SLOVENSKI STANDARD
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Insulating oil - Determination of fibre contamination by the counting method using a microscope

Insulating oil - Determination of fibre contamination by the counting method using a microscope

Isolieröl - Bestimmung der Faserverunreinigungen mittels eines Zählverfahrens mit einem Mikroskop

Huiles isolantes - Détermination de la contamination par fibres par la méthode du comptage avec utilisation d'un microscope

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English version

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CENELEC
European Committee for Electrotechnical Standardization
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Foreword

This European Standard was prepared by the CENELEC BTWG 99-1, Insulating oil.

The text of the draft was submitted to the Unique Acceptance Procedure and was approved by CENELEC as EN 50353 on 2000-12-01.

The following dates were fixed:

- latest date by which the EN has to be implemented at national level by publication of an identical national standard or by endorsement (dop) 2002-04-01

- latest date by which the national standards conflicting with the EN have to be withdrawn (dow) 2004-01-01

Annexes designated "normative" are part of the body of the standard.
In this standard, annex A is normative.

Warning This European Standard calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

Introduction

It is generally recognized that fibres and other particles have a detrimental effect on the dielectric strength of insulating oil. It has long been a requirement to include in specifications for insulating liquids that the fluid be clear and free from visible particulate matter or sediment. This Standard provides the methodology for testing for contamination by fibres and consists of filtering a known volume of the insulating liquid under vacuum assisted conditions through a membrane filter to collect contaminants on the filter surface. The membrane is then examined microscopically to count and size the fibres.

1 Scope

This European Standard specifies two methods for determining the fibre contamination of mineral insulating oil used in electrotechnical equipment, based on filtering a sample of oil and examining and counting the number of fibres on the surface of the filter using an optical microscope. Fibres down to 100 μm in length can be sized and counted by these methods. The methods are applicable both to unused oils and to oils in service.

The method specified in the main text is applicable to oil samples taken from barrelled oil or from electrotechnical equipment in service, in which the fibre concentration is expected to be relatively high. The alternative method specified in annex A is applicable to samples taken from new oil in bulk containers, or from large transformers which have not been operated much since they were filled, in which the fibre concentration is expected to be relatively low.

NOTE These methods are also suitable for other insulating liquids such as silicones and synthetic esters.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other standards. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

IEC 60475 *Method of sampling liquid dielectrics*

ISO 5598 *Fluid power systems and components - Vocabulary*

3 Terms and definitions

For the purposes of this European Standard, the terms and definitions given in ISO 5598 and the following apply.

3.1
blank count
count of the number of fibres introduced from other sources, such as reagents or cleaning of glassware

3.2
effective filtration area
area of a membrane filter open to flow during filtration of the fluid

NOTE For commercially obtainable gridded membrane filters of diameter 37 mm, this is generally accepted as 100 grid squares of dimension 3,1 mm, with a nominal area of 960 mm^2.

3.3
detectable fibre
fibre longer than 100 µm with a length-to-width ratio greater than or equal to 5:1

NOTE For the purposes of this standard, detectable fibres are defined as longer than 100 µm because experience has shown that only these fibres cause severe degradation of dielectric strength in practical situations.

4 Principle

A known volume of insulating oil is filtered, using vacuum assistance, through a membrane filter to separate contaminants from the sample liquid and deposit them on the surface of the membrane filter. The contamination is examined microscopically to size and count any fibres present.

5 Reagents

NOTE These chemicals are used for cleaning and rinsing the apparatus.

5.1 *Liquid detergent*, without solid residue.

5.2 *Distilled or de-mineralized water*.

5.3 *2-propanol*, acetone-free.

5.4 *Solvent* - petroleum ether, boiling range 100°C to 120°C preferred, but heptane, hexane and cyclohexane are suitable alternatives.

WARNING - Petroleum ether and many of its alternatives are highly flammable solvents which have low flash points. It is essential that appropriate safety measures are observed during use of these solvents. Only small quantities (e.g. not greater than about 100 ml) should be used on the workbench and larger quantities should be handled in a fume cupboard to avoid the inhalation of fumes from these solvents.

6 Apparatus

NOTE An example of suitable apparatus is shown in Figure 1.

6.1 *Membrane filter holder*, consisting of the following components:

- a glass funnel of 250 ml capacity, with a loosely fitting glass cap;
- a clamping device;
- a suitable base to support the membrane filter (e.g. a glass frit).

NOTE If problems are experienced due to build-up of electrostatic charge on the filter during filtration, it may be necessary to use an earthed metal anti-static base to support the membrane filter.

6.2 *Membrane filters*, with a pore diameter of 1,2 µm or less. Gridded filters shall be used for manual counting and ungridded filters shall be used if an automatic counting system is to be employed (see 6.8).

NOTE 1 It is essential that a type of filter is selected which is known to be compatible with the oil sample and with the solvents which are to be used (see clause 5).

NOTE 2 Black membranes may be helpful for the purpose of counting, by improving the visibility of fibres where most of them are translucent, transparent or white in colour.

6.3 *Vacuum flask* (for waste filtrate), glass, with side arm, of 1 l capacity and capable of accepting the membrane filter holder (6.1).

6.4 *Vacuum pump*, capable of establishing a vacuum to assist the flow of oil through the membrane.

NOTE A condensing trap may be used to protect the vacuum pump from vapours.

6.5 *Filtered-solvent dispenser*, comprising a pressure-operated system which discharges the solvent through an in-line membrane filter with a pore diameter of 1,2 μm or less.

NOTE The type of filter selected shall be compatible with the solvents used (see clause 5).

6.6 *Tweezers,* stainless steel, flat ended, un-serrated.

6.7 *Petri dishes*, glass, with cover.

6.8 *Microscope*, capable of producing magnifications of ×50, equipped with fine and coarse focus controls, having an eyepiece graticule, and fitted with a mechanical stage so that the effective filtration area of the filter can be scanned.

NOTE The microscope may be fitted with an image analysis system if automatic determination of fibre size distribution is required.

6.9 *Stage micrometer*, graduated in 0,1 mm and 0,01 mm divisions, with its calibration traceable to national standards.

6.10 *External lamp*, of variable intensity, if the microscope does not include an inbuilt light source.

6.11 *Sample bottles*, minimum capacity 250 ml, cylindrical, wide-mouthed, clear glass with a non-fibre-shedding, oil-tight closure.

7 Cleaning procedure

Clean the filtration apparatus, tweezers and petri dishes, and the sample bottles and closures as follows:

a) wash the glassware, and the tweezers, in warm tap water/liquid detergent solution;

b) rinse three times with distilled or de-mineralized water;

c) rinse with filtered 2-propanol to remove water;

d) rinse with filtered petroleum ether, using a spirally directed jet where possible;

e) suspend the funnel in suitable sized beaker, wash down with petroleum ether and then cover;

f) leave a little solvent in each sample bottle and cap with a pre-cleaned closure.

NOTE Solvent evaporation slightly pressurises the bottle and therefore excludes contamination when the bottle is opened.

8 Sampling

Clean the sampling equipment in accordance with clause 7.

Ensure that the sampling equipment is free from fibrous contamination by flushing with the oil being sampled.

Extract a sample (of minimum volume 250 ml) of the insulating oil from the reservoir/ container in accordance with the method laid down in IEC 60475.

NOTE The fibre count of an oil sample from any item of electrotechnical equipment can depend on the sampling point, the time elapsed since filling, the oil circulation rate and/or the time the oil is left to stand. Circulation of oil in the tank of the electrical equipment before taking the samples can be expected to yield more consistent fibre count results.

Storage of the samples will generally lead to sedimentation and coalescence of fibres and particles. Therefore, the analysis should be undertaken as soon as possible after sampling. It has been established that analysis of a sub-sample from a sample container may produce inaccurate results. Therefore the total contents of the sample container shall be used for the test.

9 Filtration procedure

9.1 General

A blank test, to determine the blank count, in accordance with 9.3, shall be carried out before the filtration of each batch of oil samples (see 9.2). If the batch of oil samples is so large that the analysis takes more than a week, a blank test, to determine the blank count, shall be carried out once a week.

It is essential to minimize fibre contamination during filtration and counting. Therefore, it is recommended that these procedures are carried out in either:

a) a laminar flow cabinet; or

b) a clean room reserved specifically for microscopy, in which no activities likely to generate fibres are carried out.

Precautions should also be taken to minimize fibre shedding from the operators' clothing. It is recommended that operators should wear non fibre-shedding overclothes and should cover their hair.

NOTE The material of these overclothes shall not pose high risk for the wearer in case of fire.

9.2 Filtration of oil sample

9.2.1 Using cleaned tweezers, remove a membrane filter from its container, inspect it (using the microscope) to ensure that it is fibre-free and place it, grid side up, centrally on the screen of the filter holder base. Carefully install and clamp the funnel in position. Do not remove the cap from the funnel opening until ready to start filtration.

9.2.2 Connect the vacuum pump to the side arm of the vacuum flask.

9.2.3 After noting any details (sample identity etc.), remove any tied-on or loosely attached labels from the sample bottle, taking great care to retain the sample identity.