

# INTERNATIONAL STANDARD



**Oil-filled electrical equipment – Sampling of free gases and analysis of free and dissolved gases in mineral oils and other insulating liquids – Guidance**

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## INTERNATIONAL ELECTROTECHNICAL COMMISSION

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**OIL-FILLED ELECTRICAL EQUIPMENT –  
SAMPLING OF FREE GASES AND ANALYSIS  
OF FREE AND DISSOLVED GASES IN MINERAL OILS  
AND OTHER INSULATING LIQUIDS – GUIDANCE**

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**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 60567 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications. It is an International Standard.

This fifth edition cancels and replaces the fourth edition published in 2011. This edition constitutes a technical revision. **1**

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

- a) a new normative Annex F relating to DGA analysis of insulating liquids other than mineral oils (esters and silicones) has been added;
- b) Clause 4 to Clause 11 and informative Annex A to Annex E remain devoted to mineral oils;
- c) two new mercury-free gas extraction methods are described in Annex B (low pressure vacuum extraction and mechanical oscillation).

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

Draft	Report on voting
10/1207/FDIS	10/1211/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](http://www.iec.ch/members_experts/refdocs). The main document types developed by IEC are described in greater detail at [www.iec.ch/publications](http://www.iec.ch/publications).

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- reconfirmed,
- withdrawn, or
- revised.

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## INTRODUCTION

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

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

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

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

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

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

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

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

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

### 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~~ can lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

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

## Environment

**WARNING** – This document is applicable to insulating oils, chemicals and used sample containers.

Attention is drawn to the fact that, at the time of writing of this document, many insulating oils in service are known to be contaminated to some degree by polychlorinated biphenyls (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. Disposal or decontamination of these oils ~~should be carried out strictly according to local regulations~~ can be subject to regulatory requirements. Every precaution should be taken to prevent the release of any type of insulating oil into the environment, including those partially biodegradable with time.

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# OIL-FILLED ELECTRICAL EQUIPMENT – SAMPLING OF FREE GASES AND ANALYSIS OF FREE AND DISSOLVED GASES IN MINERAL OILS AND OTHER INSULATING LIQUIDS – GUIDANCE

## 1 Scope

This document deals with the techniques for sampling free gases from gas-collecting relays from power transformers. Three methods of sampling free gases are described.

The techniques for sampling oil from oil-filled equipment such as power and instrument transformers, reactors, bushings, oil-filled cables and oil-filled tank-type capacitors are no longer covered by this document, but are instead described in IEC 60475:2014/2022, 4.2.

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

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

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

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

## 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 60296, *Fluids for electrotechnical applications – ~~Unused Mineral insulating oils for transformers and switchgear~~ Mineral insulating oils for electrical equipment*

IEC 60475:2014/2022, *Method of sampling insulating liquids*

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

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

ISO 5725-1, Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions

~~ASTM D2780, Standard Test Method for Solubility of Fixed Gases in Liquids~~

### 3 Terms, definitions, symbols and abbreviated terms

#### 3.1 Terms and definitions

No terms and definitions are listed in this document.

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>

#### 3.2 Symbols and abbreviated terms

##### 3.2.1 Symbols

The symbols used in 8.5.2 are:

$V$	total volume of the vial
$V_G$	volume of the gas phase in the vial
$V_L$	volume of the oil phase in the vial
$C_G$	concentration of gas (i) in the gas phase of vial, obtained by GC (gas chromatography)
$C_L^0$ *	concentration of gas (i) in the oil sample, obtained directly from $C_G$ using calibration curves with gas-in-oil standards
$P, t$	atmospheric pressure and temperature when the oil sample was analysed ( $P$ in kPa; $t$ in °C)
$P_s, t_s$	atmospheric pressure and temperature when the gas-in-oil standard, or the gas standard, was analysed ( $P_s$ in kPa; $t_s$ in °C)
$K$	partition coefficient of gas (i), for the calculation of $C_L^0$ using gas standards
$C_L^0$	concentration of gas (i) in the oil sample

##### 3.2.2 Abbreviated terms

DGA	dissolved gas analysis
FID	flame ionization detector
GC	gas chromatography
GILS	gas-in-liquid standards
GIOS	gas-in-oil standards
HID	helium ionization detector
ID	inner diameter
NIST	National Institute of Standards and Technology
OD	outer diameter
OLTC	on-load tap-changer

PLOT	porous large open tubular
PTFE	polytetrafluoroethylene
TCD	thermal conductivity detector

## 4 Sampling of gases from gas-collecting ~~(Buchholz)~~ relays

### 4.1 General remarks

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

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

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

Gas samples ~~should~~ shall be properly labelled (see Clause 5) and analysed without undue delay to minimize hydrogen loss from the syringe used for gas sampling (e.g. within a maximum period of one week).

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

Of the three methods described in 4.2, 4.3 and 4.4, the syringe method is recommended. The other two methods are alternatives to be used exclusively in case of serious hindrance.

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

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

### 4.2 Sampling of free gases by syringe

#### 4.2.1 Sampling equipment

NOTE Numbers in brackets refer to those circled numbers in the relevant figure.

See Figure 1. The equipment shall be as follows:

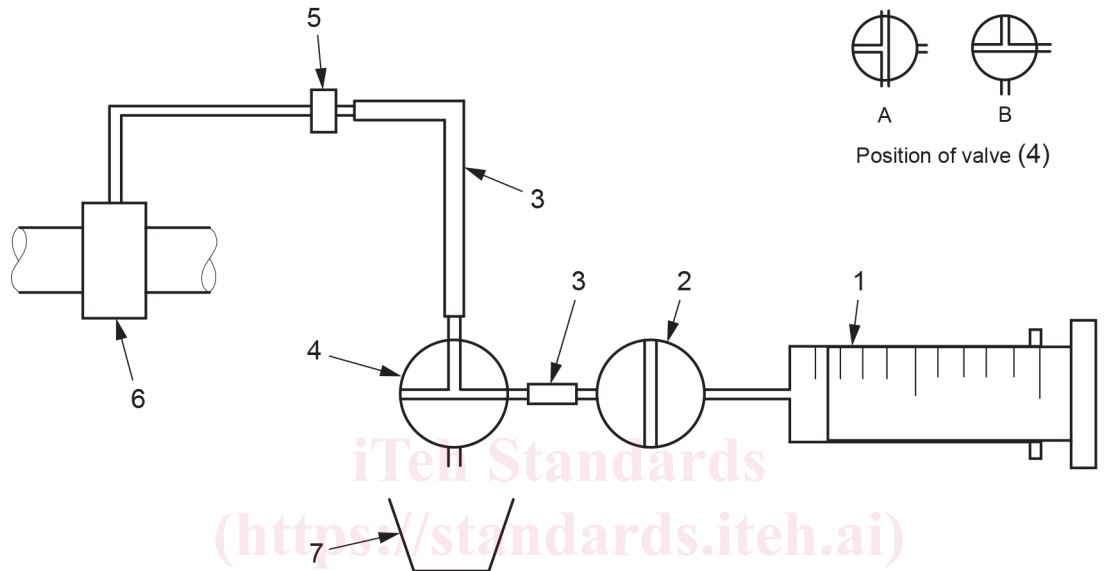
- a) Impermeable oil-resistant plastic or rubber tubing (3) provided with a connector to fit onto a suitable sampling connection of the gas-collecting relay. To avoid cross-contamination, the tubing should be used only once.
- b) Gas-tight syringes of suitable volume (1) (25 ml to 250 ml). Medical or veterinary quality glass syringes with ground-in plungers may be suitable; alternatively, syringes with oil-proof seals may be used. The syringe should be fitted with a cock enabling it to be sealed. It is often convenient to use the same syringes for both gas sampling and for oil sampling (see IEC 60475:2014/2022, 4.2.2).

The gas tightness of a glass syringe used for gas sampling may be tested by storing an oil sample containing a measurable quantity of hydrogen for at least two weeks and analysing

aliquots for hydrogen at the beginning and end of the period. An acceptable syringe will permit losses of hydrogen of less than 2,5 % per week. General experience suggests that all-glass syringes leak less than those using plastic seals. Improvement of the gas tightness may be obtained by the use of a lubricant such as a light grease or transformer oil.

It is a good practice to test the integrity of syringes and valve system before the sampling. A recommended procedure is given in IEC 60475:2011/2022, Annex B.

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



**Key**

- 1 syringe
- 2 stopcock
- 3 rubber connecting tubing
- 4 three-way valve
- 5 equipment sampling valve
- 6 gas-collecting relay valve
- 7 waste vessel

**Figure 1 – Sampling of gas by syringe**

**4.2.2 Sampling procedure**

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

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

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

Label carefully the sample (see Clause 5).