IEC 62021-3

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INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

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

Liquides isolants – Détermination de l'acidité – Partie 3: Méthodes d'essai pour les huiles non minérales isolantes

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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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 62021-3 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications.

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

FDIS	Report on voting
10/936/FDIS	10/942/RVD

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

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

A list of all parts in the IEC 62021 series, published under the general title *Insulating liquids – Determination of acidity*, can be found on the IEC website.

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

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- withdrawn,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

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INTRODUCTION

Health and safety

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

The insulating liquids which are the subject of this standard should be handled with due regard to personal hygiene. Direct contact with the eyes may cause slight 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 procedures referenced in this standard involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

Environment

This standard involves non-mineral insulating oils, chemicals, used sample containers and fluid-contaminated solids. The disposal of these items should be carried out according to local regulations with regard to their impact on the environment. Every precaution should be taken to prevent the release into the environment of these oils.

INSULATING LIQUIDS – DETERMINATION OF ACIDITY –

Part 3: Test methods for non-mineral insulating oils

1 Scope

This part of IEC 62021 describes two procedures for the determination of the acidity of unused and used electrical non-mineral insulating oils. Method A is potentiometric titration and Method B is colourimetric titration.

NOTE 1 In unused and used non-mineral insulating oils, the constituents that may be considered to have acidic characteristics include organic acids, phenolic compounds, some oxidation products, resins, organometallic salts and additives.

The method may be used to indicate relative changes that occur in non-mineral insulating oil during use under oxidizing conditions regardless of the colour or other properties of the resulting non-mineral oil.

The acidity can be used in the quality control of unused non-mineral insulating oil.

As a variety of oxidation products present in used non-mineral insulating oil contribute to acidity and these products vary widely in their corrosion properties, the test cannot be used to predict corrosiveness of non-mineral insulating oil under service conditions.

NOTE 2 The acidity results obtained by potentiometric test method may or may not be numerically the same as those obtained by colourimetric methods, but they are generally of the same magnitude.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60475, *Method of sampling insulating liquids*

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

ISO 6619, *Petroleum products and lubricants – Neutralization number – Potentiometric titration method*

3 Terms and definitions

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

3.1
acidity
quantity of base, expressed in milligrams of potassium hydroxide per gram of sample, required to titrate potentiometrically or colourimetrically a test portion in a specified solvent to the end point

3.2
non-mineral insulating oil
insulating liquid, not derived from petroleum crudes

3.3
unused oil
non-mineral insulating oil that has not been used in, or been in contact with, electrical equipment

4 Method A: Automatic potentiometric titration

4.1 Principle

Any acid-base titration may be conducted potentiometrically. The test portion of the insulating fluid is dissolved in solvent and titrated potentiometrically with alcoholic potassium hydroxide using a glass-indicating electrode and a reference electrode. The potential difference (which can be expressed as pH after calibration) is measured after the successive addition of known increments of alcoholic potassium hydroxide.

Where a strong point of inflection is detected from the first derivative of the titration curve, this should be used as the end point. If only a weak inflection point is present, the potential difference corresponding to pH of 11,5 has been found more reproducible and less instrument-dependent.

4.2 Reagents and auxiliary products

4.2.1 Reagents

Only reagents of recognized analytical grade and de-ionized water or water of equivalent purity shall be used.

4.2.2 Titration reagent

Standard alcoholic solution between 0,01 mol/l and 0,05 mol/l potassium hydroxide (KOH).

EXAMPLE Preparation of 0,01 mol/l potassium hydroxide in 2-propanol.

Add 0,6 g of potassium hydroxide to 1 000 ml ± 10 ml of 2-propanol. Boil gently for 10 min to effect solution. Cool and stopper the flask.

Allow the solution to stand in the dark for 2 days and then filter the supernatant liquid through a 5 µm membrane filter. Store in a suitable amber glass bottle.

The concentration of this solution is approximately 0,01 mol/l and shall be standardized as described in 4.7.2.

Store in such a manner that the solution is protected from atmospheric carbon dioxide by means of a guard tube containing soda-lime absorbent and in such a way that it does not come into contact with cork, rubber or saponifiable stopcock grease.

Commercial alcoholic potassium hydroxide solution may be used, if necessary diluting to 0,01 mol/l with 2-propanol. This shall be standardized as described in 4.7.2.

NOTE 1 For oils with high acidity, which may give an extended titration time, it may be helpful to carry out a pre-test using 0,1 mol/l potassium hydroxide titrant to determine a suitable titrant concentration.

NOTE 2 For periodic tests on equipment in service, faster titration may be achieved by the use of 0,05 or 0,1 mol/l potassium hydroxide by agreement between the laboratory and the equipment owner, although this may result in poorer precision and detection limit.

4.2.3 Titration solvent

The titration solvent is as follows:

– 2-propanol (isopropanol, IPA), pure.

2-propanol is the preferred solvent. It should be noted that the use of other solvents might change the dissociation potential and thus the neutralisation point.

4.2.4 Potassium hydrogen phthalate, primary standard

This should be dried before use for 2 h at 105 °C.

A 0,1 mol/l solution of hydrochloric acid in de-ionized water, prepared as in ISO 6619, may be used. Other acids may be used, e.g. benzoic acid, provided they are certified against a primary standard.

4.2.5 Reference electrode electrolyte

Prepare a solution of potassium chloride in de-ionized water, or lithium chloride in ethanol, at the concentration recommended by the electrode manufacturer. Commercially available solutions may be used where available.

4.2.6 Aqueous buffer solutions

Buffer solutions of suitable pH for calibration of electrodes, for example, pH 4, pH 7 and pH 11 or close to pH 12.

4.2.7 Glass electrode cleaning solution

Weigh 8 g of ammonium peroxydisulfate into a glass beaker. Carefully add 100 ml of 98 % sulphuric acid and gently stir. Before use, the solution should be left overnight for the solid to dissolve completely.

WARNING Ammonium peroxydisulfate is a strong oxidizing agent. Sulphuric acid is a strong corrosive agent. Handle carefully.

Commercially available cleaning solutions as recommended by the electrode manufacturer may be used.

4.3 Apparatus

4.3.1 Potentiometric titration apparatus

An automatic pH titrimeter or an instrument for a potentiometric titration capable of titrating to a fixed end-point using either variable or fixed titrant increments.

The instrument shall be protected from stray electrical fields so that no change of the reading is produced by touching any part of the system with a grounded lead.

An automatic burette with a dispensing accuracy of ±0,005 ml or better is required.

A reservoir for the titrating solution. It should be fitted with a guard tube containing soda lime or other carbon dioxide absorbing material.

4.3.2 Glass indicator electrode

A glass electrode specifically designed for non-aqueous titrations is recommended.

The electrode shall be connected to the potentiometer by means of a suitably screened cable such that the resistance between the screening and the entire length of the electrical connection is greater than 50 000 MΩ.

4.3.3 Reference electrode

The electrode shall be made of glass and shall be reserved for non-aqueous titrations.

Certain alternative electrode-electrolyte combinations have been found to give satisfactory results, although the precision using these alternatives has not been determined. Combined electrodes may be used provided they otherwise conform to this standard and have at least a similar speed of response.

4.3.4 Stirrer

The stirrer should have a variable speed and be fitted with a propeller, paddle or magnetic bar of chemically inert surface material. It shall be electrically grounded to avoid any change in the meter reading during the course of the titration.

4.3.5 Titration vessel

This should be as small as possible, sufficient to contain the solvent, sample, stirrer and electrodes and be inert to the reagents. Glass vessels are preferred to prevent build-up of electrostatic charge.

4.3.6 Titration stand

This should comprise a suitable stand to support the beaker, electrodes, stirrer and burette.

4.4 Sampling

Samples shall be taken following the procedure given in IEC 60475.

Ensure that the test portion is representative by thoroughly mixing, as any sediment present may be acidic or have adsorbed acidic material from the liquid phase.

4.5 Preparation and maintenance of electrode system

4.5.1 Preparation

Although electrodes are not particularly fragile, they should be handled carefully at all times.

Rinse the electrodes with 2-propanol and finally with de-ionized water.

Following each titration immerse the electrodes in de-ionized water to remove any surplus electrolyte adhering to the outside of the electrode and allow excess water to drain off. The immersion time should be sufficient to prevent any memory effects on subsequent titrations.

When in use, any plug that is present on the reference electrode should be removed and the electrolyte level in the electrode kept above that of liquid in the titration vessel to prevent entry of contaminants into the electrode.

4.5.2 Maintenance

4.5.2.1 Glass electrode

Clean the electrode weekly by immersing the tip in 0,1 mol/l hydrochloric acid for 12 h followed by washing with de-ionized water. If more aggressive cleaning is required, immerse the electrode tip in cleaning solution (see 4.2.7) for 5 min and follow this by thorough washing with de-ionized water. This treatment should be carried out on a monthly basis when the electrode is in regular use.

When not in use, immerse the lower half of the electrode in de-ionized water. Do not allow the electrode to dry out. If this occurs it may be possible to reactivate by immersing in cleaning solution (see 4.2.7) as detailed above.

4.5.2.2 Reference electrode

Drain and fill the electrode with electrolyte solution (see 4.2.5) according to the manufacturer's recommendations. When using the sleeve-type electrode, carefully remove the ground-glass sleeve and thoroughly wipe both ground-glass sleeve surfaces. Replace the sleeve loosely and allow a few drops of electrolyte to drain through to flush the ground-glass joint and to wet the ground surfaces thoroughly with electrolyte. Set the sleeve in place and refill with electrolyte (see 4.2.5).

When not in use, immerse the electrode in electrolyte (see 4.2.5) keeping the level of the electrolyte in the electrode above that of the immersion fluid level. The filling apertures should be covered during storage.

The electrode should be cleaned as necessary (at least weekly) by flushing with de-ionized water.

4.6 Calibration

4.6.1 Calibration of pH titrimeter

Determine the pH reading for the buffer solutions (see 4.2.6) on a daily basis. The value of the titration end-point of pH 11,5 is then extrapolated and shall be entered into the instrumental programme.

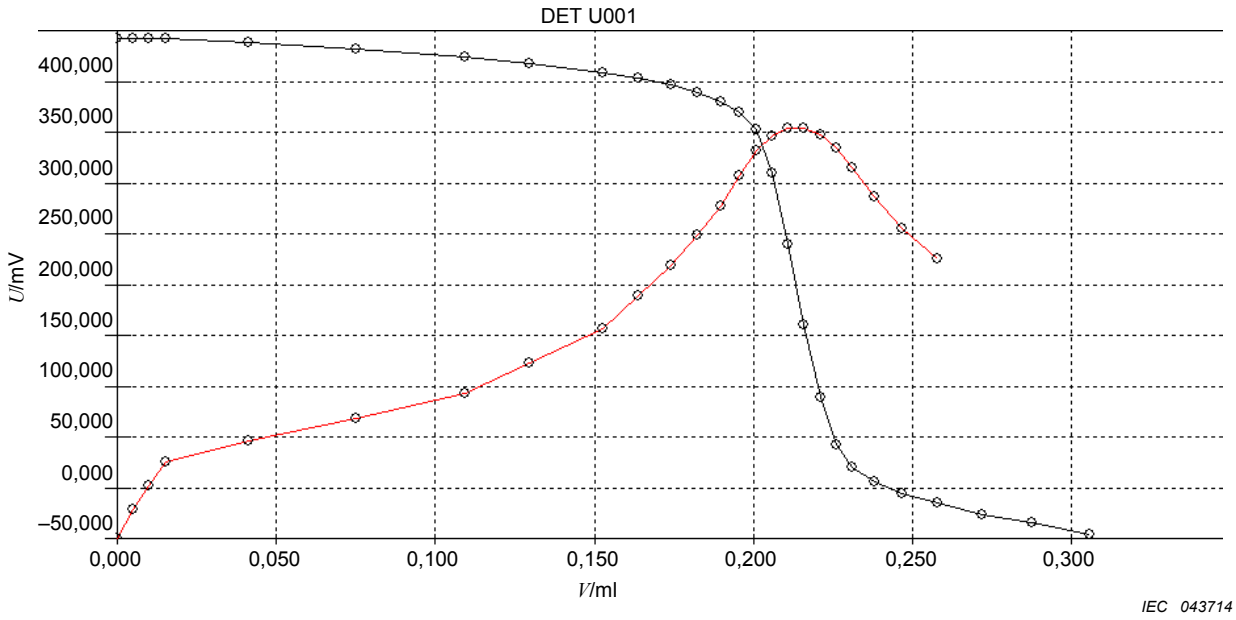
The linearity and slope of the potentiometric titrator over the pH range 4 to 11 should comply with the electrode manufacturer's tolerances.

Temperature correction shall be applied.

Owing to the significant effect of temperature on the pH of the buffer solutions (see 4.2.6), it is desirable to keep the temperature as close to the buffer manufacturer's calibration temperature as possible.

4.6.2 Settings for the potentiometric instrument

Set a potential for an end point titration (usually between –50 mV and –100 mV), which allows the recording of the whole titration curve. For this purpose, use the titration procedure as described in 4.7.3 with the addition of 100 µl of 0,1 mol/l hydrochloric acid (see 4.2.4). See Figure 1.



Key

Black dotted line	first derivative
Red dotted line	exact volume of titrant added (ml).

Figure 1 – Potentiometric titration curve

Most instruments calculate automatically the first derivative of the potential titration curve and the exact volume of titrant added.

4.7 Procedure

4.7.1 General

Set up the apparatus in accordance with the manufacturer's instructions.

Rinse and fill the burette with an alcoholic solution of potassium hydroxide between 0,01 mol/l and 0,05 mol/l (see 4.2.2).

Standardize the 0,01 mol/l or 0,05 mol/l alcoholic potassium hydroxide solution at least every two weeks against potassium hydrogen phthalate (see 4.7.2).

Carry out a blank titration on the solvent (see 4.7.3) each day and after changing to a fresh batch of solvent.

Prepare and titrate a sample of the non-mineral insulating oil against alcoholic potassium hydroxide (see 4.7.4).

4.7.2 Standardization of alcoholic potassium hydroxide solution

Standardize the alcoholic potassium hydroxide solution potentiometrically against 0,1 g to 0,16 g of the potassium hydrogen phthalate, weighed to an accuracy of 0,0002 g and dissolved in approximately 100 ml of carbon dioxide free water.

Depending on the capacity of the titration vessel, the amount of potassium hydrogen phthalate may need to be less than 0,1 g, with a smaller volume of water used to dissolve it. The volume of water shall be enough to dissolve the phthalate and to ensure the complete immersion of the electrode bulb.

Calculate the molarity to the nearest 0,0005, expressed as mol/l, using the following formula.

$$\text{Molarity} = \frac{1000 \times m \times p}{204{,}23 \times V} \quad (1)$$

where

m is the mass of potassium hydrogen phthalate in g;

p is the purity of potassium hydrogen phthalate;

204,23 is the molecular weight of potassium hydrogen phthalate, in g/mol;

V is the volume of alcoholic KOH solution (see 4.2.2) used to titrate the solution, in ml.

Alternatively, standard 0,1 mol/l acid may be used to standardize the alcoholic KOH (see 4.2.4).

$$\text{Molarity} = \frac{V_A \times M_A}{V_B} \quad (2)$$

where

V_A is the volume of 0,1 mol/l standard hydrochloric acid used to titrate the solution, in ml;

M_A is the molarity of the standard hydrochloric acid in mol/l;

V_B is the volume of potassium hydroxide solution, in ml.

4.7.3 Blank titration

Perform a blank titration in duplicate as in 4.7.4, on 20 ml ± 0,1 ml of the solvent (see 4.2.3) daily and after changing to a fresh batch of solvent.

Blank titrations shall be continued until two consecutive titrations differ by no more than 0,005 ml, based on 20 ml of solvent and the mean of these is calculated as V_0 (see 4.8).

Where a higher solvent volume than 20 ml is required because of apparatus constraints, the same volume of solvent shall be used for the sample titration.

High values may arise from carbon dioxide absorption or inherent 2-propanol acidity. If the blank value is greater than 0,06 ml (based on 20 ml of solvent), steps shall be taken to remove the cause of the high values.

4.7.4 Sample titration

Prepare the sample for titration as described in 4.4 and weigh 5 g ± 0,1 g of the non-mineral insulating oil to the nearest 0,01 g into the titration vessel. Add 20 ml ± 0,1 ml of titration solvent (see 4.2.3).

The amount of solvent added may depend on the testing device used, the volume and shape of vessel, etc. Add an amount of titration solvent sufficient to ensure the complete immersion of electrode's bulb.

Place the titration vessel on the titration stand and stir the solution until the sample has dissolved and the pH reading is constant, taking care to limit the speed of stirring to avoid spattering and/or stirring air into the solution.