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

**Guide pour le prélèvement des gaz et de l'huile dans le matériel électrique
rempli d'huile et pour l'analyse des gaz libres et dissous**

**Anleitung zur Probenahme von Gasen und von Öl aus ölgefüllten elektrischen
Geräten und zur Analyse von freien und gelösten Gasen**

National foreword

This British Standard has been prepared under the direction of the General Electrotechnical Engineering Standards Committee. It is identical with IEC 567 'Guide for the sampling of gases and of oil from oil-filled electrical equipment and for the analysis of free and dissolved gases'.

NOTE 1. Wherever page numbers are quoted in connection with references to figures, they are IEC page numbers.

NOTE 2. Throughout this document, the pressure unit 'millibar' has been used. Its relationship with the SI unit of pressure (the pascal) is given by: $1 \text{ mbar} = 10^2 \text{ N/m}^2 = 0.1 \text{ kPa}$.

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GUIDE FOR THE SAMPLING OF GASES AND OF OIL FROM OIL-FILLED ELECTRICAL EQUIPMENT AND FOR THE ANALYSIS OF FREE AND DISSOLVED GASES

1. Introduction

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

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

In the case of a fault, its type and its severity may often be inferred from the composition of the gases and the rate at which they are formed. In the case of an incipient fault, the gases formed remain partly dissolved in the liquid insulation; free gases will be found only in special cases. The dissolved gases will divide between the gaseous and liquid phases by diffusion. Diffusion and achievement of saturation both take time, during which serious damage to the equipment can occur undetected.

Periodic analyses of oil samples for the amount and composition of dissolved gases form a means of detecting faults.

The growing importance of gas analysis of samples drawn from electrical equipment has led to the preparation of this guide to the procedures to be used for the sampling from oil-filled electrical equipment of gases and oils containing gases and for their subsequent analysis.

The provision of a guide for the evaluation of the results of gas analysis will be the aim of future work.

2. Scope

This guide deals with the methods for sampling and analysis of free gases and for sampling and analysis of gases dissolved in oil, from oil-filled equipment such as power and instrument transformers, and oil-filled cables.

This guide assumes a measure of competence in the technique of gas chromatography and omits, for brevity, many details which may be found in practical manuals on these techniques.

The methods described in the main part of this guide are suitable for all samples and take into account the problems imposed by the transport of samples by unpressurized air freight, and where considerable differences in ambient temperature exist between the plant and the examining laboratory.

Alternative methods of sampling may be used and are given in Appendices A and B. Appendices C and D describe alternative methods applicable for the preparation of oil samples for the analysis of their dissolved gases and for the analysis itself.

Where, by agreement between manufacturer and user, dissolved gas analysis is required during the testing or early life of the equipment, an agreed standard of reproducibility of results between different laboratories shall be obtained, and it will be necessary for both parties to agree upon the details of the method to be used.

Interpretation and judgement of the significance of the results will be improved if the same apparatus and techniques are used throughout an investigation. This is particularly important in assessing the rate of gas formation in equipment by analysis of samples drawn at successive times.

3. Sampling

3.1 *Sampling of gases from gas cushions and gas-collecting (Buchholz) relays*

3.1.1 *General remarks*

Gas samples from relays should be taken from the equipment with the minimum delay. Changes in composition caused by the selective reabsorption of components may occur if free gases are left in contact with oil.

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

Gas samples should be properly labelled (see Clause 4) and analysed without delay, preferably within a maximum period of two weeks.

Oxygen, if present in the gas, may react with any oil drawn out with the sample. Oxidation is delayed by excluding light from the sample, for example by wrapping the syringe in aluminium foil. The transport of samples is facilitated by the use of special containers which hold the samples firmly in place during transport.

The methods given in Appendix A may be used as alternatives to the method given below.

Changes in the composition of gases generated by a fault always occur during their movement to the gas collecting relay. Information on the duration of a fault may often be inferred by comparing the composition of free gases with those remaining dissolved in the oil and, since selective reabsorption of components can also occur in the oil remaining within the relay, undue delay in sampling may conceal valuable evidence.

3.1.2 *Sampling equipment*

- a) An impermeable oil-resistant plastic or rubber tube provided with a connector to fit onto a suitable sampling connection of the relay or gas cushion.
- b) A gas-tight syringe of suitable size (25 cm³ to 250 cm³). Medical or veterinary quality glass syringes with ground-in plungers may be suitable; alternatively, syringes with oil-proof seals may be used (see Item b) of Sub-clause 3.2.2).
- c) Transport containers. These should be designed to hold the syringe firmly in place during transport.

3.1.3 *Sampling procedure*

The apparatus is connected as shown in Figure 1, page 43. The connections should be as short as possible and filled with oil at the start of sampling.

Sampling valve ⑤ is opened. If a positive pressure exists, the three-way cock ④ is carefully opened and any oil present allowed to flow to waste. When gas reaches the three-way cock ④,

the latter is turned to disconnect the waste and connect the syringe. Cock ② is then opened and the syringe allowed to fill under the hydrostatic pressure, taking care that its plunger is not expelled. When a sufficient sample has been taken, cock ② is closed and the apparatus is disconnected.

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

In the absence of a positive pressure within the equipment, a supplementary air pump should be connected between the equipment sampling screw and cock ② and used to pump gas from the equipment.

The equipment sampling valve ⑤ must be closed at the end of sampling.

3.2 *Sampling oil from oil-filled equipment*

3.2.1 *General remarks*

The method of sampling with syringes (given below) is suitable irrespective of the mode of transportation of samples. Other methods, given in Appendix B, may be used if there are no significant changes of temperature and pressure during transportation. For routine sampling on a large scale, the use of bottles or sampling tubes, rather than syringes, may be the most economical method. The methods described are suitable for large oil-volume equipment such as power transformers. With small oil-volume equipment, it is essential to ensure that the total volume of oil drawn off does not endanger the operation of the equipment.

The selection of points from which samples are drawn should be made with care. Normally, the sample should be taken from a point where it is representative of the body of the oil in the equipment and where changes in composition such as those due to pump cavitation do not exist. It will sometimes be necessary, however, deliberately to draw samples where they are not expected to be representative, for example, in trying to locate the site of a fault.

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

Some of the dissolved oxygen present in the oil sample may be consumed by oxidation. The reaction can be delayed by exclusion of light, e.g. by wrapping the sampling vessel in aluminium foil.

Samples should be carefully labelled (see Clause 4).

3.2.2 *Sampling equipment*

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

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

- b) *Gas-tight syringe*

This may be all glass or have plastic or oil-proof rubber seals and a glass or plastic plunger. The size may be 25 cm³ to 250 cm³, largely depending on the sensitivity of the

analytical procedure and the volume of oil of the system to be sampled. The syringe should be fitted with a cock enabling it to be sealed.

The gas tightness of a pattern of syringe may be tested by storing an oil sample in a syringe for two weeks and analysing aliquots for hydrogen at the beginning and at the end of this period. An acceptable syringe will permit losses of hydrogen of less than 2.5% each week.

c) Transport containers

These should be designed to hold the syringe firmly in place during transport but allow the syringe plunger to be free.

3.2.3 Sampling procedure

- 1) The blank flange or cover of the sampling valve is removed and the outlet cleaned with a lint-free cloth to remove all visible dirt. The apparatus is then connected as shown in Figure 2, page 44, and the main sampling valve opened.
- 2) The three-way cock is adjusted to allow 1 litre to 2 litres of oil to flow to waste (see note).
- 3) The three-way cock is then turned to allow oil to enter the syringe slowly. The plunger should not be withdrawn but allowed to move back under the pressure of the oil.
- 4) The three-way cock is then turned to allow the oil in the syringe to flow to waste and the plunger pushed to empty the syringe. Confirm that the inner surfaces of the syringe and plunger are completely oiled.
- 5) The procedure described in Item 3) of this sub-clause is then repeated.
- 6) The cock on the syringe is then closed and the sampling valve closed.
- 7) The syringe is then disconnected.

The sample should be carefully labelled (see Clause 4).

Note. — In the case of small oil-volume equipment, the procedure described in Item 2) of this sub-clause should be omitted and a small-volume syringe used.

4. Labelling of samples

Oil and gas samples should be properly labelled before dispatch to the laboratory and the following information is desirable:

- Customer or plant.
- Identification of equipment.
- Object of sampling.
- Date of sampling.
- Load at time of sampling.
- Point where sample is taken.
- Type of oil (if known).
- Volume of oil in equipment.
- Temperature of sample when drawn (in the case of oil).
- Other pertinent data: e.g. type of tap changer if fitted, operation of pumps, oil preservation system, etc.

5. Preparation of samples for analysis

5.1 Gas samples

Gas samples require no special preparation and must be analysed in the condition in which they are received at the laboratory.

5.2 Oil samples

5.2.1 General remarks

These samples require preliminary treatment to remove the dissolved gases for analysis. In the method described in Sub-clause 5.2.3, the gases are extracted from the oil by vacuum. The procedure described in Sub-clause 5.2.3 can deal with any volume of oil and is suitable for both low and high gas contents. With a suitable apparatus, the volume of gases extracted can be measured. The extraction of gases will, however, be incomplete, particularly that of the more soluble components, but for the gases listed in Sub-clause 6.1, it will normally be possible to extract 97% to 99% of the total quantities present.

Oil samples may, on contraction, release bubbles of gas which change the composition of the gases which remain dissolved, and this may cause an error in the analysis. The magnitude of the error will depend on the size of bubble in relation to the volume of the oil sample and on the solubility of the gases.

The bubble may sometimes be redissolved by carefully warming the sample and holding it for some time at the temperature at which it was taken. Alternatively, the whole of the sample, including the bubble, can be introduced into the laboratory gas extraction apparatus.

Some other methods are available which are specially suitable for routine use. These are described in Appendix C.

5.2.2 Extraction apparatus

A suitable design of apparatus is shown in Figure 3, page 45.

The requirements of any apparatus used are as follows:

- a) It should be capable of subjecting the oil to a vacuum of below 0.1 mbar.
- b) It should be vacuum-tight. When subjected, when empty, to a vacuum of 0.1 mbar and left for 3 h, the vacuum should not have changed.
- c) It should be tested for efficiency, and the number of operations needed to extract at least 97% of the dissolved gases should be established by a preliminary test.

This may be done by extracting two identical samples, one for n strokes and another for $2n$ strokes, and establishing a value of n such that no significant difference between the two analyses can be found.

- d) It should permit the measurement of the extracted gas to be made to the nearest 0.05 cm^3 , or corresponding pressure and to ensure that the gas extracted from different strokes is well mixed before analysis.

Note. — Attention is drawn to the need for adequate precautions in the use of mercury to avoid danger to the health of operators.

5.2.3 Extraction procedure (Figure 3)

- 1) Weigh the syringe ① and connect it to the degassing flask ⑩.
- 2) Evacuate the apparatus to 0.1 mbar using either the Toepler pump ⑫ or an auxiliary pump.
- 3) Open the syringe cock and admit oil into the degassing flask and switch on the magnetic stirrer.

Degassing is improved by increasing the surface of the oil such as by allowing the oil to enter through a sintered filter or by impinging the jet of oil against the wall of the flask. The syringe stopcock should be closed while some oil still remains in the syringe, thus avoiding subjecting the syringe to vacuum.

- 4) Operate the Toepler pump and transfer the gas into the burette ⑬. Degassing will be complete when no increase in the volume of extracted gas is observed (see Item *c*) of Sub-clause 5.2.2).
- 5) Reweigh the syringe to obtain the mass of oil degassed. Measure the volume of extracted gas and record pressure and temperature.
- 6) Calculate the gas content of the oil sample in microlitres per litre at 20 °C and 1013 mbar by the equation:

$$\frac{P}{1013} \times \frac{293}{273+t} \times \frac{Vd}{m} \times 10^6$$

where:

P = pressure of the extracted gas, in millibars

t = temperature of the extracted gas, in degrees Celsius

V = volume of the extracted gas, in cubic centimetres, at pressure P

d = density of the oil, in grams per cubic centimetre, at 20 °C

m = mass of the degassed oil, in grams

6. Gas analysis by gas-solid chromatography

6.1 General remarks

Gas samples, whether obtained from gas cushions or gas collection relays or removed from an oil sample, are analysed by chromatography. The gases to be determined are:

- hydrogen: H_2
- oxygen: O_2
- nitrogen: N_2
- methane: CH_4
- ethane: C_2H_6
- ethylene: C_2H_4
- acetylene: C_2H_2
- carbon monoxide: CO
- carbon dioxide: CO_2

The determination of C_3 hydrocarbons and argon may be useful.

A number of methods may be used for the analysis. The method described in Clause D1 is an example of one which is acceptable.

To obtain the sensitivity required in Clause 7, both a flame ionization detector for hydrocarbons and a thermal conductivity unit for atmospheric gases CO, CO₂ and H₂ will be needed.

A single stationary phase capable of resolving all the gases under isothermal conditions is not available.

Single column operation is possible but only with temperature programming and sub-ambient starting temperature. Isothermal operation requires two columns and these may be operated in parallel or switched.

The carrier gas may be helium or argon. Helium gives improved sensitivity for carbon monoxide with loss of sensitivity for hydrogen, while for argon improved sensitivity for hydrogen is gained at the expense of loss of sensitivity for carbon monoxide.

A wide range of compositions can be expected and, to avoid constant alterations to the attenuator settings, it will be convenient to use an integrator.

6.2 *Apparatus*

6.2.1 *Gas chromatography*

Any instrument, preferably fitted with both flame ionization and thermal conductivity detectors, can be used. Gas samples shall be injected preferably by means of a properly designed injection valve capable of injection of 0.5 cm³ to 5 cm³ gas volume.

A precision gas-tight syringe may be used instead of the injection valve.

6.2.2 *Columns*

Several columns have been tried and found to be suitable. Some suitable arrangements are described in Appendix D, but many other arrangements are possible.

6.2.3 *Carrier gas*

Helium or argon of grades suitable for gas chromatography are normally used.

6.3 *Calibration*

The chromatograph is calibrated by the injection of known amounts of pure gases to establish the calibration curve and the retention time. For daily calibration checks, it is convenient to use a standard gas mixture containing a suitable known amount of each of the gas components to be analysed and diluted by nitrogen. Calibration may be in terms of molarity, partial pressure or volume.

6.4 *Procedure for gas analysis*

6.4.1 *General remark*

Any convenient method may be used provided that the sensitivity requirements are met. One example is given in detail in Clause D1 and others in outline in Clause D2.