

Insulating liquids — Measurement of relative permittivity, dielectric dissipation factor ($\tan \delta$) and d.c. resistivity

The European Standard EN 60247:2004 has the status of a
British Standard

ICS 17.220.99; 29.040.10

National foreword

This British Standard is the official English language version of EN 60247:2004. It is identical with IEC 60247:2004. It supersedes BS 5737:1979 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee GEL/10, Fluids for electrotechnical applications, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 26 April 2004

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 27 and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments

© BSI 26 April 2004

ISBN 0 580 43672 1

EUROPEAN STANDARD

EN 60247

NORME EUROPÉENNE

EUROPÄISCHE NORM

April 2004

ICS 17.220.99; 29.040.10

English version

**Insulating liquids –
Measurement of relative permittivity,
dielectric dissipation factor ($\tan \delta$) and d.c. resistivity
(IEC 60247:2004)**

Liquides isolants –
Mesure de la permittivité relative,
du facteur de dissipation diélectrique
($\tan \delta$) et de la résistivité en courant
continu
(CEI 60247:2004)

Isolierflüssigkeiten –
Messung der Permittivitätszahl,
des dielektrischen Verlustfaktors ($\tan \delta$)
und des spezifischen Gleichstrom-
Widerstandes
(IEC 60247:2004)

This European Standard was approved by CENELEC on 2004-04-01. CENELEC members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CENELEC member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CENELEC member into its own language and notified to the Central Secretariat has the same status as the official versions.

CENELEC members are the national electrotechnical committees of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

CENELEC

European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

Central Secretariat: rue de Stassart 35, B - 1050 Brussels

© 2004 CENELEC - All rights of exploitation in any form and by any means reserved worldwide for CENELEC members.

Ref. No. EN 60247:2004 E

Foreword

The text of document 10/573/FDIS, future edition 3 of IEC 60247, prepared by IEC TC 10, Fluids for electrotechnical applications, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 60247 on 2004-04-01.

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) 2005-01-01
- latest date by which the national standards conflicting with the EN have to be withdrawn (dow) 2007-04-01

Annex ZA has been added by CENELEC.

Endorsement notice

The text of the International Standard IEC 60247:2004 was approved by CENELEC as a European Standard without any modification.

CONTENTS

INTRODUCTION.....	5
1 Scope.....	6
2 Normative references.....	6
3 Terms and definitions	6
4 General	7
4.1 Permittivity and dielectric dissipation factor ($\tan \delta$)	7
4.2 Resistivity.....	8
4.3 Sequence of determinations.....	8
4.4 Factors leading to erroneous results	8
5 Apparatus.....	9
5.1 Test cell	9
5.2 Test equipment.....	10
5.3 Glassware	10
5.4 Measuring instrument for permittivity and $\tan \delta$	10
5.5 Measuring instrument for d.c. resistivity	10
5.6 Time-measuring device.....	10
5.7 Safety.....	10
6 Cleaning solvent	11
7 Cleaning the test cell	11
7.1 Trisodium phosphate cleaning procedure	11
7.2 Storage of cell	12
8 Sampling	12
9 Preparation of samples	12
10 Conditioning and filling the test cell	13
10.1 Cell conditioning	13
10.2 Filling the cell	13
11 Test temperature	13
12 Measurement of dissipation factor ($\tan \delta$).....	14
12.1 Test voltage.....	14
12.2 Measurement	14
12.3 Report.....	14
13 Measurement of relative permittivity	14
13.1 Measurement	14
13.2 Report	15
14 Measurement of d.c. resistivity.....	15
14.1 Test voltage.....	15
14.2 Time of electrification	15
14.3 Measurement	15
14.4 Report	16

Annex A (informative) Example of an alternative procedure for cleaning the test cell –
Ultrasonic procedure 17

Annex B (informative) Example of a simplified cleaning procedure for a test cell 18

Annex C (informative) Alternative procedures for routine testing of dielectric dissipation
factor and resistivity of insulating liquids 19

Annex ZA (normative) Normative references to international publications with their
corresponding European publications 27

Figure 1 – Example of a three-terminal cell for measurements on liquids22

Figure 2 – Example of screening for the cell of Figure 1.....23

Figure 3 – Example of assembling drawing of cell.....24

Figure 4 – Example of a two-terminal cell for measurements in liquids25

Figure 5 – Example of a test cell designed for low-loss dielectric liquids26

INTRODUCTION

Health and safety

General caution. 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.

Environment

This standard gives rise to insulating liquids, chemicals, used sample containers and oil 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 liquids.

INSULATING LIQUIDS – MEASUREMENT OF RELATIVE PERMITTIVITY, DIELECTRIC DISSIPATION FACTOR ($\tan \delta$) AND DC RESISTIVITY

1 Scope

This International standard describes methods for the determination of the dielectric dissipation factor ($\tan \delta$), relative permittivity and d.c. resistivity of any insulating liquid material at the test temperature.

The methods are primarily intended for making reference tests on unused liquids. They can also be applied to liquids in service in transformers, cables and other electrical apparatus. However the method is applicable to a single phase liquid only. When it is desired to make routine determinations, simplified procedures, as described in Annex C, may be adopted.

With insulating liquids other than hydrocarbons, alternative cleaning procedures may be required.

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 60093, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials*

IEC 60250, *Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths*

IEC 60475, *Method of sampling liquid dielectrics*

IEC 61620, *Insulating liquids – Determination of the dielectric dissipation factor by measurement of the conductance and capacitance – Test method*

3 Terms and definitions

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

3.1

permittivity (relative)

the relative permittivity of an insulating material is the ratio of capacitance C_x of a capacitor in which the space between and around the electrodes is entirely and exclusively filled with the insulating material in question, to the capacitance C_0 of the same configuration of electrodes in vacuum.

The capacitance C_a of the configuration of electrodes in air can normally be used instead of C_0 to determine the relative permittivity with sufficient accuracy

3.2 dielectric dissipation factor ($\tan \delta$) DDF

dielectric dissipation factor, DDF, ($\tan \delta$) of an insulating material is the tangent of the loss angle.

The loss angle is the angle by which the phase difference between applied voltage and the resulting current deviates from $\pi/2$ rad when the dielectric of the capacitor consists exclusively of the insulating material

NOTE For practical purposes, measured values below 0,005 for $\tan \delta$ and power factor are essentially the same. A simple conversion can be used to convert one to the other. The power factor is the sine of the loss angle and the relationship between power factor and dielectric dissipation factor can be expressed as follows:

$$PF = \frac{DDF}{\sqrt{1 + (DDF)^2}} \quad (1)$$

3.3 d.c. resistivity (volume)

the volume resistivity of an insulating material is the quotient of a d.c. electrical field strength and the steady state current density within the material

NOTE The unit of resistivity is the ohmmetre (Ωm).

4 General

Permittivity, $\tan \delta$ and resistivity, either separately or together, are important indicators of the intrinsic quality and degree of contamination of an insulating fluid. These parameters may be used to interpret the deviation from desired dielectric characteristics and the potential influence on performance of equipment in which the fluid is used.

4.1 Permittivity and dielectric dissipation factor ($\tan \delta$)

The permittivity and the dielectric dissipation factor ($\tan \delta$) of electrical insulating liquids depend to a considerable extent on the test conditions under which they are measured, in particular on the temperature and on the frequency of the applied voltage. Permittivity and dissipation factor are the measurements of dielectric polarization and conductivity of the material.

At power frequency and sufficiently high temperature, as recommended in these methods, the losses may be attributable exclusively to the conductivity of the liquid, that is, to the presence of free charge carriers in the liquid. Measurements of the dielectric properties of high purity insulating liquids are therefore of value as an indication of the presence of contaminants.

The dielectric loss factor is usually inversely proportional to the measuring frequency and varies with the viscosity of the medium. The value of the test voltage when measuring the dissipation factor is less important and often governed by the sensitivity of the measuring bridge. However, it must be borne in mind that too high a voltage stress results in secondary phenomena at the electrodes, dielectric heating, discharges etc.

While relatively large amounts of impurities produce a comparatively small change in permittivity, the $\tan \delta$ of insulating liquids may be strongly affected by traces of dissolved contaminants or colloidal particles. Some liquids are much more sensitive to contamination than hydrocarbon liquids due to their higher polarity, which results in turn in higher solvent power and dissociation capability. Therefore, they require comparatively greater care in handling than hydrocarbon liquids.

Since the initial value is thought to be representative of the actual conditions of the liquid, it appears most desirable that $\tan \delta$ should be measured as soon as temperature equilibrium has been reached. $\tan \delta$ is very sensitive to changes of temperature. Its increase, with increases in temperature is generally exponential. It is therefore, necessary to carry out measurements, under sufficiently precise temperature conditions. The procedure described below allows the test specimen to attain temperature equilibrium with the test cell in minimum time.

4.2 Resistivity

The conventional resistivity as measured by this standard is generally not the true resistivity. Application of a d.c. voltage will change the initial characteristics of the liquid with time, due to charge migration. The true resistivity can only be obtained at low voltage, immediately after application of the voltage. This standard uses a relatively high voltage for an extended time and the result will generally be different from that from IEC 61620.

Measurements of resistivity of liquids to this standard, depends on a number of test conditions, namely:

a) Temperature

Resistivity is very sensitive to changes of temperature, its dependence on the inverse of the temperature, expressed in Kelvin, (1/K) is generally exponential. It is therefore necessary to carry out measurements under sufficiently precise temperature conditions.

b) Magnitude of the electrical field

The resistivity of a given specimen may be influenced by the applied stress. For results to be comparable, measurements shall be made with approximately equal voltage gradients and with the same polarity. The gradients and the polarity shall be noted.

c) Time of electrification

Upon the application of d.c. voltage, the current flow through the specimen decreases due to the sweep of charge carriers to the electrodes. The conventional arbitrary time of electrification is 1 min. Variation in the time of electrification can result in appreciable variation in the test results. (Some high viscosity fluids may require considerably longer electrification time (see 14.2).)

4.3 Sequence of determinations

Application of d.c. voltage to a specimen can modify the results of a subsequent a.c. determination of $\tan \delta$.

When permittivity, $\tan \delta$ and resistivity measurements have to be made consecutively on the same specimen, the a.c. determination shall always be made before applying the d.c. voltage to the specimen. The cell electrodes should be short-circuited for a minimum of 1 min after the a.c. tests, before beginning the resistivity measurements.

4.4 Factors leading to erroneous results

Only gross contamination is likely to affect permittivity. However, DDF and resistivity can be strongly affected by minute amounts of contamination.

Unreliable results usually originate from contamination due to improper sampling or handling of liquid specimens, from incomplete cleaning of the cells or from the absorption of water, and especially from the presence of undissolved water.

Extended exposure to light during storage may lead to deterioration of dielectric characteristics. Standardized procedures for the storage and transfer of the liquid samples and for the construction and cleaning of test cells are recommended so that errors caused by contamination are minimized.

5 Apparatus

5.1 Test cell

The same test cell can be used for measurements of permittivity, $\tan \delta$ and d.c. resistivity. A cell suitable for these purposes shall meet the following general requirements.

5.1.1 The design of the cell shall be such as to allow easy dismantling for cleaning of all its parts and reassembling without significantly changing the capacitance of the empty cell. The design shall also permit the use of the cell at the required constant temperature and shall provide means to measure and control the temperature of the liquid to the required accuracy. This may be achieved by means of an externally heated oven/bath or alternatively by internal electrical heating of the cell.

5.1.2 The materials used in constructing the cell shall be non-porous and capable of withstanding the required temperature satisfactorily. The alignment of the electrodes shall not be influenced by change of temperature.

5.1.3 The surfaces of the electrodes in contact with the liquid under test shall have a smooth finish to make their cleaning easier. There should be no chemical interactions between the liquids under test and the electrodes. These should also be unaffected by the cleaning materials. Cells made of stainless steel have been found satisfactory for testing all types of insulating liquids. Aluminium or its alloys should not be used because they may be attacked by alkaline detergents.

NOTE Generally, plated surfaces have been found less satisfactory than solid metal electrodes. However, surfaces plated with gold, nickel or rhodium have been found satisfactory provided they are well plated and remain undamaged. Rhodium-plated invar has been found satisfactory and has the additional advantage of low thermal expansion. Nickel or gold-plated brass and nickel-plated stainless steel have also been used.

5.1.4 Solid insulating materials used to support the electrodes shall have a low $\tan \delta$ and a high resistivity. They should not absorb, or be adversely affected by, reference liquids, test liquids or cleaning materials.

NOTE Fused silica is generally considered to be a suitable material for use as an insulating material in the cell. As a consequence of the difference between the coefficients of linear expansion of usual metals and of fused silica, a sufficient radial clearance is necessary between the joints. This clearance may decrease the accuracy of electrode gap spacing.

5.1.5 The distance across the surface of the liquid and across the solid insulating material, between the guard electrode and the measuring electrode, shall be great enough to withstand the test voltage used.

5.1.6 Any cell which meets the requirements given in 5.1.1 to 5.1.5 may be used. Examples of cells (not exclusive or exhaustive) which may be used with low viscosity liquids and up to 2 000 V are shown diagrammatically in Figures 1 to 5.

In the three-terminal cells, provision is made for an efficient electrical guard system which adequately shields the measuring electrode. Three-terminal cells shall be preferred when the most accurate determinations of permittivity are to be made. If necessary, in the case of measurements for which special screening is required, a removable screening cup can be added and electrically bonded to the appropriate conductor of the coaxial cable used for the connection to the bridge (see Figure 2).

With two-terminal cells, the shield on the lead usually connected to the guard electrode shall be securely clipped to the cable insulation in order to prevent contact with any other surface. When these cells are used for resistivity measurements it should be established that the resistance across the insulators, with the cell empty, is at least 100 times the resistance which is measured through the liquid. For a.c. measurements corresponding values for $\tan \delta$ shall be established.

For highly insulating liquids, the additional losses due to the insulators that support the electrodes may alter the measurement. For this reason, it is recommended to use test cells in which there is no bridge made from any solid insulating material between the two measurement electrodes. The dissipation factor of such an empty cell should be lower than 10^{-6} at 50 Hz.

To minimize the effect of contaminants from the surfaces in contact with the liquid, it is recommended to use cells with a low ratio of electrode surface area to liquid volume (for example, $<5 \text{ cm}^{-1}$).

5.2 Test equipment

Test equipment shall be adequate to maintain the test cell with $\pm 1 \text{ }^\circ\text{C}$ of the prescribed temperature and shall provide screened electrical connections to the cell. The test cell shall be properly insulated from the earthed enclosure of the test equipment.

5.3 Glassware

Borosilicate laboratory glassware such as beakers, graduated cylinders, pipettes, etc., shall be used. All glassware used in handling samples shall be cleaned at least to the standard specified in Clause 6, and carefully dried.

5.4 Measuring instrument for permittivity and $\tan \delta$

Any a.c. capacitance and $\tan \delta$ measuring device capable of a measuring precision and resolution appropriate to the samples to be tested can be used.

Examples of a.c. capacitance bridges and testing circuits may be found in IEC 60250.

5.5 Measuring instrument for d.c. resistivity

Suitable instruments and test circuits are described in IEC 60093. Any apparatus with the precision and resolution appropriate to the samples to be tested may be used.

5.6 Time-measuring device

A time-measuring device, accurate to 0,5 s, shall be used for measuring the time of electrification.

5.7 Safety

DANGER – Ensure that equipment safety devices are functioning properly.

6 Cleaning solvent

Solvents for cleaning the test cell shall have a purity at least corresponding to the requirements for technical grades and shall have been shown to have no influence on test results. Solvents shall be stored in brown glass bottles.

If the solvent is delivered in drums, any particulate matter shall be removed by filtration and the filtered solvent stored in properly marked brown glass bottles.

Hydrocarbon solvents such as petroleum spirit (boiling range 60 °C to 80 °C), n-heptane, cyclohexane and toluene are suitable for hydrocarbon oils. Ethanol is recommended for organic esters and toluene for silicone liquids. Other insulating liquids may require special solvents.

7 Cleaning the test cell

The cleanliness of the test cell is of paramount importance when measuring dielectric properties because of the extreme susceptibility of insulating liquids to the influence of the most minute contamination.

The cell shall always be cleaned immediately before use in a reference test.

It is strongly recommended that cells in continuous use for routine measurements are regularly cleaned.

Cleaning need not precede a routine measurement made subsequent to one in which the property measured was within the limiting value specified and the liquid was of a similar chemical type. Prior to the next test, the cell shall be flushed with a volume of the next sample corresponding to at least three fillings of the cell.

When cells are to be regularly used for testing fluids with similar chemical type and having similar electrical properties, they may be stored filled with a clean sample of the fluid and simply flushed with a volume of the next sample corresponding to at least three fillings of the cell.

Many different types of cleaning procedure can be used provided that they have been proved to be efficient.

Examples of alternative procedures are given in Annexes A and B.

The following reference procedure shall be used in the case of dispute between laboratories.

CAUTION: Appropriate precautions against fire and toxic effects on personnel shall be observed when using solvents.

7.1 Trisodium phosphate cleaning procedure

Dismantle the cell completely.

Wash all the component parts thoroughly with two portions of the solvent (see Clause 6). Rinse all parts with acetone and then wash them with a mild abrasive soap or mild abrasive detergent.

Abrasive particles and rubbing action should be such as to cause no deterioration of the smooth finish of metal surfaces.

Boil all parts for at least 5 min in 5 % trisodium phosphate solution in distilled or de-ionized water, followed by several rinses with distilled or de-ionized water.

Boil all parts in distilled or de-ionized water for at least 30 min.

Thoroughly dry the component parts in an oven heated at 105 °C to 110 °C for no longer than 120 min because certain materials may deteriorate. Drying time depends on the design of the cell but usually 60 min to 120 min is satisfactory to remove any moisture.

Reassemble the test cell while hot, ensuring that any surfaces that will be wetted by the liquid are not touched with bare hands.

7.2 Storage of cell

When the cell is not in use, it is recommended that it remains filled with either a clean sample of the insulating liquid normally used, or, where different liquids are tested, with a solvent which has no detrimental effect on the cell.

Cells not in regular use shall be cleaned, dried, assembled and stored in a dry and dust free cabinet.

Alternatively, follow the manufacturer's recommendations.

8 Sampling

Insulating liquids for use in these tests shall be sampled in accordance with the method described in IEC 60475.

Samples shall be stored and transported in their original containers and shielded from light.

9 Preparation of samples

Unless specifically required by the specification relevant to the liquid under test, no treatment, such as filtration, drying, etc., shall be applied.

If it is necessary to preheat the sample, this shall be done whenever possible in the original sample container after removal of sufficient samples for other tests. Sufficient space should be left for the expansion of the liquid to avoid breakage of the container.

Where samples have to be transferred to other containers these shall be covered glass beakers or glass-stoppered Erlenmeyer flasks cleaned at least to the standard of Clause 7.

If the test has to be carried out at ambient temperature, the original container shall be allowed to stand in the room in which the test is to be made until the sample attains ambient temperature. Where tests are to be carried out at an elevated temperature and the sample cannot be heated in the test cell, the sample container or the auxiliary receptacles shall be stoppered, ensuring there is an appropriate volume in the containers to allow for expansion of the liquid. The containers are then heated in an oven to a temperature 5 °C to 10 °C above the required test temperature.

With liquids that are susceptible to oxidation, the heating period shall not exceed 1 h.

If the liquid has to be heated in a separate oven, it may be found desirable to restrict the use of an oven to a particular class of liquid to prevent contamination effects.

In order that representative specimens may be obtained, the sample is homogenized by tilting the container and gently swirling the liquid several times immediately prior to taking a sample for test.

Wipe the mouth of the container with a clean lint-free cloth and pour off a small portion of the liquid to wash the external surface.

10 Conditioning and filling the test cell

10.1 Cell conditioning

After the electrodes have been cleaned and dried, take care not to touch their surfaces with bare hands. Pay attention also to the cleanliness of surfaces on which cell components are placed. Do not expose them to contamination from vapours or dust.

To eliminate any influence of the cell cleaning procedures on subsequent tests, it is essential to pre-condition a dry cleaned cell with two cell fillings of the next liquid to be tested. High viscosity liquids may need a longer pre-conditioning time.

10.2 Filling the cell

Rinse the cell three times with a portion of the oil sample, drain and discard the liquid. If the inner electrode(s) need to be removed to perform this, avoid resting them on any surface and avoid the collection of dust particles on wetted surfaces of the cell.

Refill the cell, taking care to avoid entrapment of air bubbles. Bring the cell and contents to the required test temperature. The time required to reach the test temperature will depend on the heating method and may typically vary from 10 min to 60 min. Measurement must be started within 10 min of reaching a temperature within ± 1 °C of the required test temperature

Great care should be taken to avoid contact of the liquid or the cell parts with any source of contamination.

It may be found desirable to restrict the use of a cell to a particular class of liquid. Contaminants that are benign in one liquid could be active in another with the possibility of transfer of the contaminants.

Ensure the environmental atmosphere is, as far as possible, free of any vapours or gases liable to affect the quality of the liquid.

11 Test temperature

These test methods are suitable for testing insulating liquids over a wide range of temperatures. Tests should be carried out at 90 °C unless otherwise specified by the appropriate specification for the particular liquid application.

Temperature measurement shall be capable of resolving to within 0,25 °C.

12 Measurement of dissipation factor ($\tan \delta$)

12.1 Test voltage

The a.c. voltage shall be such as to subject the liquid to an electrical stress between 0,03 kV/mm and 1 kV/mm. The voltage shall be sinusoidal at a frequency between 40 Hz and 62 Hz.

NOTE Generally, within the above frequency range, conversion of results from one frequency to another can be made using the formula:

$$\tan \delta_{f1} = (\tan \delta_{f2}) \frac{f_2}{f_1} \quad (2)$$

12.2 Measurement

Where cells are not automatically heated, carry out the dissipation factor measurement within 10 min of reaching a temperature within ± 1 °C of the required test temperature. Apply voltage only during the measurement. On completing the initial measurements (including permittivity and resistivity if required), drain the cell. Refill with a second specimen of the sample following the same procedure and exercising the same precautions used in making the first filling, but omitting rinsing. Repeat the measurement. The two values of $\tan \delta$ should not differ from each other by more than 0,0001 plus 25 % of the higher of the two values.

NOTE Requirement for a repeat measurement should be qualified for only low values of $\tan \delta$. Routine tests do not need to repeat measurements.

If this requirement is not met, continue with further fillings until two consecutive values for $\tan \delta$ are obtained which agree to within 0,0001 plus 25 % of the higher value of the two values being compared. These shall be accepted as valid measurements.

12.3 Report

Report the dissipation factor ($\tan \delta$) of the sample as the mean of the two valid measurements.

The report shall also include:

- a) the electrical stress;
- b) the frequency of applied voltage;
- c) the temperature of test.

13 Measurement of relative permittivity

13.1 Measurement

Measure the capacitance of the clean test cell, first with dry air as the dielectric and then after filling with a liquid of known relative permittivity ϵ_n . Calculate the "electrode constant" C_e and the correction capacitance C_g from:

$$C_e = \frac{C_n - C_a}{\epsilon_n - 1} \quad (3)$$

$$C_g = C_a - C_e \quad (4)$$

where

C_n is the capacitance of the cell filled with the calibration liquid having the relative permittivity ϵ_n ;

C_a is the capacitance of the cell with air as the dielectric.

Measure the capacitance C_x of the cell filled with the liquid under test and calculate the relative permittivity ε_x from

$$\varepsilon_x = \frac{C_x - C_g}{C_e} \quad (5)$$

Repeat the test until two consecutive measurements differ by no more than 5 % of the higher value. These shall be accepted as the valid measurement.

NOTE 1 Maximum accuracy is obtained if values C_a , C_n and ε_n are known at the temperature for which the value C_x is determined.

NOTE 2 When well designed, previously checked three-terminal cells are used or where lower accuracy is acceptable, the term C_g may be neglected, and the relative permittivity may be calculated from the simplified formula:

$$\varepsilon_x = \frac{C_x}{C_a} \quad (6)$$

13.2 Report

Report the relative permittivity of the sample as the mean of the valid measurements.

The report shall also include:

- a) the type of cell used and its capacitance with air as the dielectric;
- b) the electrical stress;
- c) the frequency of applied voltage;
- d) the temperature of test.

14 Measurement of d.c. resistivity

14.1 Test voltage

The d.c. test voltage shall be such as to subject the liquid to an electrical stress of 250 V/mm unless otherwise specified.

14.2 Time of electrification

The conventional arbitrary time of electrification is (60 ± 2) s. Variation in the time of electrification can result in appreciable variation in the test results.

14.3 Measurement

If the dissipation factor has been measured on the test specimen, the electrodes should be short-circuited for 60 s, and resistivity measurement started immediately afterwards.

If only resistivity is to be measured, start the determination as quickly as possible, and not more than 10 min after achieving a temperature within ± 1 °C of the required test temperature

Establish electrical connections to the measuring apparatus and to the voltage supply so that the inner electrode of the cell is connected to earth. Apply d.c. voltage to the outer electrode and, at the end of the electrification time, record the current and voltage readings.

NOTE 1 Alternatively, instruments reading resistance may be used, provided the other requirements (for example, a stress of 250 V/mm) are complied with.

Short-circuit the cell electrodes for a period of 5 min.

Discard the liquid in the cell, pour in a second specimen from the sample and repeat the measurements.

Calculate the resistivity in ohmmetres by means of:

$$\rho = K \frac{U}{I} \quad (7)$$

where

U is the reading of the test voltage, in volts;

I is the reading of the current, in amperes;

K is the cell constant, in metres.

The cell constant K is calculated from the capacitance according to:

$$K \text{ (m)} = 0,113 \times \text{capacitance (pF) of the empty cell}$$

NOTE 2 0,113 is 10^{-12} times the reciprocal of the permittivity of free space.

Direct reading apparatus that performs the calculation automatically may be used.

The measurements on two consecutive fillings shall not differ from each other by more than 35 % of the highest of the values. If this requirement is not met, continue with further fillings until values of resistivity from consecutive samples agree to within 35 % of the highest values.

These shall be accepted as valid measurements.

NOTE 3 A second measurement on each filling, made with the polarity of the applied voltage reversed, may provide information on the cleanliness of the cell and other phenomena. In order to avoid erroneous results, care is necessary with certain electronic instruments that do not have a switch for such reversal.

14.4 Report

Report resistivity of the sample as the mean of the valid measurements.

The report should also include:

- a) the electrical stress;
- b) the time of electrification;
- c) the temperature of test.

Annex A (informative)

Example of an alternative procedure for cleaning the test cell – Ultrasonic procedure

Dismantle the cell completely.

Wash all the component parts thoroughly with two portions of solvent (see Clause 6).

Immerse all parts of the cell, excluding any glass and rubber 'O' rings, in the solvent in a suitable ultrasonic cleaning bath for approximately 10 min.

Remove the cell parts from the bath and wash with clean solvent.

Allow solvent to evaporate in a dust free environment. To ensure complete evaporation, place cell parts in an oven heated at 105 °C to 110 °C for no longer than 120 min.

Reassemble the test cell while hot, ensuring that any surfaces that will be wetted by the liquid are not touched with bare hands.

Annex B
(informative)

Example of a simplified cleaning procedure for a test cell

Dismantle the cell completely, wherever possible.

Wash all the component parts thoroughly with two portions of solvent (see Clause 6).

Rinse all parts first with acetone and then with hot tap water followed by several rinsings of distilled water.

Thoroughly dry the component parts in an oven heated at 105 °C to 110 °C for no longer than 120 min because certain materials may deteriorate. Drying time depends on the design of the cell but 60 min to 120 min is usually satisfactory to remove any moisture.

Annex C (informative)

Alternative procedures for routine testing of dielectric dissipation factor and resistivity of insulating liquids

C.1 General

These simplified procedures are of value when testing a group of samples to ascertain whether the dissipation factor and resistivity of hydrocarbon and other liquids in service in electrical equipment, as well as of unused insulating liquids, are worse than certain specified values.

The test methods described in this annex are less precise than those previously described but they allow for a greater rapidity of measurement with a level of accuracy still quite acceptable.

C.2 Test cell

Test cells modified to allow the replacement of the liquid without opening the cell may also be used.

It may be found desirable to restrict the use of one cell to a particular class of liquid.

C.3 Test chamber

A forced draught air oven, a heating jacket or an oil- (or glycerol-) filled bath able to maintain the test cell at a sufficiently uniform temperature so that the required test temperature and the temperature of the inner electrode agree within 2 °C. The use of a hot plate is less satisfactory because variations of temperature throughout the cell may lead to unreliable results.

C.4 Test temperature

Measurements may be made when test specimens are within ± 2 °C of the desired temperature.

C.5 Cleaning the test cell

Where the cleaning procedures described in Clause 7 cannot be used, it is imperative that each laboratory evolves a good cleaning procedure for the cell used so that results obtained are repeatable and reasonably consistent with those obtained in accordance with the more elaborate procedure described in Clause 7.

The precautions on choice of solvents in Clause 6 should equally be followed.

The following procedure will often suffice for testing hydrocarbon liquids:

- Dismantle the cell completely, whenever possible.
- Wash all the component parts thoroughly with two portions of solvent (see Clause 6).
- Rinse all parts first with acetone and then with hot tap water followed by several rinsings with distilled water.
- Dry the component parts thoroughly in an oven at 105 °C to 110 °C for no longer than 90 min. Actual drying time depends on the design of the cell but 60 min to 90 min is usually satisfactory to remove any moisture.

When a number of samples of the same type of unused liquid have to be tested consecutively, the same test cell may be used without intermediate cleaning, provided that the value of the property for the sample previously tested is better than the specified value. If the value of the last sample tested is worse than the specified value, the test cell shall be cleaned before being used for further tests.

C.6 Preparation of specimens and filling the test cell

Samples should be stored and handled by following the precautions outlined in Clause 9.

Specimens of unused liquids shall be prepared, preheated and poured into the cell as described in Clauses 9 and 10. Preheating on a hot plate is permissible but the sample shall be continuously stirred to avoid local overheating.

An alternative procedure for low-viscosity hydrocarbon liquids, and particularly mineral oil, consists in filling the cool cell with an oil specimen at room temperature and placing it in the heating chamber maintained at the desired temperature. The heating rate shall allow the liquid in the cell to attain the test temperature within 1 h after heating has been started.

Test cells continuously used without intermediate cleaning between tests on different samples should always be flushed with a volume of the next sample corresponding to at least three fillings of the cell.

Aged hydrocarbon oils may sometimes require special precautions to avoid further oxidation during heating or retention at a high temperature.

Information about the effect of suspended foreign matter can be obtained by testing before and after filtration through a fritted glass filter of porosity grade 4.

C.7 Test voltage

Dissipation factor tests should be made with a stress in the range 0,03 kV/mm to 1 kV/mm. The actual stress shall not be so high as to cause secondary phenomena at the electrodes.

The d.c. test voltage chosen for resistivity measurements shall be such as to subject the liquid to an electrical stress of 50 V/mm to 250 V/mm.

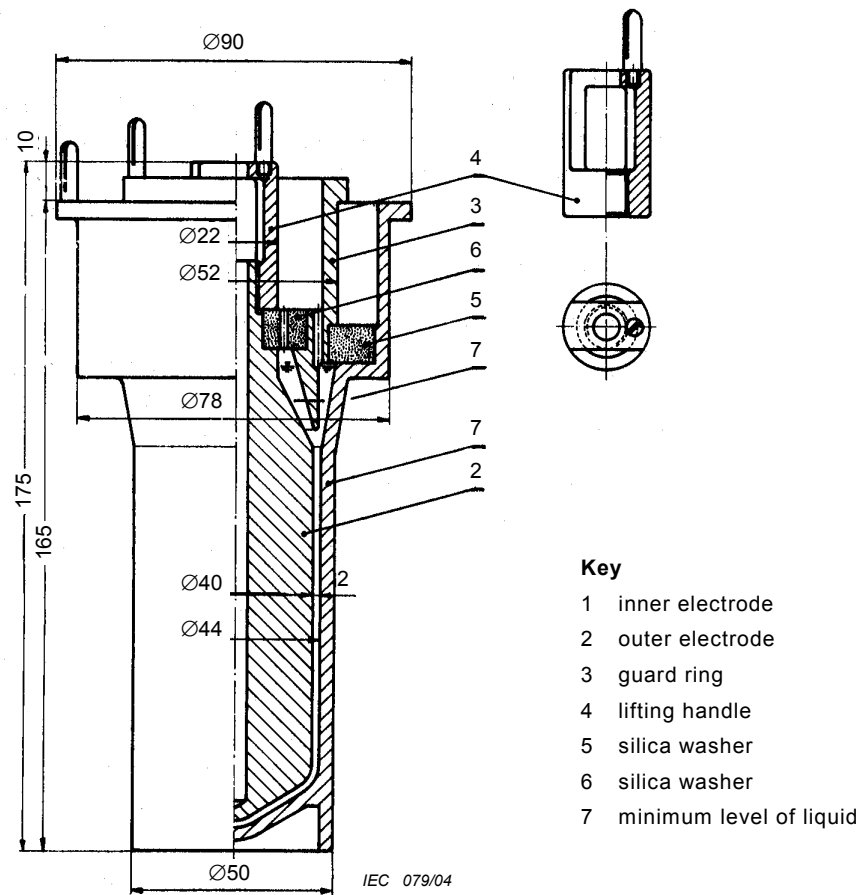
C.8 Measurement

When the liquid is not heated in the cell, the reading should be made after a standing period in the cell of 10 min to 15 min and when the temperature of the inner electrode is within ± 2 °C of the prescribed temperature.

If heating is carried out in the cell, the liquid should reach the test temperature within 1 h (the tolerance of 2 °C still applies) and the measurement then made.

When a.c. tests are required these shall always be made before applying d.c. voltage.

A single specimen may be tested for each sample.

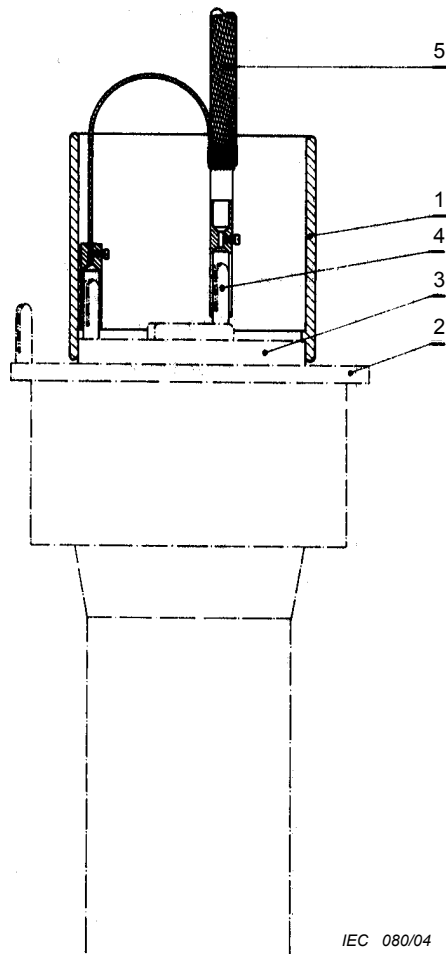


Volume of liquid is approximately 45 cm³.

All surfaces in contact with liquid shall have a smooth finish.

Dimensions in millimetres

Figure 1 – Example of a three-terminal cell for measurements on liquids



Key

- 1 removable screening cap (stainless steel)
- 2 outer electrode
- 3 guard ring
- 4 inner electrode ring
- 5 screened cable

IEC 080/04

Figure 2 – Example of screening for the cell of Figure 1

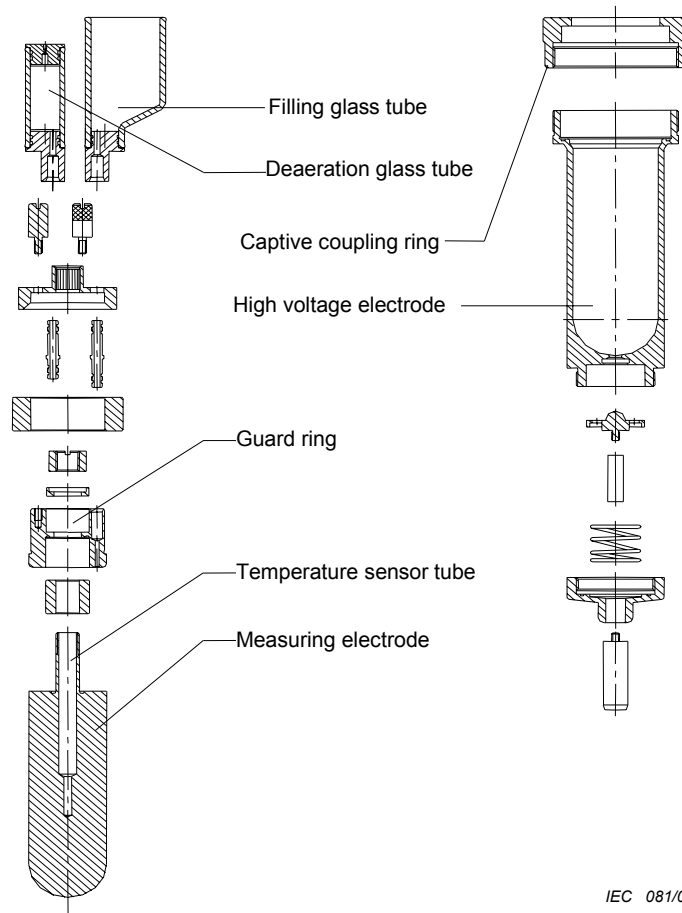
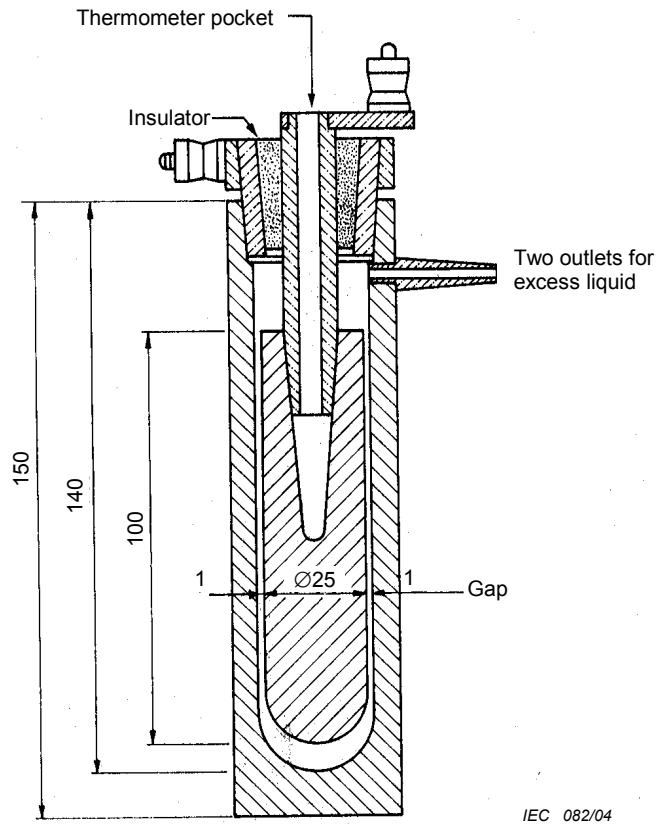


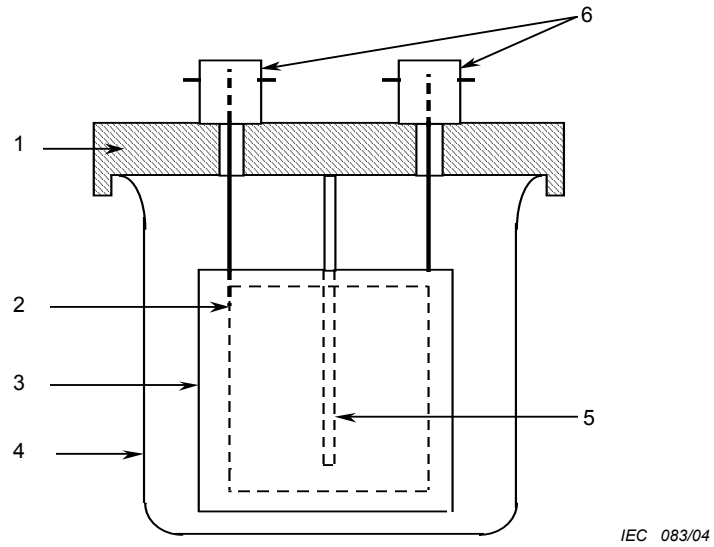
Figure 3 – Example of assembling drawing of cell



Quantity of liquid required to fill cell: 15 cm³ approximately.

Dimensions in millimetres

Figure 4 – Example of a two-terminal cell for measurements in liquids



Key

- 1 cover
- 2 inner electrode
- 3 outer electrode
- 4 stainless steel vessel
- 5 sheath for temperature measurement
- 6 BNC plugs for electrical connection

Figure 5 – Example of a test cell designed for low-loss dielectric liquids

Annex ZA (normative)

Normative references to international publications with their corresponding European publications

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.

NOTE Where an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN/HD</u>	<u>Year</u>
IEC 60093	- ¹⁾	Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials	HD 429 S1	1983 ²⁾
IEC 60250	- ¹⁾	Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths	-	-
IEC 60475	- ¹⁾	Method of sampling liquid dielectrics	-	-
IEC 61620	- ¹⁾	Insulating liquids - Determination of the dielectric dissipation factor by measurement of the conductance and capacitance - Test method	EN 61620	1999 ²⁾

1) Undated reference.

2) Valid edition at date of issue.

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001. Email: orders@bsi-global.com. Standards are also available from the BSI website at <http://www.bsi-global.com>.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: +44 (0)20 8996 7111. Fax: +44 (0)20 8996 7048. Email: info@bsi-global.com.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002. Fax: +44 (0)20 8996 7001. Email: membership@bsi-global.com.

Information regarding online access to British Standards via British Standards Online can be found at <http://www.bsi-global.com/bsonline>.

Further information about BSI is available on the BSI website at <http://www.bsi-global.com>.

Copyright

Copyright subsists in all BSI publications. BSI also holds the copyright, in the UK, of the publications of the international standardization bodies. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI.

This does not preclude the free use, in the course of implementing the standard, of necessary details such as symbols, and size, type or grade designations. If these details are to be used for any other purpose than implementation then the prior written permission of BSI must be obtained.

Details and advice can be obtained from the Copyright & Licensing Manager. Tel: +44 (0)20 8996 7070. Fax: +44 (0)20 8996 7553. Email: copyright@bsi-global.com.