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Insulating liquids – Determination of acidity

Part 2: Colourimetric titration

Liquides isolants – Détermination de l'acidité

Partie 2: Titrage colorimétrique

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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 62021-2 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
10/692/FDIS	10/696/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 of IEC 62021 series, 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 maintenance result 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- withdrawn;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.

INTRODUCTION

The standardized method given in IEC 62021-1 is a method for measurement of acidity in used and unused mineral oil and is a potentiometric titration requiring special instrumentation for measurement of acidity. Historically, acidity of insulating oil was measured by colourimetric titration as described in IEC 60296, 1982 edition. With the revision of IEC 60296, the colourimetric titration was deleted as that method used high volumes of sample and solvent, generating undesirable volumes of waste.

However, there is still a market requirement for having colourimetric titration as many labs use this method.

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 mineral oils which are the subject of this standard should be handled with due regard to personal hygiene. Direct contact with 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 tests specified in this standard involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

This standard involves mineral oils, chemicals and used sample containers. The disposal of these items should be carried out in accordance with current national legislation with regard to the impact on the environment. Every precaution should be taken to prevent the release into the environment of mineral oil.

INSULATING LIQUIDS – DETERMINATION OF ACIDITY –

Part 2: Colourimetric titration

1 Scope

This part of IEC 62021 describes a procedure for determination of the acidity of unused and used electrical mineral insulating oils.

NOTE 1 In unused and used 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 a mineral insulating oil during use under oxidizing conditions that may or may not be shown by other properties of the resulting mineral oil.

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

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

NOTE 2 The acidity results obtained by this test method may or may not be numerically the same as those obtained by potentiometric methods, but they are generally of the same magnitude. The potentiometric method uses an endpoint at pH 11,3 to ensure titration of all species, whereas the colourimetric methods uses an indicator changing colour at approximately pH 9,5. This may lead to slightly higher results for oils with acidities above 0,3 mg KOH/g oil when using the potentiometric method.

2 Normative references

The following referenced documents are indispensable for the application 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 60475: *Method of sampling liquid dielectrics*

IEC 60567: *Oil-filled electrical equipment – Sampling of gases and of oil for analysis of free and dissolved gases – Guidance*

ISO 5725: *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 colourimetrically a test portion in a specified solvent to the neutralization point of Alkali Blue 6B

3.2
unused oil
mineral insulating oil that has not been used in, or been in contact with electrical equipment

4 Principle

The test portion is dissolved in a specified solvent and titrated colourimetrically with alcoholic potassium hydroxide to a specified colour using Alkali Blue 6B indicator.

5 Reagents

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

5.1 Titration reagent

Standard alcoholic solution 0,05 mol/l potassium hydroxide.

Add 3,0 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 0,05 mol/l and shall be standardized as described in 8.1. For periodic tests on equipment in service, faster titration may be achieved by the use of 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.

Commercial alcoholic potassium hydroxide solution may be used, if necessary diluting to 0,05 mol/l with 2-propanol. This shall be standardized as described in 8.1.

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

5.2 Titration solvent

2-propanol (isopropanol; IPA), pure.

5.3 Potassium hydrogen phthalate, primary standard

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

5.4 Standard hydrochloric acid solution

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, provided they are certified against a primary standard.

5.5 Alkali blue 6B indicator solution

Dissolve 2 g ± 0.1 g of alkali blue 6B in 100 ml of 2-propanol or azeotropic ethanol containing 1 ml of the hydrochloric acid solution. After 24 h, carry out a titration to check whether the indicator has been sufficiently sensitized. The indicator is satisfactory if the colour changes distinctly from blue to red comparable to that of a 10 % solution of cobalt nitrate. If sensitization is insufficient, repeat the addition of the hydrochloric acid solution and check again after 24 h. Continue until sensitization is satisfactory. Filter and store in a brown bottle in the dark.

Commercial alkali blue 6B solution may be used as an alternative if the concentration is within the range 0,05 % to 5 %. If the concentration is not 2 %, the amount added to the solvent in 8.2 and 8.3 should be adjusted to maintain the same ultimate concentration.

5.6 Cobalt nitrate solution

$Co(NO_3)_2.6H_2O$ solution, 10 % in water.

6 Apparatus

6.1 Titration vessel

This should be as small as possible, sufficient to contain the solvent, sample and stirrer and be inert to the reagents. Glass conical vessels are preferred.

6.2 Stirrer

Stirring may be manual by swirling the solution on the titration vessel, or mechanically using a variable speed stirrer fitted with a propeller, paddle or magnetic bar of chemically inert surface material.

6.3 Burette

A burette or syringe capable of adding aliquots of 0,001 ml shall be used.

7 Sampling

Samples shall be taken following the procedure given in IEC 60475 and/or IEC 60567.

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.

8 Procedure

Rinse and fill the burette with 0,05 mol/l alcoholic potassium hydroxide solution (5.1).

Standardize the alcoholic potassium hydroxide solution at least every two weeks against potassium hydrogen phthalate (5.3) or certified standard 0,1 mol/l acid.

Carry out a blank titration on the solvent (5.2) each day and after changing to a fresh batch of solvent.

8.1 Standardization of alcoholic potassium hydroxide solution

Standardize the alcoholic potassium hydroxide solution, using a suitable indicator, against 0,1 g to 0,16 g of potassium hydrogen phthalate, weighed to an accuracy of 0,000 2 g and dissolved in approximately 100 ml of carbon dioxide free water.

Alternatively the standardization can be performed by potentiometric titration.

Calculate the molarity M to the nearest 0,000 5 using Equation (1):

$$\text{Molarity} = \frac{1\,000 \times m \times p}{204{,}23 \times V} \qquad (1)$$

where

m is the mass of potassium hydrogen phthalate, in grams;

p is the percent purity of the potassium hydrogen phthalate;

V is the volume of potassium hydroxide solution, in millilitres.

Alternatively, certified standard 0,1 mol/l acid may be used to standardize the alcoholic potassium hydroxide solution.

Calculate the molarity M to the nearest 0,000 5 using Equation (2):

$$\text{Molarity} = \frac{V_1 \times M_A}{V_0} \qquad (2)$$

where

V_1 is the volume of 0,1 mol/l standard acid used to titrate the solution, in millilitres;

M_A is the molarity of the standard hydrochloric acid;

V_0 is the volume of potassium hydroxide solution, in millilitres.

8.2 Blank titration

Perform a blank titration at a temperature not above 25 °C on 10 ml ± 0,1 ml aliquots of the solvent containing 0,5 % of alkali blue 6B indicator solution (5.5) using the standardized alcoholic potassium hydroxide solution. The endpoint shall be as soon as a colour change from blue to a red colour comparable to that of the cobalt nitrate solution (5.6) is obtained and persists for at least 15 s.

Carry out triplicate titrations and calculate the mean result, in millilitres to the nearest 0,001 ml, as the blank value V_0.

Protect the solvent from atmospheric carbon dioxide and use within 8 h.

8.3 Sample titration

Weigh 5 g of sample to the nearest 0,01 g into the titration vessel. Add 10 ml ± 0,1 ml of the solvent solution containing 0,5 % of alkali blue 6B indicator solution (5.5). Swirl to dissolve the oil and immediately titrate at a temperature not above 25 °C with the standardized potassium hydroxide solution. A typical end point is as described in 8.2. However, since the colour change may vary for different oils, pre-titration may be necessary to establish this. In such cases, the endpoint shall be reached as soon as a stable colour change, which persists for at least 15 s, is obtained.

NOTE Before titrating, the colour may vary from blue to green and at the endpoint from red to light orange to dark yellow-brown, depending on the original colour of the oil.

Carry out determinations for each oil sample and note the result, in millilitres, to the nearest 0,001 ml, as the titration value V_1.

9 Calculation of results

Calculate, for each determination, the acidity to the nearest 0,005, expressed as mg KOH/g of oil, using Equation (3):

$$\text{Acidity} = \frac{(V_1 - V_0) \times M \times 56{,}1}{m} \quad (3)$$

where

V_1 is the volume of alcoholic KOH solution used to titrate the test sample, in millilitres;

V_0 is the mean volume of alcoholic KOH solution used for the blank titration, in millilitres;

M is the molarity of alcoholic KOH solution;

m is the mass of the test portion used, in grams.

10 Precision

The repeatability and reproducibility limits were established in accordance with ISO 5725.

10.1 Repeatability

The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values shown below only in one case in 20:

– unused oils –15 %;

– used oils –10 %.

NOTE The repeatability values for unused oils only apply where the result is significantly above the quantification limit, which has been established as 0,01 mg KOH/g oil.