SLOVENSKI STANDARD
SIST EN 60814:1999

01-januar-1999

Insulating liquids - Oil-impregnated paper and pressboard - Determination of water by automatic coulometric Karl Fischer titration (IEC 60814:1997)

Insulating liquids - Oil-impregnated paper and pressboard - Determination of water by automatic coulometric Karl Fischer titration

Isolierflüssigkeiten - Ölimprägniertes Papier und ölimprägnierter Presspan - Bestimmung von Wasser durch automatische coulometrische Karl-Fischer-Titration

Isolants liquides - Cartons et papiers imprégnés d'huile - Détermination de la teneur en eau par titrage coulométrique de Karl Fischer automatique

Ta slovenski standard je istoveten z: **EN 60814:1997**

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29.040.10	Izolacijska olja	Insulating oils

SIST EN 60814:1999 **en**

EUROPEAN STANDARD

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English version

Insulating liquids - Oil-impregnated paper and pressboard Determination of water by automatic coulometric Karl Fischer titration (IEC 60814:1997)

Isolants liquides - Cartons et papiers imprégnés d'huile - Détermination de la teneur en eau par titrage coulométrique de Karl Fischer automatique (CEI 60814:1997)

Isolierflüssigkeiten - Ölimprägniertes Papier und ölimprägnierter Presspan Bestimmung von Wasser durch automatische coulometrische Karl-Fischer-Titration (IEC 60814:1997)

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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.

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CENELEC

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

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Foreword

The text of document 10/406/FDIS, future edition 2 of IEC 60814, prepared by IEC TC 10, Fluids for electrotechnical applications, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 60814 on 1997-10-01.

This European Standard supersedes HD 487 S1:1987.

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) 1998-07-01
- latest date by which the national standards conflicting with the EN have to be withdrawn (dow) 1998-07-01

Annexes designated "normative" are part of the body of the standard.
Annexes designated "informative" are given for information only.
In this standard, annex ZA is normative and annex A is informative.
Annex ZA has been added by CENELEC.

Endorsement notice

The text of the International Standard IEC 60814:1997 was approved by CENELEC as a European Standard without any modification.

Annex ZA (normative)

Normative references to international publications with their corresponding European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. 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 (including amendments).

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

Publication	Year	Title	EN/HD	Year
IEC 60475	1974	Method of sampling liquid dielectrics	-	-
EN 60567	1992	Guide for the sampling of gases and of oil from oil-filled electrical equipment and for the analysis of free and dissolved gases	EN 60567	1992
ISO 595-1	1986	Reusable all-glass or metal-and-glass syringes for medical use Part 1: Dimensions	-	-
ISO 595-2	1987	Part 2: Design, performance requirements and tests	EN ISO 595-2	1994

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Isolants liquides – Cartons et papiers imprégnés d'huile – Détermination de la teneur en eau par titrage coulométrique de Karl Fischer automatique

Insulating liquids – Oil-impregnated paper and pressboard – Determination of water by automatic coulometric Karl Fischer titration

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

INSULATING LIQUIDS – OIL-IMPREGNATED PAPER AND PRESSBOARD – DETERMINATION OF WATER BY AUTOMATIC COULOMETRIC KARL FISCHER TITRATION

FOREWORD

1) The IEC (International Electrotechnical Commission) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of the 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, the IEC publishes International Standards. 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. The 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 the 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 National Committees.

3) The documents produced have the form of recommendations for international use and are published in the form of standards, technical reports or guides and they are accepted by the National Committees in that sense.

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

International Standard IEC 60814 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications.

This second edition of IEC 60814 cancels and replaces the first edition published in 1985 of which it constitutes a technical revision.

It also cancels IEC 60733, published in 1982.

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

FDIS	Report on voting
10/406/FDIS	10/422/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.

Annex A is for information only.

INSULATING LIQUIDS – OIL-IMPREGNATED PAPER AND PRESSBOARD – DETERMINATION OF WATER BY AUTOMATIC COULOMETRIC KARL FISCHER TITRATION

1 General

1.1 *Scope*

This International Standard describes methods for the determination of water in insulating liquids and in oil-impregnated cellulosic insulation with coulometrically generated Karl Fischer reagent.

The method in clause 2 is applicable to water concentrations above 2 mg/kg in liquids having viscosity of less than 100 mm^2/s at 40 °C.

The test method in clause 3, where water is extracted by means of a nitrogen stream, is the preferred method for insulating liquids of viscosity higher than 100 mm^2/s.

Clause 4 describes methods for the determination of water content in oil-impregnated paper and pressboard over the range 0,1 % to 20 % by mass.

1.2 *Normative references*

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All normative documents are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

IEC 60475: 1974, *Method of sampling liquid dielectrics*

IEC 60567: 1992, *Guide for the sampling of gases and of oil from oil-filled electrical equipment and for the analysis of free and dissolved gases*

ISO 595-1: 1986, *Reusable all-glass or metal-and-glass syringes for medical use – Part 1: Dimensions*

ISO 595-2: 1987, *Reusable all-glass or metal-and-glass syringes for medical use – Part 2: Design, performance requirements and tests*

2 Direct titration for low viscosity liquids

2.1 *Field of application*

This method is applicable to water concentrations above 2 mg/kg in liquids having viscosity up to 100 mm^2/s at 40 °C. The precision data given in 2.10 apply only to new liquids.

NOTES

1 For liquids in service, the accuracy of the method may be affected by the presence of contaminants and degradation products.

2 The method has been designed to be particularly suitable to hydrocarbon and ester liquids. With other liquids, particularly silicone fluids, methanol free reagents must be used.

2.2 *Chemistry*

The reactions occurring in a Karl Fischer titration are known to be complex, but are essentially of water with iodine, sulphur dioxide, an organic base and an alcohol in an organic solvent. The original Karl Fischer reagent used pyridine and methanol, and the reactions may be expressed as:

$$H_2O + I_2 + SO_2 + 3C_5H_5N \rightarrow 2C_5H_5N.HI + C_5H_5N.SO_3 \quad (1)$$

$$C_5H_5N.SO_3 + CH_3OH \rightarrow C_5H_5NH.SO_4.CH_3 \quad (2)$$

Other base-alcohol combinations are possible and may be necessary for titrations on some insulating liquids.

In coulometric Karl Fischer titration, the sample is mixed with a base/alcohol solution of iodide ion and sulphur dioxide. Iodine is generated electrolytically and reacts with water in a similar way to that shown in reactions (1) and (2). Iodine is generated in proportion to the quantity of electricity according to Faraday's law, as shown by the following reaction:

$$2\ I^- - 2\ e \rightarrow I_2 \quad (3)$$

One mole of iodine reacts with one mole of water stoichiometrically as shown in reactions (1) so that 1 mg of water is equivalent to 10,72 C (number of coulombs). Based on this principle it is possible to determine the amount of water directly from the quantity of electricity (number of coulombs) required for the electrolysis.

2.3 *Apparatus*

2.3.1 *Principle of operation*

The titration vessel has the configuration of an electrolysis cell with two compartments connected by a porous diaphragm. The anodic compartment contains the mixture of reagent-solvent and sample (anodic solution), the cathodic compartment (generator assembly) contains anhydrous reagent (cathodic solution). On both sides of the diaphragm are located the electrolysis electrodes.

NOTE – Titrators without the porous diaphragm may be used.

Iodine generated by the electrolysis, as shown in reaction (3), reacts with the water in a similar way to the Karl Fischer reactions (1) and (2). The end-point of the reaction is detected by a pair of platinum electrodes immersed in the anodic solution. At the end of the titration, excess iodine depolarizes the dual platinum electrodes, giving a change in the current/voltage ratio which is used to activate the end-point indicator and to stop the current integrator.

The current integrator integrates the current consumed during the electrolysis, calculates the water equivalent according to Faraday's law, and finally displays it in micrograms of water.

2.3.2 *Description of the apparatus*

Commercial coulometric Karl Fischer titrators use proprietary circuitry. The following description of one suitable form of apparatus is given for illustrative purposes only.

The block diagram shown in figure 1 illustrates the apparatus and includes the components detailed below.