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INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

**Insulating liquids – Test methods for oxidation stability
Test method for evaluating the oxidation stability of insulating liquids in the delivered state**

**Isolants liquides – Méthodes d'essai de la stabilité à l'oxydation
Méthode d'essai pour évaluer la stabilité à l'oxydation des isolants liquides tels que livrés**

IEC 61125:2018-01(en-fr)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

COMMISSION ELECTROTECHNIQUE INTERNATIONALE

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INSULATING LIQUIDS – TEST METHODS FOR OXIDATION STABILITY

Test method for evaluating the oxidation stability of insulating liquids in the delivered state

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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International Standard IEC 61125 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications.

This second edition cancels and replaces the first edition published in 1992 and Amendment 1:2004. This edition constitutes a technical revision.

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

a) the title has been modified to include insulating liquids different from mineral insulating oils (hydrocarbon);
b) the method applies for insulating liquids in the delivered state;
c) former Method C is now the main normative method;
d) precision data of the main normative method has been updated concerning the dissipation factor;

e) former Method A has been deleted;

f) former Method B has been transferred to Annex B;

g) a new method evaluating the thermo-oxidative behaviour of esters is included in Annex C.

The text of this standard is based on the following documents:

FDIS	Report on voting
10/1047/FDIS	10/1052/RVD

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

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

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- withdrawn,
- replaced by a revised edition, or
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INSULATING LIQUIDS – TEST METHODS FOR OXIDATION STABILITY

Test method for evaluating the oxidation stability of insulating liquids in the delivered state

1 Scope

This document describes a test method for evaluating the oxidation stability of insulating liquids in the delivered state under accelerated conditions regardless of whether or not antioxidant additives are present. The duration of the test can be different depending on the insulating liquid type and is defined in the corresponding standards (e.g. in IEC 60296, IEC 61099, IEC 62770). The method can be used for measuring the induction period, the test being continued until the volatile acidity significantly exceeds 0,10 mg KOH/g in the case of mineral oils. This value can be significantly higher in the case of ester liquids.

The insulating liquid sample is maintained at 120 °C in the presence of a solid copper catalyst whilst bubbling air at a constant flow. The degree of oxidation stability is estimated by measurement of volatile acidity, soluble acidity, sludge, dielectric dissipation factor, or from the time to develop a given amount of volatile acidity (induction period with air).

In informative Annex B, a test method for evaluating the oxidation stability of inhibited mineral insulating oils in the delivered state by measurement of the induction period with oxygen is described. The method is only intended for quality control purposes. The results do not necessarily provide information on the performance in service. The oil sample is maintained at 120 °C in the presence of a solid copper catalyst whilst bubbling through a constant flow of oxygen. The degree of oxidation stability is estimated by the time taken by the oil to develop a determined amount of volatile acidity (induction period with oxygen). Additional criteria such as soluble and volatile acidities, sludge and dielectric dissipation factor can also be determined after a specified duration.

In informative Annex C, a test method intended to simulate the thermo-oxidative behaviour of ester insulating liquids (headspace of air at 150 °C for 164 h) is described.

Additional test methods such as those described in IEC TR 62036 based on differential scanning calorimetry can also be used as screening tests, but are out of the scope of this document.

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 60247, *Insulating liquids – Measurement of relative permittivity, dielectric dissipation factor (tan δ) and d.c. resistivity*

IEC 62021-2, *Insulating liquids – Determination of acidity – Part 2: Colorimetric titration*

IEC 62021-3, *Insulating liquids – Determination of acidity – Part 3: Test methods for non-mineral insulating oils*

IEC 60422:2013, *Mineral insulating oils in electrical equipment – Supervision and maintenance guidance*

ISO 383, *Laboratory glassware – Interchangeable conical ground joints*

ISO 4793, *Laboratory sintered (fritted) filters – Porosity grading, classification and designation*

ISO 6344-1, *Coated abrasives – Grain size analysis – Part 1: Grain size distribution test*

ISO 3104, *Petroleum products – Transparent and opaque liquids – Determination of kinematic viscosity and calculation of dynamic viscosity*

ASTM E287, *Standard specification for laboratory glass graduated burets*

3 Terms and definitions

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

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

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

3.1
unused insulating liquid
insulating liquid that has not been used in, or been in contact with electrical equipment or other equipment not required for manufacture, storage or transport

Note 1 to entry: See also IEC 60296, IEC 61099 and IEC 62770.

3.2
recycled insulating liquid
insulating liquid previously used in electrical equipment that has been subjected to re-refining or reclaiming (regeneration) off-site

Note 1 to entry: Any blend of unused and recycled oils is to be considered as recycled.

3.3
oxidation stability
ability of an insulating liquid to withstand oxidation under thermal stress and in the presence of oxygen and a copper catalyst

Note 1 to entry: Oxidation stability gives general information about the stability of the insulating liquid under service conditions in electrical equipment. The property is defined as resistance to formation of acidic compounds, sludge and compounds influencing the dielectric dissipation factor (DDF) under given conditions. Test durations for insulating liquids are described in the corresponding standards.

3.4
induction period with air
graphical representation of the oxidation rate over the entire period which can be obtained by titrating volatile acidity daily (or at other suitable time interval) and plotting the cumulated results against time

Note 1 to entry: The induction period with air is determined by reading the time corresponding to 0,10 mg KOH/g volatile acidity in the case of mineral oil. In the case of ester liquids a higher value needs to be established.

3.5
volatile acidity
measurement of the amount of oxidation products collected in the water phase in the absorption tube

3.6
soluble acidity
acidity (neutralization value) of oil as a measure of the acidic degradation products in the insulating liquid

Note 1 to entry: The acidity of an oxidized oil is due to the formation of acidic oxidation products. Acids and other oxidation products will, in conjunction with water and solid contaminants, affect the dielectric and other properties of the oil. Acids have an impact on the degradation of cellulosic materials and may also be responsible for the corrosion of metal parts in a transformer.

3.7
total acidity
sum of volatile and soluble acidity

3.8
sludge
polymerized degradation product of solid and liquid insulating material

Note 1 to entry: Sludge is soluble in oil up to a certain limit, depending on the oil solubility characteristics and temperature.

3.9
dielectric dissipation factor
DDF
measure for dielectric losses within the oil

Note 1 to entry: High DDF values can indicate contamination of the oil by polar contaminants or poor refining quality.

Note 2 to entry: DDF shall be measured at 90 °C, and in accordance with IEC 60247.

4 Apparatus

4.1 General principle of the method

The liquid sample to be tested, through which a stream of air is bubbled, is maintained for a given period at 120 °C in the presence of solid copper. The resistance to oxidation is evaluated from the amount of total sludge, total acidity and dielectric dissipation factor formed or from the time to develop a given amount of volatile acidity (induction period with air).

4.2 Equipment

4.2.1 Heating arrangement

In order to achieve accurate measurements of the oxidation stability a strict control of the temperature is of high importance. A thermostatically-controlled aluminium alloy block heater or oil bath may be used to maintain the insulating liquid in the desired number of oxidation tubes at the required temperature of 120 °C ± 0,5 °C (as examples see Figure 1 and Figure 3). This temperature shall be read on a thermometer (see Annex A) inserted in an oxidation tube to within 5 mm from the bottom; this oxidation tube shall be filled with the insulating liquid up to the immersion line of the thermometer and placed in the heating bath.

The temperature of the upper surface of the thermal insulation top shall be maintained at 60 °C ± 5 °C. Measure this temperature by the use of a thermometer in a drilled aluminium block (see Figure 2). The surfaces of this block, other than that against the upper surface of the heating device, are protected by suitable thermal insulation of nominal 4 mm thickness.

The thermal characteristics of this insulation shall be such as to allow the specified temperatures to be achieved. This block should be placed as near to the holes as practicable and within the area of the upper surface covering the heating device.

When using an aluminum heating block, the oxidation tubes are inserted into the holes to an overall depth of 150 mm. The depth of the holes in the heating part of the block shall be at least 125 mm and short aluminum alloy collars, passing through the insulating cover and surrounding each oxidation tube, will ensure heating over the 150 mm length of the tube.

In the case of oil baths, the oxidation tubes shall be immersed to a depth of 137 mm in the oil and to an overall depth of 150 mm in the bath (see Figure 3).

For both types of heating devices, the height of the oxidation tubes above the upper surface shall be 60 mm and the diameter of the holes shall be just sufficient to allow insertion of the specified tubes. In the case of slackness a 25 mm internal diameter O-ring may be placed around the tube and pressed against the thermal insulation top or inserted into the annular space between the tube and the thermal insulated top. The heating bath should be equipped with supports to hold the absorption tubes.

When in use the heater shall be shielded from direct sunlight and air draughts.

NOTE When oil baths are used, it would be safer to place them in a fume hood.

4.2.2 Test vessels

Test tubes of borosilicate or neutral glass provided with a 24/29 ground joint (see ISO 383), of the following dimensions in mm:

– overall length 210 ± 2
– external diameter 26 ± 0,5
– wall thickness 1,4 ± 0,2
– height of the head 28 ± 2
– air inlet tube:
  - external diameter 5,0 ± 0,4
  - wall thickness 0,8 ± 0,1

The test tube is fitted with a Drechsel head to which is attached the inlet tube which extends to within 2,5 mm ± 0,5 mm from the bottom and has its end ground at an angle of 60° to the horizontal axis (see Figure 4).

4.2.3 Absorption tubes

These are identical to the test vessels and the distance between the axes of the two tubes shall be 150 mm ± 50 mm (see Figure 4 and Figure 5). Connections between the test and absorption tubes should be as short as possible, of glass tubing butt-jointed to the vessels by means of short flexible sleeves. Silicone rubber sleeving has been found suitable for this purpose; however, the exposed silicone rubber surface shall be minimized in order to avoid acids from being absorbed by the material. The absorption tubes are mounted outside the heating device.

4.2.4 Filtering crucibles

Gooch-type crucibles with fused-in fritted glass disk according to ISO 4793 porosity 4, designation grade P 16 of, for example, 35 ml capacity.

NOTE Alternatively polymeric membrane filters can be used, provided they are compatible with the insulating liquids and solvents. Suitable membranes consist of a mixture of cellulose esters (cellulose nitrate + cellulose acetate) with the following characteristics:

pore size: 8 µm;

thickness: 150 µm;

operating temperature: 120 °C in sterilizer and 75 °C under continuous filtration.

The filtration is improved by impregnating the membrane with a suitable wetting agent (e.g. octyl ethoxylate).

4.2.5 Porcelain vessels

Porcelain crucibles, capacity: 50 ml.

NOTE Alternatively, aluminium foil pans of the same capacity can be used.

4.2.6 Flowmeter

For measuring gas flow-rate a soap bubble flowmeter, a calibrated capillary tube flowmeter or an electronic device can be used.

4.2.7 Timer

For measuring gas flow-rate with soap bubble flowmeter. Subdivisions of the graduation: 0,2 s.

4.2.8 Gas supply

To obtain accurate results it is of high importance to control and maintain the gas flow constant and have a consistent high quality of the gas, this is obtained by the following procedure: gas (oxygen or air according to the method) from a compressed gas cylinder or line, is dried by passing through a scrubber bottle containing concentrated sulphuric acid and then through a tower filled with alternate layers of glass wool and soda lime.

Alternatively drying tubes or a commercial gas purifier may be used.

The dried gas is passed into the oxidation tube via a flow control system which shall be suitable for the specified flow-rate. This may consist of a manifold, connected to the gas-purifying train, with a number of tappings, each provided with a fine-control adjustable needle valve and supplying the gas to one oxidation tube.

The rate of gas flow may be conveniently measured by means of a flowmeter (see 4.2.6). In that case, the difference in level of the liquid in the two limbs of the flowmeter should be sufficiently great to ensure that adequate sensitivity of measurement is obtained over the range of gas flow-rates.

However, any system known to be of equal or greater efficiency can be used.

NOTE A two-stage pressure regulator and a pressure compensator vessel can be helpful to achieve the required accurate regulation of the gas pressure.

4.2.9 Analytical balance

Readability 0,1 mg.

4.2.10 Burette

Volume 10 ml with graduations of 0,01 ml, class A according to ASTM E287.

4.2.11 Volumetric pipette

Volume 25 ml, class A according to ASTM E287.

4.2.12 Volumetric flask

Volume 500 ml, class A according to ASTM E287.

4.2.13 Graduated measuring cylinder

Volume 100 ml, class A according to ASTM E287.

4.2.14 Thermometer

A thermometer conforming to the requirements given in Annex A.

4.2.15 Erlenmeyer flask

Erlenmeyer flask, volume 500 ml, with ground glass stopper.

4.3 Reagents

4.3.1 Normal heptane

n-Heptane of analytical grade is to be used.

4.3.2 Alkali blue 6B indicator according to IEC 62021-2

Alkali blue 6B indicator is also known under the chemistry index 42765.

4.3.3 Phenolphthalein indicator

1 g of phenolphthalein per 100 ml of azeotropic ethanol (about 5 % water). Alternatively isopropanol containing 5 % of water may be used.

NOTE Phenolphthalein fades rather quickly when exposed to strong direct light, should a faint tint be observed, it is suggested that a few more drops of indicator are added.

4.3.4 Potassium hydroxide according to IEC 62021-2

0,05 mol/l alcoholic solution.

4.3.5 Oxidant gas

Synthetic air or air from compressed air line, free of hydrocarbons.

4.3.6 Acetone

Acetone of analytical grade is to be used.

4.4 Cleaning of test vessels

The test and the absorption tubes shall be chemically cleaned. Wash with acetone followed by distilled or deionized water.

Drain and then soak in 95 % to 97 % sulphuric acid for a minimum of 16 h. Drain and complete removal of acid by washing, first with tap water, then with distilled or deionized water. Dry the tubes in an air oven at 105 °C for at least 3 h, and then allow cooling to room temperature in a desiccator or a dry cabinet in which they are kept ready for use. Other cleaning methods giving the same cleanliness result can be used.

4.5 Catalyst

The solid copper used as oxidation catalyst consists of a wire of soft electrolytic copper, of diameter between 1 mm and 2 mm (of such a length as to give a surface area of 28,6 cm^2 ± 0,3 cm^2). To get accurate results it is of high importance that the copper surface is properly prepared according to the following procedure:

– Immediately before use, the requisite length of copper wire is cleaned with P220 grade silicon carbide abrasive cloth (ISO 6344-1). All traces of abrasive are removed with a lintless filter paper and then with a dry, lintless cloth.

– Roll the wire into a spiral of approximately 2 cm external diameter and 5 cm long.

– The spiral is thoroughly cleaned by dipping it into normal heptane, then dried in air and immediately introduced into the test vessel.

To avoid contamination, the prepared coil shall be handled only with tweezers. The copper wire shall not be re-used.

4.6 Insulating liquid sample conditioning

Liquid to be tested shall be filtered through a previously dried (1 h at 105 °C) fritted glass filter (ISO 4793, porosity 4, designation grade P16) or on membrane filters of 8 µm to remove traces of sediment, fiber and excess water. The first 25 ml of filtrate should be discarded.

4.7 Preparation of the test

Adjust the heating bath to maintain the insulating liquid in all oxidation tubes at the required temperature of 120 °C ± 0,5 °C (thermometer complying with the requirements of Annex A).

Weigh in each 25 g ± 0,1 g of insulating liquid into three oxidation tubes and insert the catalyst coil previously prepared as described in 4.5. At least three oxidation tubes are required to be able to measure the DDF (minimum two tubes) and sludge/oil acid number (one tube). Insert the Drechsel head and place the tube into the heater using a rubber O-ring if necessary to close the gap between the tube and the thermal insulated top.

Pour 25 ml of distilled water into one absorption tube. Insert the Drechsel head and connect to the corresponding oxidation tube (see Figure 4).

Adjust the air flow to deliver 0,150 l/h ± 0,015 l/h measured by means of the flowmeter (see 4.2.6) connected to the outlet end of the absorption tube (see Figure 5).

Oxidize the insulating liquid while maintaining its temperature at 120 °C ± 0,5 °C and an air flow-rate of 0,150 l/h ± 0,015 l/h.

Check air flow and temperature daily.

4.8 Determinations on the oxidized insulating liquid

4.8.1 Sludge formation

The sludge shall be precipitated by adhering strictly to the procedure described below.

The sample of 25 g of artificially aged insulating liquid is cooled in the dark for 1 h, and is then poured into an Erlenmeyer flask.

Use 300 ml normal heptane in successive fractions to rinse out the insulating liquid adhering to the test tube, copper spiral and gas lead-in tube and add the washings to the insulating liquid in the flask.