

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

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 18, an inside back cover and a back cover. This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

This British Standard, having been prepared under the direction of the Electrotechnical Sector Board, was published under the authority of the Standards Board and comes into effect on 15 February 1998

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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.

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- latest date by which the national standards conflicting with the EN have to be withdrawn (dow) 1998-07-01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

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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 mm²/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 mm²/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 mm²/s at 40 °C. The precision data given in 2.10 apply only to new liquids.

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

NOTE 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:



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:



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.

2.3.2.1 Titration vessel assembly

An example of a suitable titration vessel assembly is shown in Figure 2. However, the changes in instrument technology are such that radically different designs may become available which comply with the technical requirements of this standard. The exemplified apparatus consists of:

- a flanged glass reaction vessel a) with sample injection plug b) and drain cock c) (optional);
- a polytetrafluoroethylene lid d), flanged to match the titration vessel, with three holes to receive the electrodes and drying tube;
- a generator assembly (combined electrolysis cell) e) consisting of a glass tube closed at its lower end by a diaphragm and equipped with platinum electrodes on each side of the diaphragm;

NOTE The diaphragm may consist of ion exchange membrane, fritted disc, ceramic filter or other system to prevent diffusion of both solutions, while allowing enough current for electrolysis.

- detector electrodes: dual platinum electrodes for measurement of potential or current f);
- a polytetrafluoroethylene coated stirrer bar g);
- drying tubes h) to protect the titration vessel and the generator assembly from atmospheric moisture;
- silicone rubber septa to seal the injection port. It is recommended that crosscuts should be made in the septa before use, to enable blunt, square-ended needles to be used for sample injection [see 2.4.2 d)]. Septa should be replaced as required to prevent air leakage as indicated by excessive instrument drift.

2.3.2.2 Detection circuit

DC constant voltage or a.c. constant current is supplied to the detector electrodes (dual platinum measuring electrodes) so that the end-point may be detected from the change of the polarized current or voltage.

2.3.2.3 Current regulator circuit

This circuit controls the electrolysis according to the signal from the detector circuit.

2.3.2.4 DC power supply

DC power supply for electrolysis.