened.

b) The three-way valve (4) is adjusted (position A) to allow 1 l to 2 l of oil to flow to waste (7).

c) The three-way valve (4) is then turned (position B) to allow oil to enter the syringe slowly (Figure 5 b)). The plunger should not be withdrawn but allowed to move back under the pressure of the oil.

d) The three-way valve (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 5 c). Confirm that the inner surfaces of the syringe and plunger are completely oiled.

e) The procedure described in steps c) and d) is then repeated until no gas bubble is present. Then the three-way valve (4) is turned to position B and the syringe filled with oil (Figure 5 d).

f) The three-way valve (~~2~~4) on the syringe and the sampling valve (5) are then closed.

g) The three-way valve (4) is turned to position C and the syringe disconnected (Figure 5).

h) When sampling for DGA, if the oil taken from the electrical equipment is hot, place the syringe in its protective box in the vertical position, standing on the piston and with the syringe tip upwards, until the oil has slowly cooled down, then install the syringe back into the holding flaps of the protective box for transportation. This will prevent the formation of bubbles in oil.

Label ~~carefully~~ the sample ~~(see 4.4)~~ in accordance with 4.4.

NOTE 1 It is good practice to avoid contamination of the outer surface of the plunger and inner surfaces of the syringe by dust or sand. Such particles can affect the sealing properties of the syringe. This kind of contamination can come from wind-swept dust or from the handling of the syringe.

NOTE 2 In the case of sealed transformers, if a bubble appears in the syringe directly after sampling, ~~it is recommended to resample~~ the sampling procedure is repeated.

4.2.3 Sampling of oil by ampoule

4.2.3.1 Sampling equipment

The following sample equipment shall be used:

a) Glass or metal ampoule, typically of volume 125 ml to 1 l. It may be closed either by stopcocks or pinchcocks on oil-compatible plastic tubing or by valves. Glass ampoules are usually made of Pyrex®[2] glass. Metal ampoules are made of stainless steel and ~~may~~ can use spring-loaded valves instead of plastic tubing as expansion devices.

   The oil-compatible plastic tubing used for ampoules should be used only once, not recycled, since it has a memory effect and ~~may~~ can contaminate the oil sample when sampling for DGA. The types of compatible plastic tubing are indicated in 4.2.1.4.

[2] Pyrex® is the trademark of a product supplied by Corelle Brands. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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 tests to be carried out and, for DGA, the likely concentration of gas in the sample, the analytical technique and the sensitivity required. For DGA after factory tests, a 250 ml ampoule has been found convenient.

b) Transport containers, designed to hold the sampling tubes firmly in place during transport.

4.2.3.2 Sampling procedure

See Figure 6.

a) The device is connected as shown in Figure 6.

b) The cocks (2) on the plastic tubing of the sampling ampoule (28) and the equipment sampling valve (5) are carefully opened so that oil flows through the sampling ampoule to waste (7). When sampling for DGA, oil should flow under a non-turbulent flow (until there are no air bubbles in the oil), to avoid the formation of bubbles in oil and the stripping of dissolved gases out of oil.

c) After the sampling ampoule (28) has been completely filled with oil, about 1 l to 2 l are allowed to flow to waste (7).

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~~ ampoule (28) is then disconnected and the sample ~~carefully~~ labelled ~~(see 4.4)~~ in accordance with 4.4.

NOTE If a glass sampling ampoule 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 ampoule in the event of it being exposed to a rise in ambient temperature. ~~Record on the label that this has been done.~~ Once completed, this procedure is also recorded on the label.

4.2.4 Sampling of oil by flexible metal bottles

4.2.4.1 Sampling equipment

The following sample equipment shall be used:

a) Flexible metal bottles capable of being sealed gas-tight, typically of volume 250 ml to 2,5 l.

Metal bottles should not be soldered, as materials used for soldering ~~may~~ can contaminate the oil. Adsorption on aluminium surfaces of water contained in oil is possible. Metal bottles made of drawn aluminium or of welded tin are flexible and do not need oil expansion devices. They should be filled completely with oil by pressing on the bottle sides before closing the bottle.

Metal bottles should be closed with a screw cap lined with a non-porous, leak-free gasket compatible with oil. Gaskets should be used only once, not recycled, except if they are lined with aluminium foil on the oil side.

For DGA and water analysis, the porosity of gaskets used should be measured by taking at least six samples of oil from a transformer into identical bottles. The hydrogen content of the oil used for testing of the sampling equipment should be at least 100 µl/l. Analyse samples for hydrogen content at intervals over a month, the first being as soon as possible after taking the samples. A bottle and seal design is acceptable if it permits losses of hydrogen of less than 2,5 % per week. Suitable bottles have, for example, screwed plastic caps holding a conical polyethylene (PE) seal or flexible gasket (see Figure 7).

For analytical tests other than DGA and water, the above requirement for gas tightness does not apply.

For mineral oils, gaskets should be made of polyethylene (PE), PTFE or nitrile-butadiene rubber (NBR) (containing more than 30 % of nitrile component).

For non mineral oils (e.g. natural and synthetic esters), gaskets should be made of PTFE (not NBR or silicone rubber).

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

4.2.4.2 Sampling procedure

See Figure 7 a).

a) The sampling valve (5) is carefully opened and about 1 l to 2 l of oil allowed to flow under a laminar flow to waste (7) through the tubing (3) ensuring that all gas bubbles are eliminated before the oil sample is collected and gases are not stripped out of the oil by the oil flow.

b) 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 from bottom up. Rinse the bottle with one-third of oil then send the oil to waste.

   When sampling for DGA, introduce the oil under a continuous, non-turbulent flow, until no gas bubbles are observed in oil when it flows out of the bottle in order to avoid the formation of bubbles in oil and the stripping of dissolved gases out of oil (otherwise significant gas loss ~~may~~ can occur). Filling the bottle should be slow enough to allow laminar flow of oil and as fast as possible to avoid gas loss to (and contamination from) the atmosphere. If the time to fill the bottle exceeds a few minutes, a new sample should be taken.

   When sampling for water, strictly follow the recommendations of 4.2.1.3.

c) Allow about two bottle volumes to overflow to waste (7), then withdraw the tubing (3) slowly with the oil still flowing. Gently squeeze the sides of the bottle so it is entirely filled with oil, then securely close with the cap.

d) Close the sampling valve (5) and disconnect the tubing. Label the sample (see 4.4).

   Tighten the cap again after the oil has cooled to ambient temperature.

4.2.5 Sampling of oil by glass and rigid metal bottles

4.2.5.1 Sampling equipment

The following sample equipment shall be used:

a) Glass or rigid metal bottles capable of being sealed gas-tight, typically of volume 125 ml to 2,5 l. Clear glass bottles shall be protected from sunlight, so the use of dark bottles is highly recommended. Even so, for samples for DGA, extra protection from light should be provided during transport and storage.

   Caps and gaskets described for flexible metal bottles in 4.2.4.1 are suitable for glass and rigid metal bottles.

b) Transport containers, designed to protect the bottle during transportation.

4.2.5.2 Sampling procedure

See Figure 7 a).

Sampling procedures are the same as for flexible metal bottles in 4.2.4.2, except that glass and rigid metal bottles should not be filled entirely with oil.

Instead, allow the oil level to fall a few centimetres from the rim so as to leave a small expansion volume of air (typically, 3,5 ml to 7 ml, or 1,5 cm to 3 cm of airspace), to allow for oil expansion with increasing temperatures. Fill with no less than 90 % of oil to allow for air expansion when temperatures decrease and avoid implosion of the glass bottle. Place the bottle cap securely in position and label the sample (see 4.4). Indicate the approximate expansion volume of air on the label. Correction for gas loss to the small headspace volume of air in the bottle ~~will~~ shall be calculated by the laboratory as indicated in Annex D of IEC 60567:2011.

Where transport and storage conditions are not particularly demanding, some companies prefer to have the bottles filled completely and closed lightly, finger-tight, with a screwed plastic cap having a conical polyethylene seal. In the event of expansion of the oil by heat, these caps act as a non-return valve, allowing a small amount of oil to escape. Where contraction by cooling occurs, the seal will prevent ingress of air. In the latter case, it is necessary for the bottle ~~will need~~ to be warmed up to the sampling temperature to re-dissolve the gases prior to analysing for dissolved gases.

For other analytical tests, an airspace can be left above the oil.

4.2.6 Sampling of oil by plastic bottles

4.2.6.1 Sampling equipment

The following sample equipment shall be used:

Plastic bottles should be made of a compatible plastic (see 4.2.1.5), which does not contaminate the oil with additives contained in the plastic. Each new type of plastic bottle should be tested for compatibility with oil. Use of virgin plastic without fillers or pigments is strongly recommended.

Plastic bottles should not be used for DGA, water content and dielectric breakdown.

Caps and gaskets described for metal bottles in 4.2.4.1 are suitable for plastic bottles.

Moulded all-plastic caps, of suitable composition as above, are suitable.

4.2.6.2 Sampling procedure

See Figure 7 a).

Sampling procedures are the same as for flexible metal bottles in 4.2.4.2.

4.3 Storage and transportation of samples

Some of the dissolved oxygen present in the oil sample ~~may~~ can be consumed, and hydrocarbons and carbon oxides formed by oxidation. This reaction is accelerated by exposure to light, therefore sampling devices made of transparent materials (syringes, glass bottles and ampoules) should be protected (for example, by wrapping them in an opaque material or placing them in a box for transportation).

In any case, the analysis should be carried out as soon as possible after sampling to avoid oxidation reactions and gas losses or pick-ups from the sampling devices.

Oil syringes (and other oil sampling devices) may be placed in sealed boxes to fully eliminate the risk of formation of bubbles in important DGA oil samples during transportation in planes, due to reduced pressure and over-saturation of gases in the oil. The syringe plunger should be allowed to move in order to prevent air ingress in case of oil volume variations.

4.4 Labelling of samples

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

The following information, as shown in Table 3, is necessary (whenever it is known).

Table 3 – Information required on oil sample labels

Transformer or other equipment	Sampling
Customer	Sampling date
Location	Sampling point
Identification number	Sampling person
Manufacturer	Reason for analysis (routine or other)
General type (transformer (generation or transmission, instrument, industrial), reactor, cable, switchgear, etc.)	Transformer non-energized, off-load energized or on-load
Rated MVA	Oil temperature when sampling
Voltage ratio	Humidity: dry – wet – fog – indoors
Type and location of OLTC	
Date of commissioning	
Oil	
Type of oil (mineral or non-mineral)	Weight (or volume) of oil
Product name	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 percentage 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;

– for water in oil analysis, temperature of oil, the method used for measuring the temperature, and whether or not the fans and pumps are running (to be able to calculate the relative humidity of oil);

– time of sampling where more than one sample is taken.

Annex A
(informative)

Procedure for sampling at intermediate levels (making up of the average sample)

A.1 Use of the thief dipper

See Figure 1 and 4.1.4.2 b). The thief dipper is immersed to the required depth. The chain attached to the central rod is then pulled, care being taken that vertical displacement of the rod does not exceed 50 mm. The dipper then fills; filling is complete when no more air bubbles escape. The dipper is then withdrawn and its contents poured into the mixing container.

A.2 Use of the pipette

See Figure 3 and 4.1.4.3. The pipette is immersed to the required depth.

A.3 Use of the siphon

See Figure 4 and 4.1.4.3. The siphon is immersed to the required depth.

A.4 General remark

The samples taken at intermediate levels for making up the average sample are transferred in the mixing container for collecting the samples as soon as they are taken. The mixture is then used to fill sampling bottles.

Annex B
(informative)

Procedure for testing the integrity of the syringes

The procedure is as follows:

a) Connect the syringe to be tested to a three-way valve (4.2.2.1).
b) Move the valve to the open position (position B or C as shown in Figure 5).
c) Press the piston fully into the syringe.
d) Close the valve by adjusting in position A.
e) Try to pull the piston from the syringe and hold it under tension for about 30 s.
f) After releasing the piston, it should return in the original position.
g) If any amount of air was locked between the piston and the syringe body, then the syringe or the valve is not hermetically sealed, and it is recommended that ~~it is~~ the syringe or the valve be replaced by a new one.

Annex C
(informative)

Procedure for sampling oil from bushings 1

The general procedures for sampling oil from electrical equipment are described in 4.2.1.1, 4.3 and 4.4.

The specific procedures for sampling oil from bushings are the following:

The oil sampling should be carried out on de-energized bushings in their normal position. This is important in assessing bushing condition.

When sampling, precautions should be taken to deal with any sudden release of oil and prevent any moisture and dust entry in the bushing and the sample.

With small oil volume bushings it is essential to ensure that the total volume of oil drawn off does not endanger the operation of the bushing. See NOTE 2 below.

The method of sampling by syringe is the preferred method of oil sampling from bushings (see 4.2.2) and is suitable irrespective of the mode of transport of samples, but other sampling devices are also applicable if compliant with the manufacturer's instructions.

WARNING – The manufacturer's instructions should be consulted before any sampling. Failure to follow the instructions from the manufacturer can lead to serious damage and bushing failure. Normally the amount of oil in bushings is limited, therefore it is not usual to carry out routine sampling. Where sampling is carried out, the effect on the oil level and pressure should be taken into consideration. Oil sampling from oil impregnated paper (OIP) bushings without an oil level gauge is not recommended.

a) Bushings fitted with a sampling point at the mounting flange
   1) Remove the protective cover of the sampling point. Clean the outlet with a lint-free cloth to remove all visible dirt (see also 4.2.1.3).
   2) Ensure that all parts of the sampling component are clean and dry.
   3) Connect the tubing to the sample point.
   4) Flush the tube as described in 4.2.2.2 b) to e) and in Figure 5.
   5) Obtain the sample for analysis as described in 4.2.2.2 e) to h) and in Figure 5.

b) Bushing not fitted with a sampling point at the mounting flange

In this case, it may be possible to take a sample from the top of the bushing. The manufacturing instructions should be consulted to determine a suitable position. Insert one end of the sampling tube into the bushing, from the top, and connect to the other end to the three-way stopcock on the syringe, using a plastic connector. Follow the same procedure as above.

In the case of bushings pressurized at ambient temperature, the procedure described in a) and b) above is not applicable. Reference should be made to the instructions of the equipment manufacturer. Oil-SF6 or oil-oil bushings, in particular, in which internal oil is in a close volume, with a bellow for oil dilatation, shall always keep a positive oil pressure to prevent air and moisture ingress when sampling. On those bushings the sampling point is always at the mounting flange.

Oil replacement: The oil removed from the bushing shall be replaced if the volume of oil extracted is more than 1 % of the total oil volume in the bushing.

The removed oil is replaced with an appropriate volume of clean, dry and degassed oil compatible with the original oil. Consultation of the manufacturer is highly recommended.

In the case of large bushings, if the oil is not to be replaced immediately, the volume removed shall be noted for replacement during subsequent sampling or maintenance.

Labelling of samples should be made in accordance with Table 3, and include the following additional information on the bushing:

– volume of oil in bushing;
– time out of service;
– storage;
– rated kV;
– rated current;
– manufacturer;
– bushing type;
– phase.

Bibliography

IEC TR 61464, *Insulated bushings – Guide for the interpretation of dissolved gas analysis (DGA) in bushings where oil is the impregnating medium of the main insulation (generally paper)*

List of comments

1 Addition of a new Annex C on oil sampling from bushings, in close collaboration with IEC Subcommittee 36A on bushings.

Oil sampling from bushings requires much more attention than from power transformers, because of the small volume of oil in bushings and of possible ingress of air bubbles.

In new Annex C have been put together procedures for sampling oil from bushing previously dispersed in various parts of IEC 60475 and in IEC TR 61464 of IEC SC 36A (now withdrawn). This will allow to more efficiently and reliably sample oil from bushings, without affecting their integrity and DGA results.

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IEC 60475

Edition 3.0 2022-05

INTERNATIONAL STANDARD

NORME INTERNATIONALE

Method of sampling insulating liquids

Méthode d'échantillonnage des liquides isolants

IEC 60475:2022-05(en-fr)

CONTENTS

INTERNATIONAL ELECTROTECHNICAL COMMISSION

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METHOD OF SAMPLING INSULATING LIQUIDS

FOREWORD

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
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IEC 60475 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications. It is an International Standard.

This third edition cancels and replaces the second edition published in 2011. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) addition of a new Annex C on sampling of oil from bushings, at the request of IEC subcommittee 36A, in order to transfer to IEC 60475 the corresponding contents of IEC TR 61464 relating to oil sampling from bushings;
b) deletion of NOTE 2 in 4.2.1.2.

The text of this International Standard is based on the following documents:

Draft	Report on voting
10/1163/FDIS	10/1173/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

INTRODUCTION

General caution, health, safety and environmental protection

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

The insulating oils which are the subject of this document should be handled with due regard to personal hygiene. Direct contact with the eyes can 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 document involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

This document is applicable to mineral oils and non-mineral oils, chemicals and used sample containers.

Attention is drawn to the fact that some mineral oils in service can still be contaminated to some degree by PCBs. If this is the case, safety countermeasures should be taken to avoid risks to workers, the public and the environment during the life of the equipment, by strictly controlling spills and emissions. The disposal or decontamination of these oils can be subject to regulatory requirements with regard to their impact on the environment. Every precaution should be taken to prevent release of mineral oil and non-mineral oil into the environment.

METHOD OF SAMPLING INSULATING LIQUIDS

1 Scope

This document is applicable to the sampling procedure used for insulating liquids in delivery containers and in electrical equipment such as power and instrument transformers, reactors, bushings, oil-filled cables, oil-filled tank-type capacitors, switchgear and load tap changers (LTCs).

This document applies to liquids the viscosity of which at the sampling temperature is less than 1 500 mm^2/s (or cSt). It applies to mineral oils and non-mineral oils (such as synthetic esters, natural esters, vegetable oils or silicones).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 60567:2011, *Oil-filled electrical equipment – Sampling of gases and analysis of free and dissolved gases – Guidance*

IEC 60970, *Insulating liquids – Methods for counting and sizing particles*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
delivery container
container used to store, transport and deliver batches of oil

EXAMPLE Drum, rail tanker, road tanker, flexible plastic bag.

3.2
electrical equipment
equipment filled with insulating oil

EXAMPLE Power and instrument transformers, reactors, bushings, oil-filled cables, oil-filled tank-type capacitors, switchgear and load tap changers (LTCs).

3.3
sampling equipment
equipment used for sampling oil from delivery containers (e.g. sampling probes, such as dippers or siphons) and from electrical equipment (e.g. connecting tubing and drain valve adapters)

Note 1 to entry: Sampling equipment also includes sample containers, waste oil containers and other accessories.

3.4
sample container
container or other device used to store and transport samples of oil for analysis

EXAMPLE Syringe, bottle, ampoule.

Note 1 to entry: Sample containers include accessories such as valves, tubing or caps attached to the container.

4 General principles for the sampling of insulating liquids

4.1 New insulating liquids in delivery containers

4.1.1 Place of sampling

The sample shall be taken from the part of the delivery container where the insulating liquid is likely to be most heavily contaminated. To evaluate the quality of a consignment, two types of samples may be normally taken:

a) composite sample: mixture of samples taken at the same level in several containers;

b) individual sample: sample or mixture of samples taken at the same level in one container.

From a delivery, individual samples of 1 l may be taken from different containers for the electric strength test. Further tests may be carried out on these samples and a complete examination on the mixture of these (composite sample).

In certain cases, it can be useful to constitute an average sample within the container. An average sample is a mixture of samples taken at different levels in one container.

1) tankers: samples should be taken from each tanker as described in 4.1.4.2 below;

2) drums: samples should be taken as described in 4.1.4.3 below.

In the case of a single drum, this shall be sampled.

In case there is more than one drum of a lot of oil, sampling procedures should be negotiated between supplier and user. For example, samples can be taken from 10 % of drums or at least two drums, whichever the largest.

4.1.2 Quantity of sample to be taken

This depends on the tests to be performed and the procedures used.

Typically, 2 l are taken.

4.1.3 Sampling equipment

4.1.3.1 General

Since the results of the tests included in IEC requirements for insulating liquids can greatly depend on the impurities in the sample, it is essential to observe the following precautions:

– separate sampling equipment shall be reserved exclusively for each type of liquid. All seals and tubing used should be compatible with the insulating liquid to be sampled;

– the equipment shall be clean and dry, following the cleaning procedures described in 4.2.1.6. Particular care should be taken to ensure the absence of any traces of solid impurities, such as dust, fibres. The use of rags for cleaning is not permitted.

4.1.3.2 Sampling probes

As examples, four types of sampling probes are described below. Other probes may also be used, provided they do not introduce any contamination. Stainless steel and aluminium are suitable.

a) Sampling from tankers

The thief dipper shown in Figure 1 is suitable for taking samples at the bottom of the container. This is a dipper constructed of stainless steel or aluminium tubes and castings, machine-finished all over. It shall be sufficiently heavy to sink in the liquid. It should always be suspended by means of a metal wire or chain. String or other fibrous materials shall not be used.

The cream dipper is used for taking top samples of insulating liquids. This probe shall be constructed as shown in Figure 2 and shall be of stainless steel.

Dimensions in millimetres

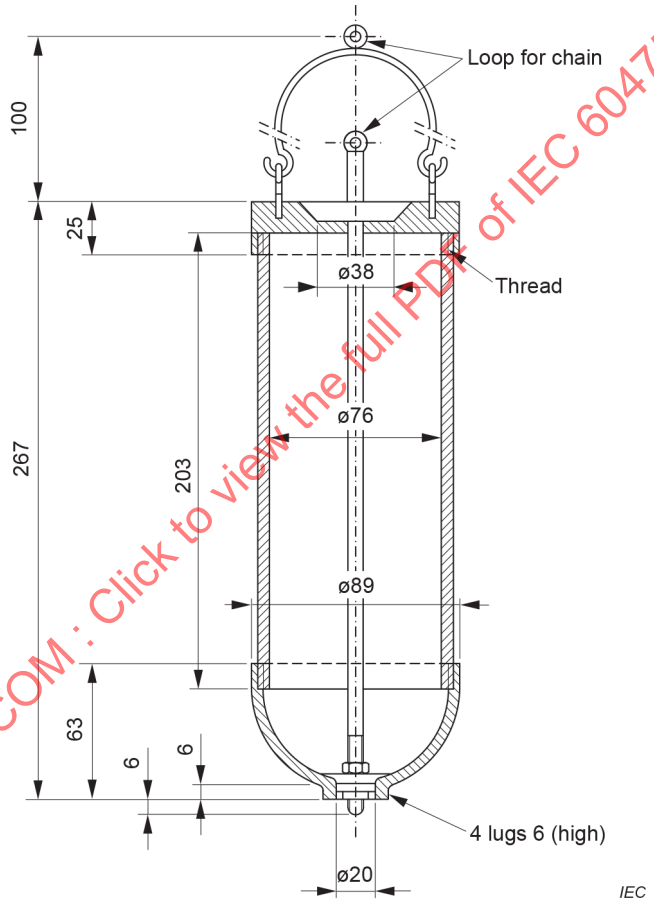


Figure 1 – Thief dipper

Dimensions in millimetres

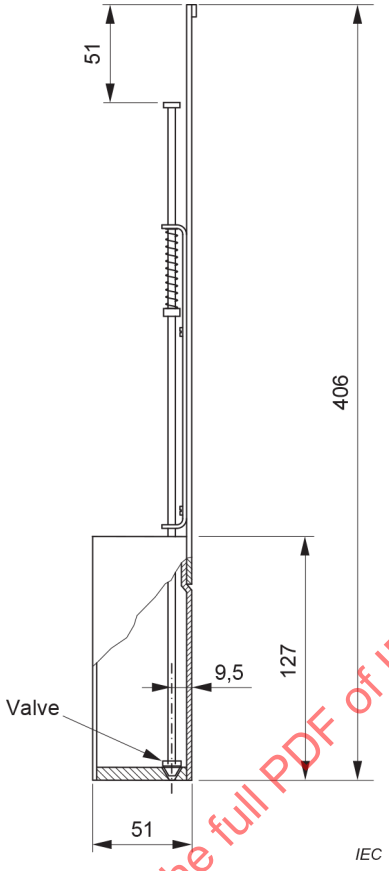


Figure 2 – Cream dipper

b) Sampling from drums

The pipette shown in Figure 3 enables samples to be taken at the bottom of drums. This pipette has a capacity of about 500 ml.

Another probe to take samples at the bottom is shown in Figure 4; it is a siphon with a glass, stainless steel or aluminium tube having an internal diameter of about 13 mm for taking off the sample liquid, and a metal tube (internal diameter 5 mm) for applying pressure. Both tubes are set in an oil-resistant bung whose dimensions correspond to the diameter of the bung hole in the drum. Commercial versions of this equipment are available. When possible, glass is preferable for probes illustrated in Figure 3 and Figure 4.

The cream dipper (Figure 2) may be used for taking top samples.

Dimensions in millimetres

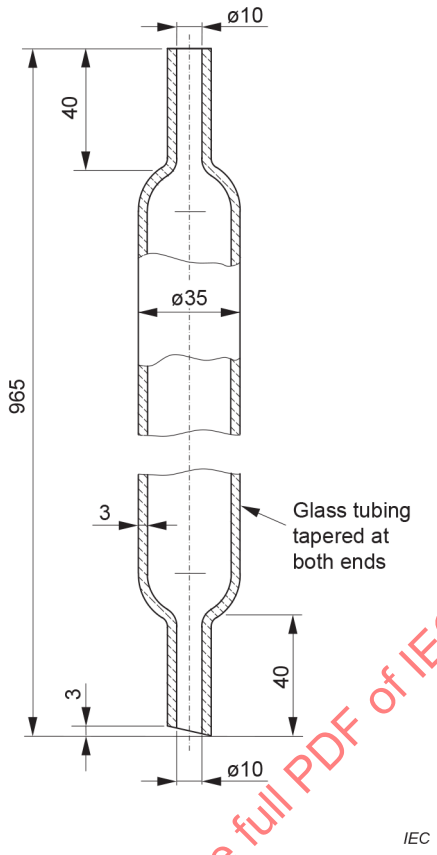


Figure 3 – Pipette

Dimensions in millimetres

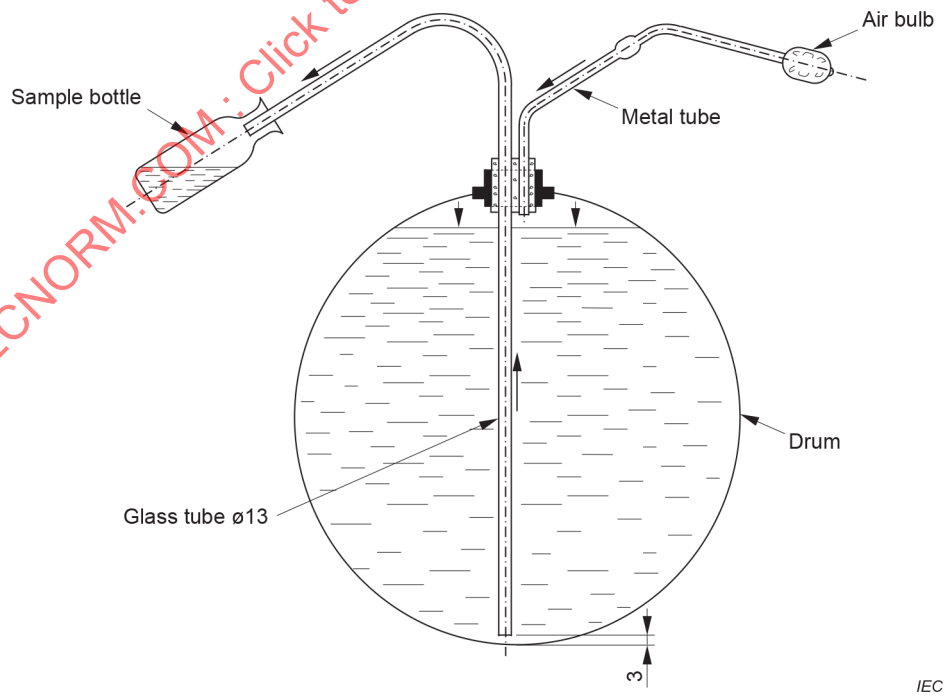


Figure 4 – Siphon

4.1.3.3 Sample containers

For storing and transporting samples, depending on the oil test to be performed, sample containers of appropriate volume shall be used. Different types of sample containers are indicated in 4.2.1.5.

For the mixing of different samples, a special sample container made of glass with a capacity of at least 6 l shall be used. These special sample containers shall be closed in a manner that allows them to be sealed, by means of oil-resistant plastic or compatible rubber tubing or screw caps equipped with a polytetrafluoroethylene (PTFE) lining. Natural rubber tubing and/or seals are not permitted. PTFE and polypropylene (PP) seals are acceptable.

Each sample container shall have a label on which are marked all the indications necessary to identify the contents, i.e. the markings of the drums or tanks, date of sampling and the name of the recipient.

4.1.3.4 Cleaning of sampling equipment

Sampling equipment shall be cleaned following the procedures described in 4.2.1.6.

4.1.4 Sampling procedure

4.1.4.1 General remarks

According to general principles for sampling (see 4.1.1), samples of new insulating liquid shall be taken from the bottom of the delivery container, where the contamination is likely to be the greatest. But in certain cases, an average sample is also of interest.

NOTE 1 To obtain an average sample, samples are taken at intermediate levels in tanks or drums. Examples of procedures are given in Annex A. A procedure is indicated in the NOTE in 4.1.4.2 a) for obtaining the equivalent of an average sample.

In Table 1 different cases are considered:

Table 1 – Types of samples of new insulating liquids

Type of delivery	Recommended sampling	Equipment to be used	Procedure	Recommended quantity
Drums	Composite	Pipette (Figure 3) or Siphon (Figure 4)	4.1.4.2	3 × 2 l
	Individual		4.1.4.2	1 l or 2 l
Tankers	Individual	Thief dipper (Figure 1) or None (valve)	4.1.4.1 b) 4.1.4.1 a)	3 × 2 l
Drums	Average	Pipette or Siphon (Figure 3 and Figure 4)	Annex A	3 × 2 l
Tankers	Average	Thief dipper (Figure 1) or None (valve)	4.1.4.1 a)	

NOTE 2 Before sampling from tankers, sufficient oil is pumped from the end of the delivery pipe, as indicated in 4.1.4.2.

Every precaution shall be taken during sampling in order to avoid contamination of the insulating liquids. Outdoor sampling of insulating liquids in rain, fog or high wind is only permitted if all precautions have been taken to avoid contamination of the liquid. In this special case, the use of a cover is necessary.

Condensation shall be avoided by warming the sampling equipment so as to be above the ambient air temperature. Before use, the equipment shall be rinsed with the liquid being sampled. The operator shall be warned not to let their hands come into contact with the surfaces of sampling equipment subsequently in contact with the oil. The insulating liquids shall be protected against light irradiation during transportation and storage.

On arrival at the laboratory, the sampling container shall not be opened immediately. It is necessary to wait until the temperature of the sample is the same as the room temperature.

4.1.4.2 Sampling from tankers

Insulating liquids may be sampled either through the tank outlet or by a thief dipper or by a cream dipper.

a) Sampling through the tank outlet

By this procedure, it is possible to obtain a sample representative of the bottom of the tank after the tank has been allowed to stand for at least 1 h after the vehicle has arrived.

NOTE It may be possible, by this procedure, to obtain the equivalent of an average sample, if the sampling is done directly after the vehicle has arrived.

In this case, the sampling procedure shall be as follows:

– remove the outlet valve shield, if fitted;
– remove all visible dirt and dust from the valve by means of lint-free clean cloths or oil-resistant synthetic sponges;
– the outlet system (pump, delivery pipe), if incorporated, shall be started or opened as appropriate in order to get a sample;
– open the valve and allow to flow, slowly, at least 10 l of insulating liquid into a waste oil container. In any case, discard at least an amount of oil equivalent to the volume of the pipe;
– rinse sampling bottles with the insulating liquid;
– fill sampling bottles at constant flow to avoid turbulence.

b) Sampling with a thief dipper or a cream dipper

This sampling should be carried out after the tank has been allowed to stand for at least 1 h after the vehicle has arrived.

1) Procedure with the thief dipper (Figure 1) (bottom samples)

For taking bottom samples (i.e. within 1 cm to 2 cm from the bottom of the tank) the dipper is lowered until the projecting stem of the valve rod strikes the bottom of the tank. The dipper then fills. Filling is complete when no more air bubbles escape. The dipper is then withdrawn and its contents poured into the sample container (in the case of an individual sample) or into the special glass sample container for collecting and mixing the various samples taken (in the case of a composite sample). In this latter case, the sample container(s) is (are) filled with the mixture so obtained. During pouring of the liquid, avoid forming air bubbles by pouring too quickly.

2) Procedure with the cream dipper (Figure 2) (top samples)

With the valve closed, fill the cream dipper by slowly immersing it in the liquid to be sampled until the rim is just below the surface of the liquid so that it will flow slowly into the dipper. Discard the first filling. Refill the dipper as above and transfer the sample to the sample container by allowing it to flow from the bottom orifice against the side of the sample container and not in a stream into the bottom of the sample container. Repeat the operation until sufficient liquid is obtained to fill the sample container (individual sample) or the special glass sample container used for mixing samples depending on the type of sample to be obtained.

4.1.4.3 Sampling from drums

Samples should be taken after the drums have been allowed to stand for at least 8 h with the bung uppermost, protected against rain and rainwater. For sampling the bottom (i.e. 3 mm up), the pipette (Figure 3) or the siphon pressure thief (Figure 4) may be used.

For taking a sample from the surface layer of the liquid, the cream dipper (Figure 2) may be used.

Examples of procedures:

a) Use of pipette (Figure 3) (bottom samples)
   - block the upper orifice of the pipette with the thumb, and then immerse the pipette in the liquid to the bottom of the drum;
   - remove the thumb to allow liquid to enter the pipette;
   - again close the upper end of the pipette with the thumb and withdraw the pipette;
   - the first filling is used for rinsing the pipette; transfer the next fillings into either a sample container (individual sample) or the special glass sample container for mixing samples (composite sample) (see 4.1.4.2 b)) taking care not to form air bubbles during pouring the liquid.

b) Use of siphon (Figure 4) (bottom samples)
   - fit the bung in which are set the riser and pressure tubes into the bung hole of the drum and ensure that this seal is airtight;
   - dip the lower end of the riser tube to about 3 mm from the bottom of the drum;
   - raise the pressure inside the drum by means of the air bulb;
   - run off enough liquid to rinse the tube and then run off the required quantity directly into the sample container (individual sample) or the special glass sample container for mixing samples (composite sample) (see 4.1.4.2 b)) taking care not to form air bubbles during pouring of the liquid.

c) Use of cream dipper device (Figure 2) (top samples)

See 4.1.4.2 b).

4.1.4.4 Sampling report

The sampling report shall give all the information necessary for identifying the sample as well as any details or special information likely to be of help to those entrusted with the tests. The type of sample (i.e. composite, individual or average sample) shall be specified. A copy of the report shall accompany each sample. The distribution of samples shall be in accordance with the agreed procedure, for example as given in the sales contract.

4.2 Sampling of oil from oil-filled equipment

4.2.1 General remarks

4.2.1.1 Safety and quality of sampling

The manufacturer's instructions for taking oil samples from the electrical equipment shall be followed. Particular attention shall be paid to the safety precautions to be taken.

Ensure that the oil in the energized electrical equipment is not under a negative pressure when taking an oil sample, since this could introduce air bubbles in the oil, induce electrical short-circuits in the equipment and put the sampling personnel at risk.

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

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

In all cases, oil sampling should be performed by properly and specifically trained personnel, especially for low volume equipment (e.g. instrument transformers).

4.2.1.2 Place of sampling

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 (for example, from the bottom oil drain valve or the oil sampling valve). 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, such as from the tap changer, selector switch or gas relay).

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.

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.

4.2.1.3 Cleaning of sampling point

Cleaning of the sampling equipment and flushing of the sampling point shall be done to prevent contamination of oil samples.

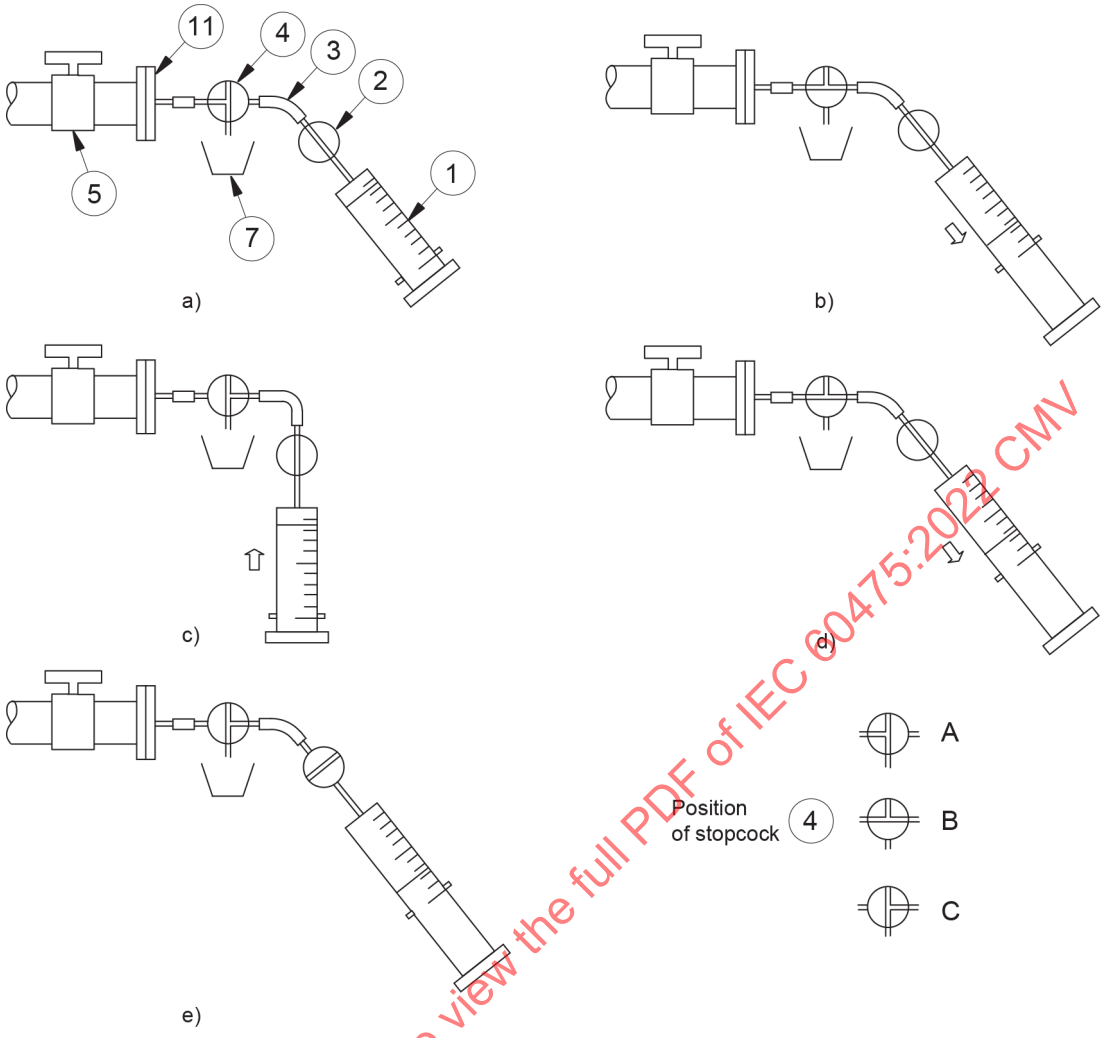
The blank flange or cover (11) of the sampling valve in Figure 5, Figure 6 and Figure 7 a) is removed and the outlet cleaned with a lint-free cloth or oil-resistant synthetic sponge to remove all visible dirt.

The drain valve is flushed with a sufficient quantity of oil (typically, 2 l to 5 l), under a turbulent flow, to eliminate any contaminants (water and particles) that might have accumulated in the drain valve and at its orifice.

Use protection gloves, preferably made of nitrile rubber, and a bucket for waste oil. The sampling point shall be cleaned each time a new sample of oil is taken.

For measuring water content in oil, sampling shall be carried out during days when the humidity of the air is as low as possible, to prevent moisture condensation on sampling equipment and contamination of the oil sample.

The temperature of oil at sampling point shall be measured with a thermometer placed in the flow of oil and indicated on the sample, to ensure the calculation of the relative humidity of oil, also recording whether or not the fans and pumps are running. In both cases, the method used to measure the temperature shall be indicated.

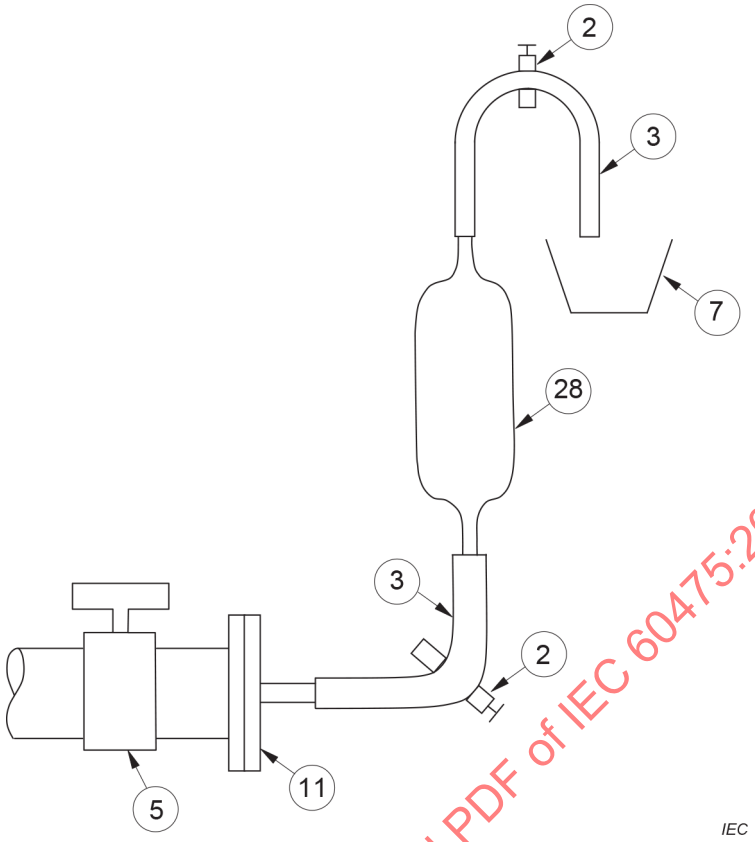


Key

a flushing position
b wetting and flushing of syringe
c emptying of syringe
d taking of sample
e disconnecting of syringe

1 syringe
2 stopcock
3 flexible connecting tubing
4 three-way valve
5 equipment sampling valve
7 waste vessel
11 blank flange

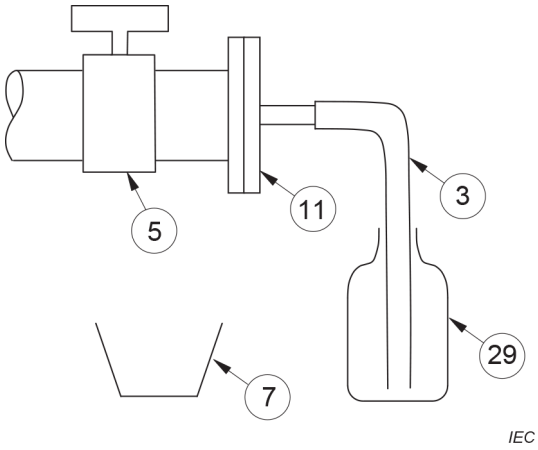
Figure 5 – Sampling of oil by syringe



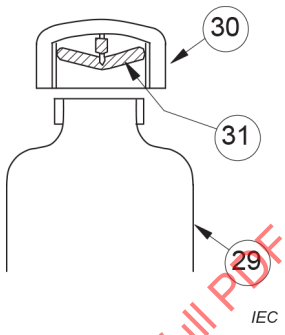
Key

2	stopcock	7	waste vessel
3	flexible connecting tubing	11	blank flange
5	equipment sampling valve	28	sampling tube

Figure 6 – Sampling of oil by ampoule



a) Example of sampling by bottle



b) Example of seal cap for bottle

Key

3	flexible connecting tubing	29	bottle
5	equipment sampling valve	30	hard plastic screw cap
7	waste vessel	31	conical soft polyethylene seal
11	blank flange		

Figure 7 – Sampling of oil by bottle

4.2.1.4 Connection between sampling point and sampling device

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

Attach a piece of oil-compatible plastic or rubber tubing to connect the sampling point to the sampling device. This tubing should be as short as possible. To avoid contamination by the previous oil sample, use a new piece of tubing, or flush the tube well and wash its outer surface with the next oil to be sampled.

Suitable tubing should be made for example of perfluorinated material (e.g. Viton®[1], Tygon®), PTFE or metal, not PVC.

[1] Viton® and Tygon® are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of these products.

4.2.1.5 Choice of sample container

Table 2 indicates the different types of sample containers that can be used depending on the oil analysis to be made.

Table 2 – Sample containers for oil tests

Sample container	Syringe	Flexible bottle	Bottle	Flexible bottle	Ampoule	Ampoule	Oil volume
Material	Glass	Metal	Glass	Plastic	Glass	Metal	ml
Oil test							
Dissolved gases	Y	Y	Y		Y	Y	25 to 100
Water	Y	Y	Y				20
Dielectric dissipation factor	Y	Y	Y	Y			200
Particles	Y	Y	Y	Y			100
Breakdown voltage		Y	Y				500 to 1 000
Other chemical and physical tests		Y	Y	Y			250
All tests							1 000 to 2 000
Volume (ml)	25 to 250	125 to 2 500			125	25 to 250	
Key Y = Yes							

Metal or plastic containers may be preferred where adequate protection of glass containers is not available for the transportation of oil samples.

For dissolved gas analysis (DGA), to minimize losses of the low solubility gases (H_2 and CO) and pick-up of air at low total gas contents, it is critical to strictly follow the sampling procedures of 4.2.2 to 4.2.5, particularly with bottles and ampoules. In addition, only the materials recommended for caps, gaskets, valves and tubing of sample containers should be used by well-trained and experienced personnel familiar with those containers.

When using bottles for DGA, water and breakdown voltage, care should be taken to minimize air contact with the oil sample during sampling and analysis.

The use of plastic bottles is not permitted for DGA, water and dielectric strength, since ambient air contamination and gas losses can occur by diffusion through the plastic. For the other tests, plastic bottles should be made of a compatible plastic (such as high-density polyethylene (HDPE), polypropylene or polycarbonate), which does not contaminate the oil with additives contained in the plastic. Each new type of plastic (and glass) bottle should be tested for compatibility with oil.

The recommendations of 4.2.1.3 and 4.2.1.6 (cleaning of sampling point and sampling equipment) should be followed strictly for water content, dielectric strength, dielectric dissipation factor, interfacial tension and particles content.

The other physical and chemical tests (viscosity, density, acidity, DBPC, furans and PCB contents, etc.) are less affected by the sample containers and sampling procedures used.

4.2.1.6 Cleaning of sampling equipment

4.2.1.6.1 Use of disposable sample containers

Use of disposable, pre-cleaned metal, plastic and glass bottles having a known level of cleanliness for dust and humidity has been found by several users more convenient than cleaning them. Such sampling devices are relatively inexpensive and available from several vendors of labware or veterinarian equipment. To verify that the cleanliness of disposable bottles is acceptable, a few un-cleaned and cleaned bottles can be tested in parallel.

4.2.1.6.2 Cleaning procedures

Non-disposable sampling devices may be cleaned in a dishwasher using a detergent, and rinsed with tap water (without detergent in the rinse aid compartment of the dishwasher). A last, optional rinse with de-ionized water may be used.

Sampling equipment and containers may also be cleaned with normal heptane.

After cleaning, the sampling devices are dried in an oven at typically 100 °C until fully dried, then allowed to cool in the oven or a dry box.

After drying, they shall be immediately closed to protect from contamination and not opened until just before use.

Appropriate cleaning of sample containers is critical for DDF and interfacial tension, which are particularly sensitive to contamination. They should not be cleaned with solvents.

Dedicated sampling containers shall be cleaned according to IEC 60970 for measuring particles content in oil.

4.2.2 Sampling of oil by syringe

4.2.2.1 Sampling equipment

The following sample equipment shall be used:

a) Graduated gas-tight syringes of a size suitable for containing adequate oil sample volume (20 ml to 250 ml), and equipped preferably with a three-way plastic valve made of nylon body and polypropylene (PP) barrel, or with a three-way stainless steel valve. The use of syringes with matched piston and barrel is preferred when sampling for DGA in order to allow the piston to flow freely with oil volume variations, and to avoid pressure and vacuum build-up in the syringe and breakage during handling. Plastic syringes should not be used.

   For plastic three-way valves, a new valve should be used each time an oil sample is taken and not recycled, because it can be contaminated with the previous oil sample and lose its gas tightness when used several times. For added protection during transportation, a stainless steel Luer-Lock cap may be placed on top of the three-way valve. This cap may be recycled after use.

   NOTE Priming the piston with clean, degassed oil has been found useful to avoid the formation of bubbles along the piston when introducing the oil sample for DGA analysis. The use of a low viscosity water-soluble lubricant has also been found useful for DGA.

   The size of sample required depends on the likely concentration of gas in the sample, the analytical techniques and the sensitivity required. For DGA after factory tests, a 250 ml syringe has been found convenient.

   See Annex B.

b) Transport containers, designed to hold the syringe firmly in place during transport but which allow the syringe plunger freedom to move and prevent its tip from contacting the container whatever its position during transportation. Cardboard boxes with removable inner cardboard flaps that hold the barrel in place have been found convenient for that purpose. Metal or plastic cylinders with inside foam packing have also been found appropriate for transportation. When sampling for DGA, the syringe should preferably be transported in the vertical position, piston upwards, to avoid the formation of bubbles in oil.

4.2.2.2 Sampling procedure

See Figure 5.

a) The electrical equipment is connected as shown in Figure 5 a), and its sampling valve (5) opened.

b) The three-way valve (4) is adjusted (position A) to allow 1 l to 2 l of oil to flow to waste (7).

c) The three-way valve (4) is then turned (position B) to allow oil to enter the syringe slowly (Figure 5 b)). The plunger should not be withdrawn but allowed to move back under the pressure of the oil.

d) The three-way valve (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 5 c). Confirm that the inner surfaces of the syringe and plunger are completely oiled.

e) The procedure described in steps c) and d) is then repeated until no gas bubble is present. Then the three-way valve (4) is turned to position B and the syringe filled with oil (Figure 5 d).

f) The three-way valve (4) on the syringe and the sampling valve (5) are then closed.

g) The three-way valve (4) is turned to position C and the syringe disconnected (Figure 5).

h) When sampling for DGA, if the oil taken from the electrical equipment is hot, place the syringe in its protective box in the vertical position, standing on the piston and with the syringe tip upwards, until the oil has slowly cooled down, then install the syringe back into the holding flaps of the protective box for transportation. This will prevent the formation of bubbles in oil.

Label the sample in accordance with 4.4.

NOTE 1 It is good practice to avoid contamination of the outer surface of the plunger and inner surfaces of the syringe by dust or sand. Such particles can affect the sealing properties of the syringe. This kind of contamination can come from wind-swept dust or from the handling of the syringe.

NOTE 2 In the case of sealed transformers, if a bubble appears in the syringe directly after sampling, the sampling procedure is repeated.

4.2.3 Sampling of oil by ampoule

4.2.3.1 Sampling equipment

The following sample equipment shall be used:

a) Glass or metal ampoule, typically of volume 125 ml to 1 l. It may be closed either by stopcocks or pinchcocks on oil-compatible plastic tubing or by valves. Glass ampoules are usually made of Pyrex®[2] glass. Metal ampoules are made of stainless steel and can use spring-loaded valves instead of plastic tubing as expansion devices.

   The oil-compatible plastic tubing used for ampoules should be used only once, not recycled, since it has a memory effect and can contaminate the oil sample when sampling for DGA. The types of compatible plastic tubing are indicated in 4.2.1.4.

[2] Pyrex® is the trademark of a product supplied by Corelle Brands. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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 tests to be carried out and, for DGA, the likely concentration of gas in the sample, the analytical technique and the sensitivity required. For DGA after factory tests, a 250 ml ampoule has been found convenient.

b) Transport containers, designed to hold the sampling tubes firmly in place during transport.

4.2.3.2 Sampling procedure

See Figure 6.

a) The device is connected as shown in Figure 6.

b) The cocks (2) on the plastic tubing of the sampling ampoule (28) and the equipment sampling valve (5) are carefully opened so that oil flows through the sampling ampoule to waste (7). When sampling for DGA, oil should flow under a non-turbulent flow (until there are no air bubbles in the oil), to avoid the formation of bubbles in oil and the stripping of dissolved gases out of oil.

c) After the sampling ampoule (28) has been completely filled with oil, about 1 l to 2 l are allowed to flow to waste (7).

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 ampoule (28) is then disconnected and the sample labelled in accordance with 4.4.

NOTE If a glass sampling ampoule 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 ampoule in the event of it being exposed to a rise in ambient temperature. Once completed, this procedure is also recorded on the label.

4.2.4 Sampling of oil by flexible metal bottles

4.2.4.1 Sampling equipment

The following sample equipment shall be used:

a) Flexible metal bottles capable of being sealed gas-tight, typically of volume 250 ml to 2,5 l.

Metal bottles should not be soldered, as materials used for soldering can contaminate the oil. Adsorption on aluminium surfaces of water contained in oil is possible. Metal bottles made of drawn aluminium or of welded tin are flexible and do not need oil expansion devices. They should be filled completely with oil by pressing on the bottle sides before closing the bottle.

Metal bottles should be closed with a screw cap lined with a non-porous, leak-free gasket compatible with oil. Gaskets should be used only once, not recycled, except if they are lined with aluminium foil on the oil side.

For DGA and water analysis, the porosity of gaskets used should be measured by taking at least six samples of oil from a transformer into identical bottles. The hydrogen content of the oil used for testing of the sampling equipment should be at least 100 µl/l. Analyse samples for hydrogen content at intervals over a month, the first being as soon as possible after taking the samples. A bottle and seal design is acceptable if it permits losses of hydrogen of less than 2,5 % per week. Suitable bottles have, for example, screwed plastic caps holding a conical polyethylene (PE) seal or flexible gasket (see Figure 7).

For analytical tests other than DGA and water, the above requirement for gas tightness does not apply.

For mineral oils, gaskets should be made of polyethylene (PE), PTFE or nitrile-butadiene rubber (NBR) (containing more than 30 % of nitrile component).

For non mineral oils (e.g. natural and synthetic esters), gaskets should be made of PTFE (not NBR or silicone rubber).

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

4.2.4.2 Sampling procedure

See Figure 7 a).

a) The sampling valve (5) is carefully opened and about 1 l to 2 l of oil allowed to flow under a laminar flow to waste (7) through the tubing (3) ensuring that all gas bubbles are eliminated before the oil sample is collected and gases are not stripped out of the oil by the oil flow.

b) 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 from bottom up. Rinse the bottle with one-third of oil then send the oil to waste.

   When sampling for DGA, introduce the oil under a continuous, non-turbulent flow, until no gas bubbles are observed in oil when it flows out of the bottle in order to avoid the formation of bubbles in oil and the stripping of dissolved gases out of oil (otherwise significant gas loss can occur). Filling the bottle should be slow enough to allow laminar flow of oil and as fast as possible to avoid gas loss to (and contamination from) the atmosphere. If the time to fill the bottle exceeds a few minutes, a new sample should be taken.

   When sampling for water, strictly follow the recommendations of 4.2.1.3.

c) Allow about two bottle volumes to overflow to waste (7), then withdraw the tubing (3) slowly with the oil still flowing. Gently squeeze the sides of the bottle so it is entirely filled with oil, then securely close with the cap.

d) Close the sampling valve (5) and disconnect the tubing. Label the sample (see 4.4).

   Tighten the cap again after the oil has cooled to ambient temperature.

4.2.5 Sampling of oil by glass and rigid metal bottles

4.2.5.1 Sampling equipment

The following sample equipment shall be used:

a) Glass or rigid metal bottles capable of being sealed gas-tight, typically of volume 125 ml to 2,5 l. Clear glass bottles shall be protected from sunlight, so the use of dark bottles is highly recommended. Even so, for samples for DGA, extra protection from light should be provided during transport and storage.

   Caps and gaskets described for flexible metal bottles in 4.2.4.1 are suitable for glass and rigid metal bottles.

b) Transport containers, designed to protect the bottle during transportation.

4.2.5.2 Sampling procedure

See Figure 7 a).

Sampling procedures are the same as for flexible metal bottles in 4.2.4.2, except that glass and rigid metal bottles should not be filled entirely with oil.

Instead, allow the oil level to fall a few centimetres from the rim so as to leave a small expansion volume of air (typically, 3,5 ml to 7 ml, or 1,5 cm to 3 cm of airspace), to allow for oil expansion with increasing temperatures. Fill with no less than 90 % of oil to allow for air expansion when temperatures decrease and avoid implosion of the glass bottle. Place the bottle cap securely in position and label the sample (see 4.4). Indicate the approximate expansion volume of air on the label. Correction for gas loss to the small headspace volume of air in the bottle shall be calculated by the laboratory as indicated in Annex D of IEC 60567:2011.

Where transport and storage conditions are not particularly demanding, some companies prefer to have the bottles filled completely and closed lightly, finger-tight, with a screwed plastic cap having a conical polyethylene seal. In the event of expansion of the oil by heat, these caps act as a non-return valve, allowing a small amount of oil to escape. Where contraction by cooling occurs, the seal will prevent ingress of air. In the latter case, it is necessary for the bottle to be warmed up to the sampling temperature to re-dissolve the gases prior to analysing for dissolved gases.

For other analytical tests, an airspace can be left above the oil.

4.2.6 Sampling of oil by plastic bottles

4.2.6.1 Sampling equipment

The following sample equipment shall be used:

Plastic bottles should be made of a compatible plastic (see 4.2.1.5), which does not contaminate the oil with additives contained in the plastic. Each new type of plastic bottle should be tested for compatibility with oil. Use of virgin plastic without fillers or pigments is strongly recommended.

Plastic bottles should not be used for DGA, water content and dielectric breakdown.

Caps and gaskets described for metal bottles in 4.2.4.1 are suitable for plastic bottles.

Moulded all-plastic caps, of suitable composition as above, are suitable.

4.2.6.2 Sampling procedure

See Figure 7 a).

Sampling procedures are the same as for flexible metal bottles in 4.2.4.2.

4.3 Storage and transportation of samples

Some of the dissolved oxygen present in the oil sample can be consumed, and hydrocarbons and carbon oxides formed by oxidation. This reaction is accelerated by exposure to light, therefore sampling devices made of transparent materials (syringes, glass bottles and ampoules) should be protected (for example, by wrapping them in an opaque material or placing them in a box for transportation).

In any case, the analysis should be carried out as soon as possible after sampling to avoid oxidation reactions and gas losses or pick-ups from the sampling devices.

Oil syringes (and other oil sampling devices) may be placed in sealed boxes to fully eliminate the risk of formation of bubbles in important DGA oil samples during transportation in planes, due to reduced pressure and over-saturation of gases in the oil. The syringe plunger should be allowed to move in order to prevent air ingress in case of oil volume variations.

4.4 Labelling of samples

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

The following information, as shown in Table 3, is necessary (whenever it is known).

Table 3 – Information required on oil sample labels

Transformer or other equipment	Sampling
Customer	Sampling date
Location	Sampling point
Identification number	Sampling person
Manufacturer	Reason for analysis (routine or other)
General type (transformer (generation or transmission, instrument, industrial), reactor, cable, switchgear, etc.)	Transformer non-energized, off-load energized or on-load
Rated MVA	Oil temperature when sampling
Voltage ratio	Humidity: dry – wet – fog – indoors
Type and location of OLTC	
Date of commissioning	
Oil	
Type of oil (mineral or non-mineral)	Weight (or volume) of oil
Product name	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 percentage 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;

– for water in oil analysis, temperature of oil, the method used for measuring the temperature, and whether or not the fans and pumps are running (to be able to calculate the relative humidity of oil);

– time of sampling where more than one sample is taken.

Annex A
(informative)

Procedure for sampling at intermediate levels (making up of the average sample)

A.1 Use of the thief dipper

See Figure 1 and 4.1.4.2 b). The thief dipper is immersed to the required depth. The chain attached to the central rod is then pulled, care being taken that vertical displacement of the rod does not exceed 50 mm. The dipper then fills; filling is complete when no more air bubbles escape. The dipper is then withdrawn and its contents poured into the mixing container.

A.2 Use of the pipette

See Figure 3 and 4.1.4.3. The pipette is immersed to the required depth.

A.3 Use of the siphon

See Figure 4 and 4.1.4.3. The siphon is immersed to the required depth.

A.4 General remark

The samples taken at intermediate levels for making up the average sample are transferred in the mixing container for collecting the samples as soon as they are taken. The mixture is then used to fill sampling bottles.

Annex B
(informative)

Procedure for testing the integrity of the syringes

The procedure is as follows:

a) Connect the syringe to be tested to a three-way valve (4.2.2.1).
b) Move the valve to the open position (position B or C as shown in Figure 5).
c) Press the piston fully into the syringe.
d) Close the valve by adjusting in position A.
e) Try to pull the piston from the syringe and hold it under tension for about 30 s.
f) After releasing the piston, it should return in the original position.
g) If any amount of air was locked between the piston and the syringe body, then the syringe or the valve is not hermetically sealed, and it is recommended that the syringe or the valve be replaced by a new one.

Annex C
(informative)

Procedure for sampling oil from bushings

The general procedures for sampling oil from electrical equipment are described in 4.2.1.1, 4.3 and 4.4.

The specific procedures for sampling oil from bushings are the following:

The oil sampling should be carried out on de-energized bushings in their normal position. This is important in assessing bushing condition.

When sampling, precautions should be taken to deal with any sudden release of oil and prevent any moisture and dust entry in the bushing and the sample.

With small oil volume bushings it is essential to ensure that the total volume of oil drawn off does not endanger the operation of the bushing. See NOTE 2 below.

The method of sampling by syringe is the preferred method of oil sampling from bushings (see 4.2.2) and is suitable irrespective of the mode of transport of samples, but other sampling devices are also applicable if compliant with the manufacturer's instructions.

WARNING – The manufacturer's instructions should be consulted before any sampling. Failure to follow the instructions from the manufacturer can lead to serious damage and bushing failure. Normally the amount of oil in bushings is limited, therefore it is not usual to carry out routine sampling. Where sampling is carried out, the effect on the oil level and pressure should be taken into consideration. Oil sampling from oil impregnated paper (OIP) bushings without an oil level gauge is not recommended.

a) Bushings fitted with a sampling point at the mounting flange
   1) Remove the protective cover of the sampling point. Clean the outlet with a lint-free cloth to remove all visible dirt (see also 4.2.1.3).
   2) Ensure that all parts of the sampling component are clean and dry.
   3) Connect the tubing to the sample point.
   4) Flush the tube as described in 4.2.2.2 b) to e) and in Figure 5.
   5) Obtain the sample for analysis as described in 4.2.2.2 e) to h) and in Figure 5.
b) Bushing not fitted with a sampling point at the mounting flange

In this case, it may be possible to take a sample from the top of the bushing. The manufacturing instructions should be consulted to determine a suitable position. Insert one end of the sampling tube into the bushing, from the top, and connect to the other end to the three-way stopcock on the syringe, using a plastic connector. Follow the same procedure as above.

In the case of bushings pressurized at ambient temperature, the procedure described in a) and b) above is not applicable. Reference should be made to the instructions of the equipment manufacturer. Oil-SF6 or oil-oil bushings, in particular, in which internal oil is in a close volume, with a bellow for oil dilatation, shall always keep a positive oil pressure to prevent air and moisture ingress when sampling. On those bushings the sampling point is always at the mounting flange.

Oil replacement: The oil removed from the bushing shall be replaced if the volume of oil extracted is more than 1 % of the total oil volume in the bushing.

The removed oil is replaced with an appropriate volume of clean, dry and degassed oil compatible with the original oil. Consultation of the manufacturer is highly recommended.

In the case of large bushings, if the oil is not to be replaced immediately, the volume removed shall be noted for replacement during subsequent sampling or maintenance.

Labelling of samples should be made in accordance with Table 3, and include the following additional information on the bushing:

– volume of oil in bushing;
– time out of service;
– storage;
– rated kV;
– rated current;
– manufacturer;
– bushing type;
– phase.

Bibliography

IEC TR 61464, *Insulated bushings – Guide for the interpretation of dissolved gas analysis (DGA) in bushings where oil is the impregnating medium of the main insulation (generally paper)*

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SOMMAIRE

COMMISSION ÉLECTROTECHNIQUE INTERNATIONALE

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MÉTHODE D'ÉCHANTILLONNAGE DES LIQUIDES ISOLANTS

AVANT-PROPOS

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L'IEC 60475 a été établie par le comité d'études 10 de l'IEC: Fluides pour applications électrotechniques. Il s'agit d'une Norme internationale.

Cette troisième édition annule et remplace la deuxième édition parue en 2011. Cette édition constitue une révision technique.

Cette édition inclut les modifications techniques majeures suivantes par rapport à l'édition précédente:

a) ajout de la nouvelle Annexe C sur l'échantillonnage de l'huile dans les traversées, à la demande du sous-comité de l'IEC 36A, afin de transférer vers l'IEC 60475 le contenu correspondant de l'IEC TR 61464 relatif à l'échantillonnage de l'huile dans les traversées;
b) suppression de la NOTE 2 du 4.2.1.2.

Le texte de cette Norme internationale est issu des documents suivants:

Projet	Rapport de vote
10/1163/FDIS	10/1173/RVD

Le rapport de vote indiqué dans le tableau ci-dessus donne toute information sur le vote ayant abouti à son approbation.

La langue employée pour l'élaboration de cette Norme internationale est l'anglais.

Ce document a été rédigé selon les Directives ISO/IEC, Partie 2, il a été développé selon les Directives ISO/IEC, Partie 1 et les Directives ISO/IEC, Supplément IEC, disponibles sous www.iec.ch/members_experts/refdocs. Les principaux types de documents développés par l'IEC sont décrits plus en détail sous www.iec.ch/publications.

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- reconduit,
- supprimé,
- remplacé par une édition révisée, ou
- amendé.

INTRODUCTION

Précautions générales, protection de la santé, de la sécurité et de l'environnement

AVERTISSEMENT – Le présent document ne vise pas à répondre à tous les problèmes de sécurité liés à son utilisation. L'utilisateur du présent document a la responsabilité de mettre en place les pratiques d'hygiène et de sécurité adéquates et de vérifier avant utilisation si des contraintes réglementaires s'appliquent.

Il convient de manipuler les huiles isolantes dont traite le présent document en respectant l'hygiène personnelle. Le contact direct avec les yeux peut provoquer une irritation. En cas de contact oculaire, il convient d'effectuer un lavage avec une grande quantité d'eau courante propre et de consulter un médecin. Certains des essais spécifiés dans le présent document impliquent des processus pouvant conduire à une situation dangereuse. L'attention est portée sur les normes correspondantes à titre de recommandation.

Le présent document s'applique aux huiles minérales et non minérales, aux produits chimiques et aux récipients d'échantillonnage usagés.

L'attention est attirée sur le fait que certaines huiles minérales en service peuvent toujours être contaminées dans une certaine mesure par des PCB. Si c'est le cas, il convient de prendre des contre-mesures de sécurité afin d'éviter les risques pour les travailleurs, le public et l'environnement au cours de la durée de vie de l'appareil, en contrôlant rigoureusement les débordements et les émissions. L'élimination ou la décontamination de ces huiles peut être soumise à des exigences réglementaires vis-à-vis de leur impact environnemental. Il convient de prendre toutes les précautions afin d'empêcher un déversement d'huile minérale et non minérale dans l'environnement.

MÉTHODE D'ÉCHANTILLONNAGE DES LIQUIDES ISOLANTS

1 Domaine d'application

Le présent document s'applique à la procédure d'échantillonnage utilisée pour les liquides isolants dans les récipients de livraison et les matériels électriques tels que les transformateurs de puissance et de mesure, les réactances, les traversées, les câbles à huile fluide, les condensateurs de puissance en huile, les appareillages de connexion et les changeurs de prises en charge (LTC - load tap changer).

Le présent document s'applique aux liquides dont la viscosité, à la température d'échantillonnage, est inférieure à 1 500 mm^2/s (ou cSt). Il concerne les huiles minérales et non minérales (les esters synthétiques, les esters naturels ou les huiles végétales et les liquides silicones, par exemple).

2 Références normatives

Les documents suivants sont cités dans le texte de sorte qu'ils constituent, pour tout ou partie de leur contenu, des exigences du présent document. Pour les références datées, seule l'édition citée s'applique. Pour les références non datées, la dernière édition du document de référence s'applique (y compris les éventuels amendements).

IEC 60567:2011, *Matériels électriques immergés – Échantillonnage de gaz et analyse des gaz libres et dissous – Lignes directrices*

IEC 60970, *Isolants liquides – Méthodes de détermination du nombre et de la taille des particules*

3 Termes et définitions

Pour les besoins du présent document, les termes et définitions suivants s'appliquent.

L'ISO et l'IEC tiennent à jour des bases de données terminologiques destinées à être utilisées en normalisation, consultables aux adresses suivantes:

- IEC Electropedia: disponible à l'adresse https://www.electropedia.org/
- ISO Online browsing platform: disponible à l'adresse https://www.iso.org/obp

3.1
récipient de livraison
récipient utilisé pour stocker, transporter ou livrer des lots d'huile

EXEMPLE Fûts, wagons-citernes, camions-citernes, sacs en plastique flexible.

3.2
matériels électriques
matériels remplis d'huile isolante

EXEMPLE Transformateurs de puissance et de mesure, réactances, traversées, câbles à huile fluide, condensateurs de puissance en huile, appareillage de connexion et changeurs de prises en charge (LTC).

3.3
matériel de prélèvement
matériels qui permettent d'échantillonner l'huile dans des récipients de livraison (sondes d'échantillonnage, écrémoirs ou siphons, par exemple) et des matériels électriques (tuyaux de raccordement et adaptateurs de vanne de vidange, par exemple)

Note 1 à l'article: Le matériel de prélèvement comprend également les récipients d'échantillonnage, les récipients d'huile usagée et les autres accessoires.

3.4
récipient d'échantillonnage
récipient ou autre dispositif de stockage et de transport des échantillons d'huile pour analyse

EXEMPLE Seringues, flacons, ampoules.

Note 1 à l'article: Le récipient d'échantillonnage comprend les accessoires tels que les vannes, les tuyaux ou les bouchons associés au récipient.

4 Principes généraux pour l'échantillonnage des liquides isolants

4.1 Nouveaux liquides isolants dans les récipients de livraison

4.1.1 Lieu d'échantillonnage

L'échantillon doit être prélevé dans la partie du récipient de livraison dans laquelle le liquide isolant est susceptible d'être le plus contaminé. Pour évaluer la qualité d'une livraison, deux types d'échantillons peuvent généralement être prélevés:

a) échantillon composite: mélange d'échantillons prélevés à un même niveau dans plusieurs récipients;
b) échantillon individuel: échantillon ou mélange d'échantillons prélevés à un même niveau dans un même récipient.

Pour une livraison, des échantillons individuels de 1 l peuvent être prélevés dans différents récipients pour l'essai de rigidité électrique. D'autres essais peuvent être effectués sur ces échantillons et un examen complet de leur mélange peut être réalisé (échantillon composite).

Dans certains cas, il peut s'avérer utile de constituer un échantillon moyen dans le récipient. Un échantillon moyen est un mélange d'échantillons prélevés à différents niveaux dans un même récipient.

1) citernes: il convient de prélever les échantillons dans chaque citerne comme cela est décrit en 4.1.4.2 ci-dessous;
2) fûts: il convient de prélever les échantillons comme cela est décrit en 4.1.4.3 ci-dessous.

Dans le cas d'un seul fût, un échantillon doit être prélevé.

En présence de plusieurs fûts d'un lot d'huile, il convient que le fournisseur et l'utilisateur conviennent de la procédure d'échantillonnage. Par exemple, les échantillons peuvent être prélevés de 10 % des fûts ou d'au moins deux fûts, selon celui qui est le plus important.

4.1.2 Quantité d'échantillons à prélever

Cela dépend des essais à réaliser et des procédures utilisées.

En règle générale, 2 l sont prélevés.

4.1.3 Matériel de prélèvement

4.1.3.1 Généralités

Comme les résultats des essais mentionnés dans les exigences de l'IEC pour les liquides isolants peuvent, en grande partie, dépendre des impuretés présentes dans l'échantillon, il est essentiel d'observer les précautions suivantes:

– un matériel de prélèvement séparé doit être réservé exclusivement pour chaque type d'huile. Il convient que tous les joints et tuyaux utilisés soient compatibles avec le liquide isolant échantillonné;

– le matériel doit être propre et sec, conformément aux procédures de nettoyage décrites en 4.2.1.6. Il convient de veiller avec un soin particulier à ce qu'il n'y ait pas traces d'impuretés solides, comme la poussière ou les fibres. L'utilisation de chiffons pour le nettoyage n'est pas permise.

4.1.3.2 Sondes d'échantillonnage

A titre d'exemple, quatre types de sondes d'échantillonnage sont décrits ci-dessous. D'autres sondes peuvent également être utilisées, à condition qu'elles n'introduisent aucune contamination. Le matériel en acier inoxydable et en aluminium convient.

a) Echantillonnage dans des citernes

La sonde représentée à la Figure 1 est adaptée pour prélever des échantillons au fond de la citerne. Il s'agit d'une sonde constituée de tubes et de moulages en acier inoxydable ou en aluminium entièrement usinés. Elle doit être suffisamment lourde pour plonger dans le liquide. Il convient qu'elle soit toujours suspendue au moyen d'un fil métallique ou d'une chaîne. Ni corde ni autre matériau fibreux ne doivent être utilisés.

L'écrémoir est utilisé pour prélever des échantillons en surface de liquides isolants. Cette sonde doit être construite suivant le schéma représenté à la Figure 2 et doit être en acier inoxydable.

Dimensions en millimètres

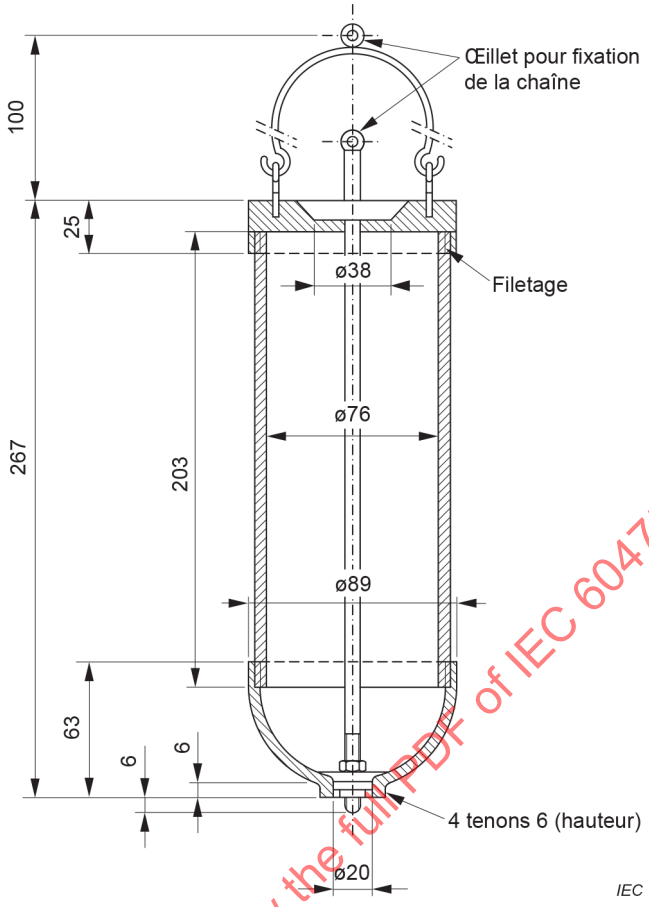

Figure 1 – Sonde

Dimensions en millimètres

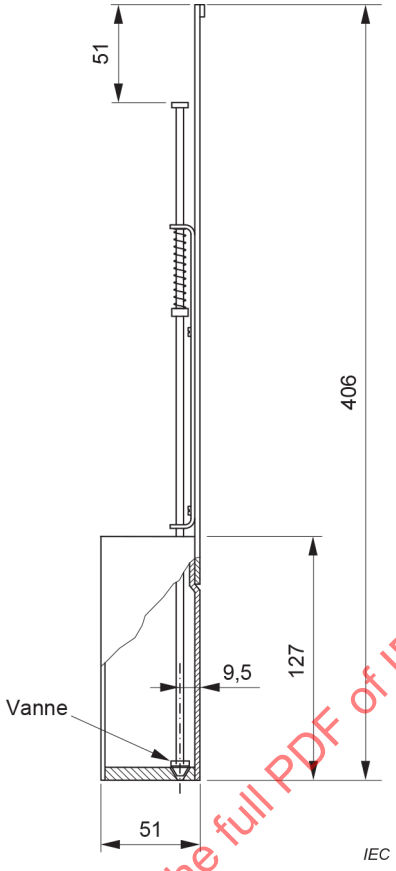

Figure 2 – Ecrémoir

b) Echantillonnage dans des fûts

La pipette représentée à la Figure 3 est prévue pour prélever des échantillons au fond des fûts. Cette pipette a une capacité d'environ 500 ml.

Une autre sonde pour prélever des échantillons au fond est représentée à la Figure 4: il s'agit d'un siphon qui comprend un tube en verre, en acier inoxydable ou en aluminium d'un diamètre intérieur d'environ 13 mm pour le prélèvement du liquide, et un tube métallique (diamètre intérieur de 5 mm) pour l'application de la pression. Les deux tubes sont sertis dans un bouchon résistant à l'huile dont les dimensions correspondent au diamètre de la bonde du fût. Il existe des versions commerciales de ces matériels. Dans la mesure du possible, il est préférable de choisir le verre pour les sondes représentées à la Figure 3 et à la Figure 4.

L'écrémoir (Figure 2) peut être utilisé pour prélever des échantillons en surface.

Dimensions en millimètres

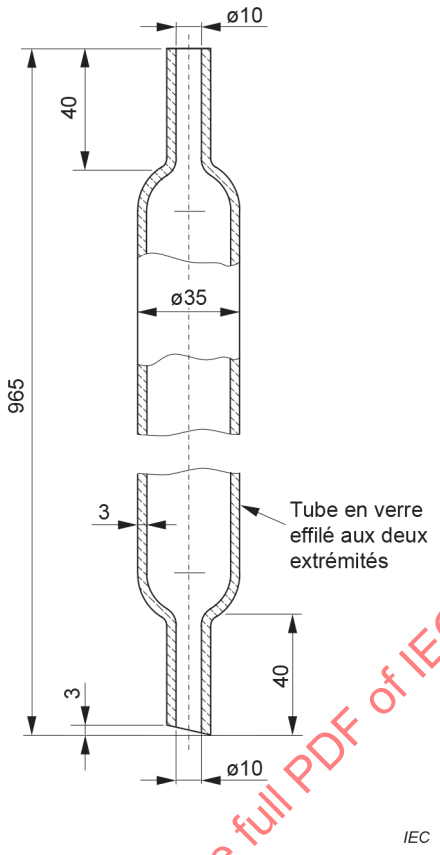

Figure 3 – Pipette

Dimensions en millimètres

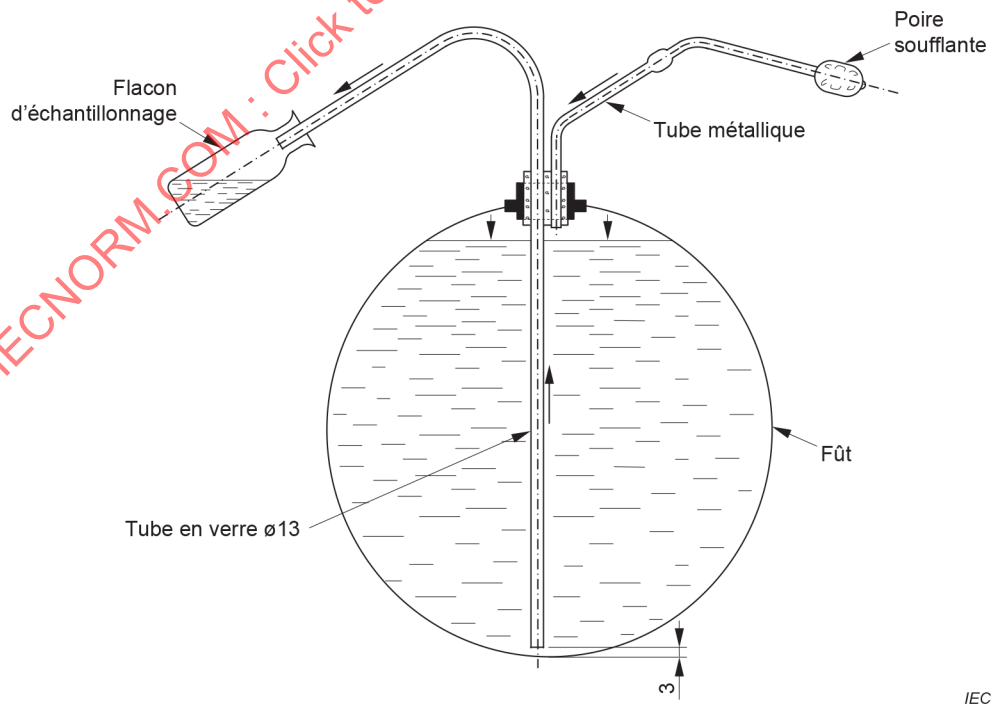

Figure 4 – Siphon

4.1.3.3 Récipients d'échantillonnage

Pour le stockage et le transport des échantillons, selon l'essai à réaliser sur l'huile, des récipients d'échantillonnage de volume approprié doivent être utilisés. Différents types de récipients d'échantillonnage sont présentés en 4.2.1.5.

Pour le mélange de différents échantillons, un récipient d'échantillonnage spécial en verre d'une capacité d'au moins 6 l doit être utilisé. Ces récipients d'échantillonnage spéciaux doivent être fermés de manière à pouvoir les sceller, au moyen d'un tuyau en plastique ou en caoutchouc compatible résistant à l'huile ou de bouchons à vis en polytétrafluoroéthylène (PTFE). Les tuyaux et/ou joints en caoutchouc naturel ne sont pas permis. Les joints en PTFE et polypropylène (PP) sont acceptables.

Chaque récipient d'échantillonnage doit porter une étiquette sur laquelle sont mentionnées toutes les indications nécessaires pour identifier le contenu, notamment le marquage des fûts ou citernes, la date de prélèvement et le nom du réceptionnaire.

4.1.3.4 Nettoyage du matériel de prélèvement

Le matériel de prélèvement doit être nettoyé conformément aux procédures décrites en 4.2.1.6.

4.1.4 Procédure de prélèvement

4.1.4.1 Remarques générales

Conformément aux principes généraux d'échantillonnage (voir 4.1.1), les échantillons de liquide isolant neuf doivent être prélevés au fond du récipient de livraison, où la contamination est vraisemblablement la plus importante. Cependant, dans certains cas, il est intéressant de prélever un échantillon moyen.

NOTE 1 Pour constituer un échantillon moyen, des échantillons sont prélevés à des niveaux intermédiaires dans les citernes et les fûts. Des exemples de procédures sont donnés à l'Annexe A. Une procédure est donnée dans la NOTE en 4.1.4.2 a) pour obtenir l'équivalent d'un échantillon moyen.

Différents cas sont examinés dans le Tableau 1:

Tableau 1 – Types d'échantillon de liquides isolants neufs

Type de livraison	Echantillonnage recommandé	Matériel de prélèvement à utiliser	Procédure	Quantité recommandée
Fûts	Echantillon composite	Pipette (Figure 3) ou Siphon (Figure 4)	4.1.4.2 4.1.4.2	3 × 2 l 1 l ou 2 l
	Echantillon individuel			
Citernes	Echantillon individuel	Sonde (Figure 1) ou Néant (vanne)	4.1.4.1 b) 4.1.4.1 a)	3 × 2 l
Fûts	Echantillon moyen	Pipette ou Siphon (Figure 3 et Figure 4)	Annexe A	3 × 2 l
Citernes	Echantillon moyen	Sonde (Figure 1) ou Néant (vanne)	4.1.4.1 a)	

NOTE 2 Avant d'échantillonner des citernes, une quantité suffisante d'huile est pompée à travers la tuyauterie de vidange, comme cela est indiqué en 4.1.4.2.

Toutes les précautions doivent être prises durant l'échantillonnage afin d'éviter toute contamination des liquides isolants. L'échantillonnage à l'extérieur sous pluie, brouillard ou vent fort est toléré seulement si toutes les précautions ont été prises pour éviter la contamination du liquide. Dans ces cas spéciaux, l'utilisation d'une bâche est nécessaire.

La condensation doit être évitée en chauffant le matériel de prélèvement de façon qu'il soit à une température supérieure à la température ambiante. Avant utilisation, le matériel doit être rincé avec le liquide à échantillonner. L'opérateur doit veiller à ne pas toucher avec les mains les surfaces du matériel de prélèvement en contact avec l'huile. Les liquides isolants doivent être protégés de la lumière durant le transport et le stockage.

A l'arrivée au laboratoire, le récipient d'échantillonnage ne doit pas être ouvert immédiatement. Il est nécessaire d'attendre que la température de l'échantillon soit la même que la température ambiante.

4.1.4.2 Échantillonnage dans des citernes

Les liquides isolants peuvent être prélevés soit par la vanne de vidange de la citerne, soit au moyen d'une sonde ou d'un écrémoir.

a) Echantillonnage par la vanne de vidange

Par cette procédure, il est possible d'obtenir un échantillon représentatif du fond de la citerne après immobilisation de la citerne pendant au moins 1 h après l'arrivée du véhicule.

NOTE Il est également possible, par cette procédure, d'obtenir l'équivalent d'un échantillon moyen, si le prélèvement est fait directement après l'arrivée du véhicule.

Dans ce cas, la procédure de prélèvement doit être la suivante:

- enlever la protection de la vanne de vidange, le cas échéant;
- éliminer toutes les saletés et poussières de la vanne à l'aide d'un chiffon non pelucheux et propre ou d'éponges synthétiques résistantes à l'huile;
- le dispositif de vidange (pompe et tuyauterie), s'il est incorporé, doit être soit mis en fonctionnement, soit ouvert afin de pouvoir prélever l'échantillon;
- ouvrir la vanne et laisser écouler, lentement, au moins 10 l de liquide isolant dans un récipient d'huile usagée. Dans tous les cas, éliminer au moins une quantité d'huile équivalente au volume du tuyau;
- rincer les flacons d'échantillonnage avec le liquide isolant;
- remplir les flacons d'échantillonnage à débit constant afin d'éviter les turbulences.

b) Echantillonnage à l'aide de la sonde ou de l'écrémoir

Il convient de procéder à ce type d'échantillonnage après immobilisation de la citerne pendant au moins 1 h après l'arrivée du véhicule.

1) Procédure avec la sonde (Figure 1) (échantillons prélevés au fond du récipient)

Pour prélever des échantillons dans le fond (c'est-à-dire à 1 cm ou 2 cm du fond de la citerne), la sonde est immergée jusqu'à ce que la partie en saillie de l'axe de la vanne atteigne le fond de la citerne. La sonde se remplit. L'opération est terminée lorsque les bulles d'air ne s'échappent plus. La sonde est ensuite retirée et son contenu versé dans le récipient d'échantillonnage (cas d'un échantillon individuel) ou dans le récipient d'échantillonnage en verre spécial prévu pour la collecte et le mélange des différents échantillons prélevés (cas d'un échantillon composite). Dans ce dernier cas, le ou les récipients d'échantillonnage sont remplis avec le mélange ainsi obtenu. Lors de l'opération, éviter de former des bulles par un déversement trop rapide.

2) Procédure avec l'écrémoir (Figure 2) (échantillons prélevés en surface)

La vanne étant fermée, remplir l'écrémoir en l'immergeant doucement dans le liquide à échantillonner jusqu'à ce que le bord supérieur soit juste en dessous de la surface du liquide, de sorte que le liquide remplisse lentement la sonde.

Eliminer le premier remplissage. Remplir de nouveau la sonde comme cela est décrit ci-dessus et transvaser l'échantillon dans le récipient d'échantillonnage en le laissant s'écouler, par l'orifice du bas, contre la paroi du récipient et non par jet direct dans le fond du récipient. Répéter cette opération de manière à obtenir suffisamment de liquide pour remplir le récipient d'échantillonnage (échantillon individuel) ou le récipient d'échantillonnage en verre spécial utilisé pour mélanger les échantillons, suivant le type d'échantillon souhaité.

4.1.4.3 Échantillonnage dans des fûts

Il convient de prélever les échantillons après que les fûts aient été stockés à l'abri de l'eau et de la pluie, bonde au-dessus, durant au moins 8 h. Pour prélever les échantillons dans le fond des fûts (c'est-à-dire à 3 mm), la pipette (Figure 3) ou le dispositif avec siphon (Figure 4) peuvent être utilisés.

Pour prélever un échantillon sur la couche de surface du liquide, l'écrémoir (Figure 2) peut être utilisé.

Exemples de procédures:

a) Utilisation de la pipette (Figure 3) (échantillons prélevés au fond du récipient)
   - fermer l'orifice supérieur de la pipette avec le pouce et ensuite immerger la pipette dans le liquide jusqu'au fond du fût;
   - enlever le pouce pour permettre au liquide de remplir la pipette;
   - boucher à nouveau la pipette avec le pouce et enlever la pipette; le premier remplissage permet de rincer la pipette;
   - transférer les remplissages suivants, soit dans un récipient d'échantillonnage (échantillon individuel), soit dans le récipient d'échantillonnage en verre spécial destiné au mélange des échantillons (échantillon composite) (voir 4.1.4.2 b)) en prenant soin de ne pas former de bulles d'air lors de toute opération de transvasement.

b) Utilisation du siphon (Figure 4) (échantillons prélevés au fond du récipient)
   - fixer, dans la bonde du fût, le bouchon dans lequel sont insérés les tubes de prélèvement et d'amenée de pression et assurer que le système est hermétique;
   - plonger l'extrémité inférieure du tube de prélèvement jusqu'à environ 3 mm du fond du fût;
   - mettre le fût sous pression par la poire soufflante;
   - laisser s'écouler suffisamment de liquide pour rincer le tube, et ensuite verser la quantité exigée directement dans le récipient d'échantillonnage (échantillon individuel) ou le récipient d'échantillonnage en verre spécial destiné au mélange des échantillons (échantillon composite) (voir 4.1.4.2 b)) en prenant soin de ne pas entraîner de bulles d'air lors de toute opération de transvasement.

c) Utilisation de l'écrémoir (Figure 2) (échantillons prélevés en surface)

Voir 4.1.4.2 b).

4.1.4.4 Rapport d'échantillonnage

Le rapport d'échantillonnage doit donner toutes les informations nécessaires pour l'identification de l'échantillon, ainsi que les informations détaillées ou spécifiques qui sont susceptibles d'aider ceux qui sont chargés d'exécuter les essais. Le type d'échantillon (composite, individuel ou moyen) doit être indiqué. Une copie du rapport doit accompagner chaque échantillon. La distribution d'échantillons doit être faite conformément à la procédure établie, par exemple dans le contrat de vente.

4.2 Échantillonnage de l'huile dans les matériels remplis d'huile

4.2.1 Remarques générales

4.2.1.1 Sécurité et qualité de l'échantillonnage

Les instructions du fabricant en matière de prélèvement des échantillons d'huile dans les matériels électriques doivent être suivies. Une attention particulière doit être accordée aux précautions de sécurité à prendre.

Vérifier que l'huile présente dans le matériel électrique sous tension n'est pas en dépression au moment du prélèvement d'un échantillon d'huile, des bulles d'air pouvant s'introduire dans l'huile, provoquer des courts-circuits électriques dans le matériel et mettre en danger le personnel chargé de l'échantillonnage.

Au cours d'un prélèvement d'huile, il convient de prendre toutes les précautions pour faire face à un écoulement soudain d'huile et empêcher un déversement accidentel d'huile.

Il est important de garder à l'esprit qu'il est essentiel de recevoir un échantillon de qualité et représentatif pour effectuer une évaluation fiable du matériel électrique. Même les méthodes d'analyses et de diagnostic les plus sophistiquées ne peuvent pas rattraper un échantillonnage défectueux.

Dans tous les cas, il convient que l'échantillonnage de l'huile soit réalisé par un personnel formé de manière correcte et spécifique, particulièrement pour les matériels contenant un petit volume d'huile (les transformateurs de mesure, par exemple).

4.2.1.2 Lieu d'échantillonnage

Il convient de choisir avec soin les zones de prélèvement. Généralement, il convient de prélever l'échantillon en un point représentatif de la totalité de l'huile de l'appareil (la vanne de vidange d'huile ou la vanne d'échantillonnage de l'huile, par exemple). Cependant, il est parfois nécessaire de prélever délibérément des échantillons là où il est évident qu'ils ne sont pas représentatifs (lors d'une tentative de localisation d'un défaut du changeur de prises, de l'interrupteur sélecteur ou d'un relais à gaz, par exemple).

Toutes les méthodes décrites conviennent pour des matériels contenant un grand volume d'huile, comme les transformateurs de puissance. Pour des matériels à faible volume d'huile, il est essentiel d'assurer que le volume total de l'huile prélevée ne mette pas en danger le fonctionnement du matériel.

Pour les transformateurs équipés de deux vannes d'échantillonnage, il convient d'utiliser la procédure suivante: ouvrir d'abord la vanne de sortie, puis la deuxième vanne. Cela est particulièrement important afin d'éviter l'entrée d'air dans les transformateurs.

4.2.1.3 Nettoyage du point d'échantillonnage

Le nettoyage du matériel d'échantillonnage et le rinçage du point d'échantillonnage doivent être effectués afin d'empêcher toute contamination des échantillons d'huile.

Enlever la bride ou le couvercle (11) de la vanne d'échantillonnage à la Figure 5, la Figure 6 et à la Figure 7 a), nettoyer l'orifice de sortie avec un chiffon non pelucheux ou une éponge synthétique résistante à l'huile pour éliminer les saletés visibles.

La vanne de vidange est rincée avec une quantité d'huile suffisante (en général, de 2 l à 5 l), sous un écoulement turbulent, afin d'éliminer tous les contaminants (eau et particules) susceptibles de s'être accumulés dans la vanne de vidange et au niveau de son orifice.