IEC 62961

Edition 1.0 2018-09

INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

Insulating liquids – Test methods for the determination of interfacial tension of insulating liquids – Determination with the ring method

Isolants liquides – Méthodes d'essai pour la détermination de la tension interfaciale des isolants liquides – Détermination par la méthode à l'anneau

IEC 62961:2018-09(en-fr)

IEC Central Office
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
info@iec.ch
www.iec.ch

About the IEC
The International Electrotechnical Commission (IEC) is the leading global organization that prepares and publishes International Standards for all electrical, electronic and related technologies.

About IEC publications
The technical content of IEC publications is kept under constant review by the IEC. Please make sure that you have the latest edition, a corrigenda or an amendment might have been published.

IEC Catalogue - webstore.iec.ch/catalogue
The stand-alone application for consulting the entire bibliographical information on IEC International Standards, Technical Specifications, Technical Reports and other documents. Available for PC, Mac OS, Android Tablets and iPad.

IEC publications search - webstore.iec.ch/advsearchform
The advanced search enables to find IEC publications by a variety of criteria (reference number, text, technical committee,…). It also gives information on projects, replaced and withdrawn publications.

IEC Just Published - webstore.iec.ch/justpublished
Stay up to date on all new IEC publications. Just Published details all new publications released. Available online and also once a month by email.

Electropedia - www.electropedia.org
The world's leading online dictionary of electronic and electrical terms containing 21 000 terms and definitions in English and French, with equivalent terms in 16 additional languages. Also known as the International Electrotechnical Vocabulary (IEV) online.

IEC Glossary - std.iec.ch/glossary
67 000 electrotechnical terminology entries in English and French extracted from the Terms and Definitions clause of IEC publications issued since 2002. Some entries have been collected from earlier publications of IEC TC 37, 77, 86 and CISPR.

IEC Customer Service Centre - webstore.iec.ch/csc
If you wish to give us your feedback on this publication or need further assistance, please contact the Customer Service Centre: sales@iec.ch.

A propos de l'IEC
La Commission Electrotechnique Internationale (IEC) est la première organisation mondiale qui élabore et publie des Normes internationales pour tout ce qui a trait à l'électricité, à l'électronique et aux technologies apparentées.

A propos des publications IEC
Le contenu technique des publications IEC est constamment revu. Veuillez vous assurer que vous possédez l'édition la plus récente, un corrigendum ou amendement peut avoir été publié.

Catalogue IEC - webstore.iec.ch/catalogue
Application autonome pour consulter tous les renseignements bibliographiques sur les Normes internationales, Spécifications techniques, Rapports techniques et autres documents de l'IEC. Disponible pour PC, Mac OS, tablettes Android et iPad.

Recherche de publications IEC - webstore.iec.ch/advsearchform
La recherche avancée permet de trouver des publications IEC en utilisant différents critères (numéro de référence, texte, comité d'études,…). Elle donne aussi des informations sur les projets et les publications remplacées ou retirées.

IEC Just Published - webstore.iec.ch/justpublished
Restez informé sur les nouvelles publications IEC. Just Published détaille les nouvelles publications parues. Disponible en ligne et aussi une fois par mois par email.

Electropedia - www.electropedia.org
Le premier dictionnaire en ligne de termes électroniques et électriques. Il contient 21 000 termes et définitions en anglais et en français, ainsi que les termes équivalents dans 16 langues additionnelles. Egalement appelé Vocabulaire Electrotechnique International (IEV) en ligne.

Glossaire IEC - std.iec.ch/glossary
67 000 entrées terminologiques électrotechniques, en anglais et en français, extraites des articles Termes et Définitions des publications IEC parues depuis 2002. Plus certaines entrées antérieures extraites des publications des CE 37, 77, 86 et CISPR de l'IEC.

Service Clients - webstore.iec.ch/csc
Si vous désirez nous donner des commentaires sur cette publication ou si vous avez des questions contactez-nous: sales@iec.ch.

IEC 62961

Edition 1.0 2018-09

INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

Insulating liquids – Test methods for the determination of interfacial tension of insulating liquids – Determination with the ring method

Isolants liquides – Méthodes d'essai pour la détermination de la tension interfaciale des isolants liquides – Détermination par la méthode à l'anneau

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

COMMISSION
ELECTROTECHNIQUE
INTERNATIONALE

ICS 29.040.10 ISBN 978-2-8322-6037-1

CONTENTS

INTERNATIONAL ELECTROTECHNICAL COMMISSION

____

INSULATING LIQUIDS – TEST METHODS FOR THE DETERMINATION OF INTERFACIAL TENSION OF INSULATING LIQUIDS – DETERMINATION WITH THE RING METHOD

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.

2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.

3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.

4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.

5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.

6) All users should ensure that they have the latest edition of this publication.

7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.

8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.

9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

International Standard IEC 62961 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
10/1062/FDIS	10/1066/RVD

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

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

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

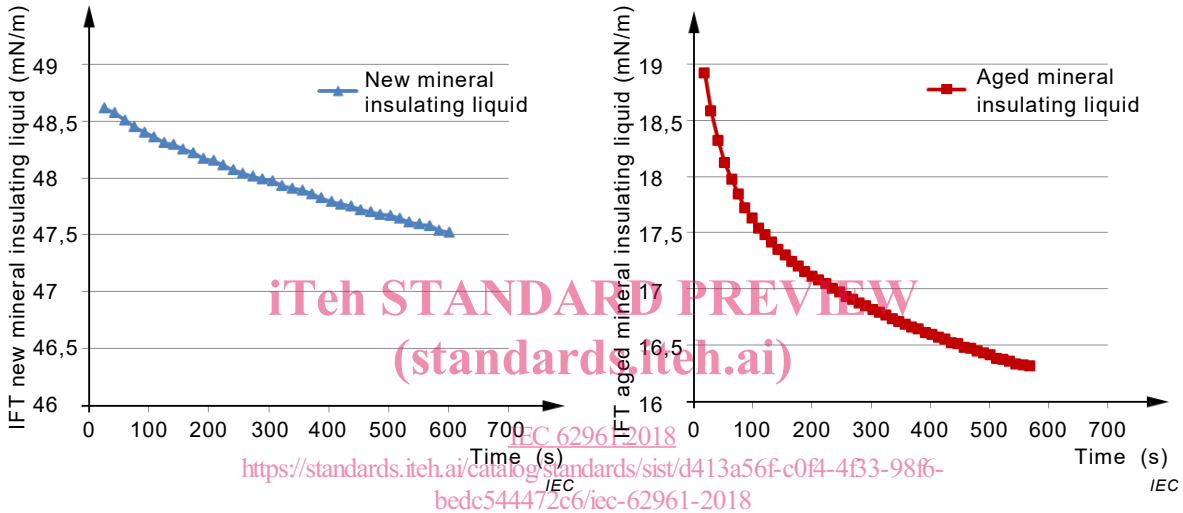
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

Interfacial tension (IFT) of insulating liquid against water has been used for a long time as a criterion for ageing evaluation. Statistical values that are used as orientation values and for their interpretation have been published in IEC 60422 [1] [1].

The interfacial tension of insulating liquids changes with time depending on the type and nature of the ageing products. This process is more pronounced with aged than with new insulating liquids. It is well known that the interfacial tension of insulating liquids depends on the interfacial concentration of the surface active amphiphilic aged products at the time of measuring (dynamic interfacial tension), see Figure 1. The adsorption procedures, and thus the attaining of a state of equilibrium, can take several minutes or even hours. With the so-called static measuring methods – e.g. the Du Noüy ring [2]– measurements are repeated on the same sample surface until no further change occurs.



a) Typical development of interfacial tension values of a new inhibited mineral insulating liquid

b) Typical development of interfacial tension values of a service aged mineral insulating liquid

Figure 1 – Typical development of interfacial tension values of new and service aged mineral insulating liquids

1 Numbers in square brackets refer to the Bibliography.

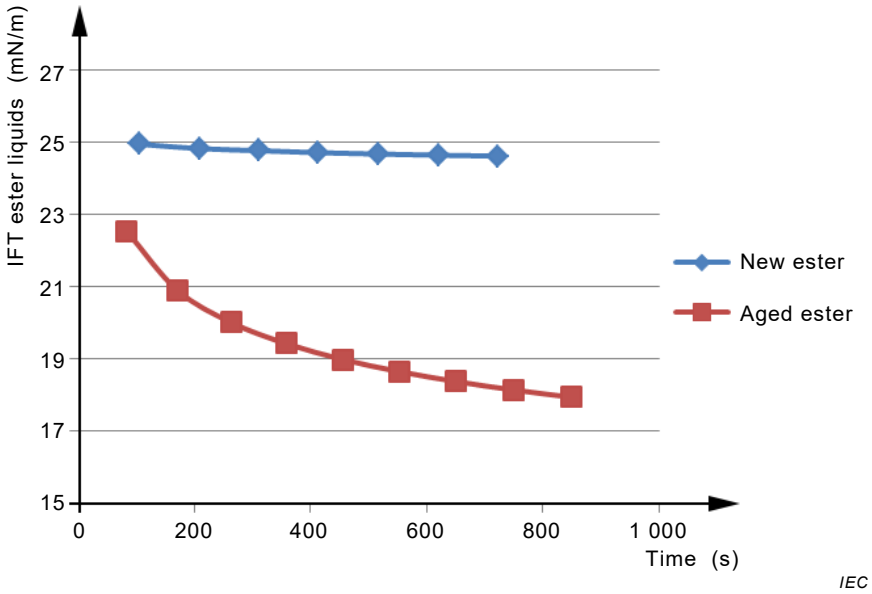


Figure 2 – Typical development of interfacial tension values of a new and a service aged ester insulating liquid

The interfacial tension of insulating liquids measured by the existing method ASTM D971 [3], working in non-equilibrium mode, provides only a single value within quite a short time (60 s) and hence might be quite different from the static interfacial value, particularly in the case of aged insulating liquids. In addition, the error of the time measurement might become a more important aspect than the performance of the measurement itself. These weaknesses of ASTM D971 could be generally compensated by replacing it with EN 14210 [4]. However, for the practical work in the laboratory, the requirement of repeating tests until "static" conditions are obtained can increase the test time dramatically.

The scope of this document is to find a compromise between the less accurate but fast ASTM D971 method and the precise, but time consuming EN 14210 procedure. Experience of the round robin tests shows clearly that the slope of the time-dependent interfacial tension curve decreases significantly over a period of 180 s in the case of both mineral insulating liquids (Figure 1 a), Figure 1 b)) and insulating synthetic and natural esters (Figure 2). A measurement is carried out after a surface age of approximately 180 s in order to obtain a value that provides a more realistic expression of the real interfacial tension, and that is less sensitive to the timing of the measurement taken, and does not overly increase the test time.

The proposed surface age of 180 s allows the distinction between differently aged ester liquids, which is not possible with ASTM D971.

The drop volume method for the determination of interfacial tension can deliver similar results as the ring method if adapted concerning the surface age. This method is described in Annex A.

Experience and results of round robin tests have shown that the deviation of tests repeated after 10 min is less than 1 mN/m per min. Such tests can be necessary in case of further comparative investigations of aged mineral and ester insulating liquids, and are described in Annex B.

INSULATING LIQUIDS – TEST METHODS FOR THE DETERMINATION OF INTERFACIAL TENSION OF INSULATING LIQUIDS – DETERMINATION WITH THE RING METHOD

1 Scope

This document establishes the measurement of the interfacial tension between insulating liquid and water by means of the Du Noüy ring method close to equilibrium conditions. In order to obtain a value that provides a realistic expression of the real interfacial tension, a measurement after a surface age of approximately 180 s is recorded.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 862, *Surface active agents – Vocabulary*

ISO 3675, *Crude petroleum and liquid petroleum products – Laboratory determination of density – Hydrometer method*

ISO 12185, *Crude petroleum and petroleum products – Determination of density – Oscillating U-tube method*

EN 14370, *Surface active agents – Determination of surface tension*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 862 and the following apply.

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

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

3.1
interfacial tension
tension at the interface between two phases

Note 1 to entry: The SI unit of interfacial tension is the Newton per metre (N/m). In practice, the submultiple millinewton per metre (mN/m) is used.

4 Principle

The maximum force, F, necessary to pull or to force a ring of perimeter πD out of the interface between insulating liquid and water in the direction of the insulating liquid is measured. The interfacial tension, σ, is obtained by calculation, where the following approximate equation (1) serves as the base:

$$\sigma = \frac{F_{\max}}{2\pi D} f \tag{1}$$

where

σ is the interfacial tension expressed as mN/m;

$F_{\max}$ is the maximum force exerted on the ring when pulled out of the liquid, in mN;

D is the mean diameter of the ring, in m;

f is a correction factor, taking into account that the measured maximum force includes the additional volume of liquid extracted together with the ring because of the finite diameter of the wire and of the lamella overlap inside the ring immediately prior to detachment. Extrapolation formulae have been reported by Zuidema and Waters [5] and others.

For the ring dimensions valid for this document and described in EN 14370, the following simplified correction factor (2) is commonly used for interfacial tension tests in the range from 4 mN/m to 50 mN/m:

$$f = 0{,}725 + \sqrt{4{,}014 \times 10^{-4} \times \frac{\gamma}{\Delta\rho} + 0{,}012\ 87} \tag{2}$$

where

f is the correction factor according to Zuidema and Waters [5];

γ is the interfacial tension without correction in mN/m;

$\Delta\rho$ is the difference in the densities between water and insulating liquid at the measuring temperature in g/cm^3. Density shall be measured in accordance with ISO 12185 (reference method), but ISO 3675 is accepted as well.

In automatic tensiometers with a built-in evaluation unit, software may perform the necessary corrections without a direct report of the measured force.

To obtain exact values for surface or interfacial tension, it is necessary to measure the maximum force on pulling the ring out of the liquid. Because of the great risk of detachment in the case of the manual apparatus, extremely smooth manipulation is necessary since the value obtained immediately prior to detachment of the film is not identical to the maximum value. Automatic tensiometers can determine the maximum value electronically and reverse the platform movement promptly prior to detachment of the film. This makes it possible to obtain reliable, accurate time-consistent serial measurements without tearing the film.

5 Apparatus

5.1 Tensiometer

The tensiometer shall be designed for a ring and shall consist mainly of two parts:

- support for the sample vessel in the form of a small horizontal platform which can be moved up and down;
- apparatus for measuring the force exerted on the ring; the uncertainty of measurement shall not exceed ± 10^{-6} N, which corresponds to a maximum error of ± 0,1 mg weight measurement.

Instead of a torsion balance as stated in ASTM D971, a lever balance or an electronic balance (laboratory, analytical or microbalance) can be used. To obtain higher efficiency and reproducibility, it is recommended to use an automatic tensiometer incorporating a balance, motor driven platform and evaluation unit.

5.2 Ring

The ring shall consist of a platinum/iridium wire with a thickness not greater than 0,4 mm and a mean circumference of 60 mm (for example: inner diameter 18,7 mm, outer diameter 19,5 mm). It shall be suspended horizontally and connected to the tensiometer.

The dimensions of the ring made of platinum-iridium alloy are specified in EN 14370 (Figure 3).

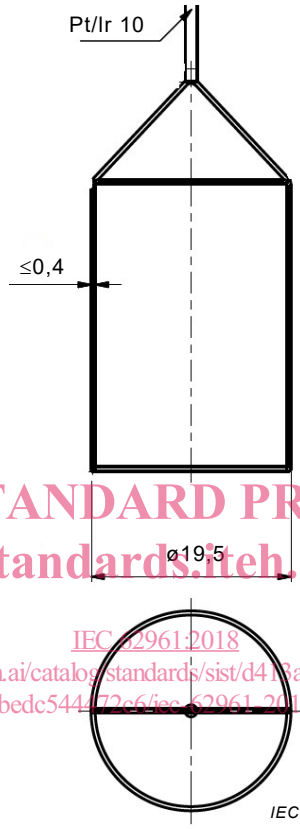


Figure 3 – Dimensions of platinum-iridium alloy ring in mm

5.3 Measuring vessel

Cylindrical glass vessel with a minimum diameter of 60 mm.

NOTE If a vessel with a diameter of less than 60 mm is used, wall effects can cause an error in the interfacial tension measurement result.

6 Preparation of apparatus

6.1 Cleaning of the measuring vessel

The vessels shall be dedicated to IFT measurement only. Rinsing with solvents with increasing polarity (such as heptane, acetone and/or 2-propanol, in this order) followed by a final rinse with hot tap water and afterwards with deionized water or bi-distilled water has been found suitable.

EXAMPLE An example of a step-by-step cleaning procedure is as follows:

- rinse three times with n-heptane (only if the vessel is being used after previous tests with insulating liquid), then afterwards with 2-propanol (also in the case of unused beakers and after water testing). Rinse with hot tap water and afterwards thoroughly with deionized water/bi-distilled water – see requirements for water in 6.3.

- a laboratory dishwasher with integrated deionized water with the required quality may be used if this provides the required cleanliness. Ensure that all washing agents are removed completely before drying.

6.2 Cleaning of the ring

The ring shall be cleaned with a suitable solvent and then by flame cleaning.

A typical cleaning procedure is as follows:

– rinse three times with n-heptane, and afterwards with deionized water;

– heat in the oxidizing flame for approximately 5 s in an ethanol or natural gas burner to red heat.

To prevent mechanical stress on the ring, twist it during this procedure.

6.3 Water used for the test

Bi-distilled or deionized water from a glass bottle with a surface tension of > 70 mN/m at maximum 25 °C (permissible range 18 °C to 25 °C) and with a known low conductivity (typically < 0,1 µS/cm). It is crucial that the water used not be from the tap and that it be free from any ions, since they may lead to a significant decrease of the measured interfacial tension. HPLC-grade water may be used, if suitable.

NOTE For pure water, the following relationship (Equation 3) between surface tension of water and temperature exists:

$$\frac{\Delta\sigma}{\Delta T} = -0{,}15 \tag{3}$$

where

$\Delta\sigma$ is the difference between the surface tension values in mN/m measured at two different temperatures;

ΔT is the difference between the two temperatures in °C.

7 Procedure

7.1 General

The measurement shall be done in the temperature range between 18 °C and 25 °C. The water and insulating liquid shall be at the same temperature. The density of the insulating liquid shall be determined at the temperature of measurement or can be calculated from a linear extrapolation of density from measurement at a standard temperature (e.g. 20 °C) to the temperature used for the IFT measurement. Round robin test results have shown that variations within this temperature range do not practically influence the test results.

The correction according to Zuidema and Waters [5] shall be used.

The correction of Zuidema and Waters [5] is the preferred correction, since it is the most widely used in existing standards (ASTM D971, EN 14210, EN 14370). Further corrections known are those of Harkins and Jordan [6], Fox and Chrisman [7], and Huh and Mason [8]. If another correction formula is used, this shall be noted in the test report.

7.2 Calibration and taring

Calibration shall be performed according to manufacturer instructions.

Tare the force sensor to zero with the attached cleaned and dry ring. Ensure that there is no contact with any sample or vessel wall.