

Method for

Determination of water in liquid dielectrics by automatic coulometric Karl Fischer titration

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- Association of Consulting Engineers
- ERA Technology Ltd
- Electricity Supply Industry in England and Wales
- Institute of Petroleum
- Institution of Electrical Engineers
- London Regional Transport

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

- British Lubricants Federation limited
- Electric Cable Makers Confederation
- Transmission and Distribution Association (BEAMA Limited)

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National foreword

This British Standard has been prepared under the direction of the General Electrotechnical Engineering Standards Committee. It is identical with IEC Publication 814:1985 “*Determination of water in insulating liquids by automatic coulometric Karl Fischer titration*”, published by the International Electrotechnical Commission (IEC).

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards. Wherever page numbers are quoted in connection with reference to figures, they are IEC page numbers.

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

Cross-references

International Standards^a Corresponding British Standards

IEC 567:1977	BS 5574:1978 <i>Guide for the sampling of gases and of oil from oil-filled electrical equipment and for the analysis of free and dissolved gases</i> (Identical)
IEC 733:1982	BS 6470:1984 <i>Method for determination of water in insulating oils, and in oil-impregnated paper and pressboard</i> (Identical)

^a Undated in the text.

The Technical Committee has reviewed the provisions of IEC 475:1974, to which reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard. A related British Standard is BS 5263:1975 “*Method of sampling liquid dielectrics*” which is stated in its foreword to be technically equivalent, but it enlarges upon the methods to be taken to ensure adequate cleaning of sampling equipment and containers and excludes the use of ether as a cleaning medium.

NOTE In clause 3.1.2, line 1, “of” should be read as “or”.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, 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.

1 Scope

This standard describes a method for the determination of water in unused or used insulating liquids with coulometrically generated Karl Fischer reagent. The method is applicable to water concentrations of 2 mg/kg to 100 mg/kg. The precision data given in Clause 10 apply only to new oils with viscosity less than 20 mm²/s at 40 °C (see note).

Appendix A contains two suitable methods for preparing the reagents which may be used in countries where it is difficult to buy prepared reagents.

This method should be supplementary to the classical Karl Fischer titration as described in IEC Publication 733: Determination of Water in Insulating Oils, and in Oil-impregnated Paper and Pressboard, and in case of dispute this latter method shall be used as a reference for arbitration.

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

2 Chemistry

The reactions occurring in a Karl Fischer titration are known to be complex but are essentially of water with iodine and sulphur dioxide and may be expressed as follows:



In coulometric Karl Fischer titration the sample is mixed with a pyridine-methanol solution of iodide ion and sulphur dioxide. Iodine is generated electrolytically and reacts with water as 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 reaction (1) so that 1 mg of water is equivalent to 10.72 C. 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.

3 Apparatus

This kind of apparatus uses patented circuitry, however the following description is of a general nature and applies to the majority of commercially existing apparatus¹⁾.

3.1 Description of the apparatus

The block diagram is illustrated in Figure 1, page 7, and includes the following components:

3.1.1 Titration vessel assembly

A suitable titration vessel assembly is shown in Figure 2, page 8, and consists of:

- Flanged glass reaction vessel (a), with sample injection plug (b) and drain cock (c).
 - Polytetrafluoroethylene lid (d), flanged to match the titration vessel, with three holes to receive the electrodes and drying tube.
 - 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 disk, ceramic filter or other system to prevent diffusion of both solutions while allowing enough current for electrolysis.
- Dual platinum electrodes for potential measurement (f).
 - Polytetrafluoroethylene coated stirring bar (g).
 - Drying tubes (h) to protect the titration vessel and the generator assembly from atmospheric moisture.

¹⁾ More information on the working of this kind of titrator can be obtained in the following papers:

- Meyer et al. "Analytical Chemistry" 1959, Vol. 31, pp. 215–219.
- Swensen et al. "Analytical Chemistry" 1963, Vol. 35, pp. 863–867.
- Bizot, "Bulletin de la Société chimique de France" 1967, n° 1, pp. 151–157.