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Petroleum products — Determination of water — Coulometric Karl Fischer titration method

Produits pétroliers — Dosage de l'eau — Méthode de titrage Karl Fischer par coulométrie

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

International Standard ISO 12937 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 6, *Bulk cargo transfer, accountability, inspection and reconciliation*.

Annex A forms a normative part of this International Standard. Annex B is for information only.

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Petroleum products — Determination of water — Coulometric Karl Fischer titration method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the direct determination of water in petroleum products boiling below 390 °C. It covers the mass fraction range 0,003 % (*m/m*) to 0,100 % (*m/m*). It is not applicable to products containing ketones or to residual fuel oils.

This International Standard may be applicable to lubricating base oils. However, the precision has not been established for these materials.

The precision given in clause 12 is based upon data obtained using dual-cell, dual-electrolyte systems.

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NOTE 1 A number of substances and classes of compounds associated with condensation or oxidation-reduction reactions interfere in the determination of water by Karl Fischer titration. In petroleum products, the most common interferences are hydrogen sulfide and mercaptan sulfur, however, mass fractions of these below 0,003 % (*m/m*) as sulfur will not cause significant interference over the range 0,003 % (*m/m*) to 0,100 % (*m/m*) water. Other organic sulfur compounds commonly present such as sulfides, disulphides and thiophenes, do not interfere.

NOTE 2 An alternative procedure is provided for information in annex B for the direct determination of water over the range 0,003 % *(V/V)* to 0,100 % *(V/V)* in petroleum products. The limitations under which this alternative volume measurement may be used are listed in annex B.

NOTE 3 For the purposes of this International Standard, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent the mass and volume fraction of a material respectively.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, 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. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

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3 Principle

A sample is visually inspected (see 6.2.1). If clear and bright, and free from both water droplets and particulate matter on swirling, a weighed portion is injected into the titration vessel of a coulometric Karl Fischer apparatus in which iodine for the Karl Fischer reaction is generated coulometrically at the anode. When all the water has been titrated, excess iodine is detected by an electrometric end-point detector and the titration is terminated. Based on the stoichiometry of the reaction, one mole of iodine reacts with one mole of water, thus the quantity of water is proportional to the total integrated current according to Faraday's Law.

If the sample is not clear and bright, or water droplets or particulate matter are observed on swirling, a portion of a solution of sodium dioctylsulfosuccinate is added prior to homogenizing with a mixer. A weighed portion is then treated as described above.

4 Chemicals and materials

4.1 Molecular sieve pellets, type 4A.

Activate in an oven at 200 °C to 250 °C for 4 h. Transfer immediately to a dry sealable bottle or desiccator and allow to cool.

4.2 Xylene, reagent grade.

Dry by adding approximately 100 g of activated molecular sieve (4.1) to 2 litres of xylene. Allow to stand overnight.

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4.3 Karl Fischer reagents

Use commercially available reagents that meet the performance requirements described in clause 8.

4.3.1 Anode electrolyte solution (anolyte)

Mix 6 parts by volume of commercial Karl Fischer anode solution with 4 parts by volume of dry xylene (4.2).

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NOTE Other proportions of Karl Fischer anode solution and xylene may be used, provided they meet the performance criteria of clause 8.

4.3.2 Cathode electrolyte solution (catholyte)

Use commercially available Karl Fischer cathode solution.

4.3.3 Single Karl Fischer solution, for use in place of dual electrolyte solutions (4.3.1 and 4.3.2) in cells with or without a diaphragm.

4.4 Sodium dioctylsulfosuccinate, reagent grade.

NOTE 1 Sodium dioctylsulfosuccinate is also sold under the names dioctyl ester of sodium sulfosuccinic acid and dioctyl sulfosuccinate sodium salt.

NOTE 2 Other anionic surfactants may be used in place of sodium dioctylsulfosuccinate, provided they meet the requirements of normative annex A.

4.4.1 Sodium dioctylsulfosuccinate solution

Dry sodium dioctylsulfosuccinate (4.4) in an oven at 105 °C to 110 °C for 4 h. Transfer immediately to a dry sealable bottle or desiccator and allow to cool. Once cool, dissolve 10 g of dried sodium dioctylsulfosuccinate in dry xylene (4.2) and make up to 100 ml with dry xylene. The mass fraction of water in this solution shall be less than 0,010 % (*m/m*) when checked by the procedure given in clause 9.

4.5 Water, conforming to grade 3 of ISO 3696.

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