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Dimethyl ether (DME) for fuels — Determination of water content — Karl Fischer titration method

Diméthylether (DME) pour carburants et combustibles — Détermination de la teneur en eau — Méthode par titrage Karl Fischer

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 4, *Classifications and specifications*.

Introduction

In general, large amounts of DME in international trade and domestic transportation can be executed using sea and/or various land transportations. Throughout loading and transportation, there is a risk of increasing the DME's water content.

DME is soluble in water and the amount of water contained in the DME gives significant detrimental influence when it is used as fuel.

Accordingly, water content in DME has to be analysed accurately using recognized procedures by the parties concerned.

In this International Standard, one of the most common practices to be applied to analysis of water content is standardized.

Dimethyl ether (DME) for fuels — Determination of water content — 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 procedure of test for the amount of water content in DME used as fuel by the Karl Fischer titration method. This procedure is applicable to determine the amount of water up to the value specified in ISO 16861.

This test method is intended for use with commercially available coulometric (or volumetric) Karl Fischer reagents and for the determination of water in DME additives, lube oils, base oils, automatic transmission fluids, hydrocarbon solvents, and other petroleum products. By proper choice of the sample size, this test method can be used to determine water from mg/kg (ppm) to percent level concentrations.

NOTE The precision of this method has been studied for a limited set of samples and content levels by a limited amount of labs. It allows establishment of a quality specification of DME but cannot be considered as a full precision determination in line with the usual statistical methodology as in ISO 4259.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 29945, *Refrigerated non-petroleum-based liquefied gaseous fuels — Dimethylether (DME) — Method of manual sampling onshore terminals*

3 Principle

A gaseous sample of DME is bubbled into the titration vessel of a coulometric (or volumetric) Karl Fischer apparatus. The titration is then performed until all of the water has been titrated, the end point is detected by an electrometric end point detector, and the titration is terminated. Based on the stoichiometry of the reaction, 1,0 mol of iodine reacts with 1,0 mol of water; thus, the quantity of water is proportional to the quantity of Karl Fisher reagent used.

4 Reagents and materials

4.1 Sample solvent, reagent grade.

Use methanol (anhydrous) with minimum purity 99,9 mass % and maximum water content 0,1 mass % (and preferably less than 0,05 mass %).

This water content could be achieved by dissolving 24 g of magnesium metal turnings in 200 ml of methanol (the reaction could be vigorous). When the reaction is completed, add 3 l of methanol. Reflux for 5 h. Distill directly into the container in which the 99,9 mass % methanol is to be kept. Vent the system through a drying tube during the distillation.

4.2 Coulometric Karl Fischer reagent.

Use standard, commercially available reagents for coulometric Karl Fischer titrations.

4.2.1 Anode solution.

Use a standard, commercially available anode Karl Fischer solution. A newly made solution should be used.

4.2.2 Cathode solution.

Use a standard, commercially available cathode Karl Fischer solution. A newly made solution should be used.

4.3 Volumetric Karl Fischer reagent.

Use standard, commercially available, pyridine-free Karl Fischer reagents for volumetric titrations (one or two components). Use as described in 4.1.

4.3.1 One component.

Use a commercial volumetric Karl Fischer reagent. Fresh Karl Fischer reagent shall be used (alternatively, the solution should be standardized or calibrated each time it is used).

4.3.2 Two components.

Use commercial volumetric Karl Fischer reagent. Mix the two reagents (part 1 and part 2) just before using. The solution should be standardized and calibrated as soon as possible.

4.3.3 Methanol (anhydrous).

4.4 Molecular sieve.

A ball- or cylindrical-shaped molecular sieve shall be used. All vented ends of the system shall be through drying tubes filled with this molecular sieve. Alternatively, anhydrous calcium chloride could be used.

4.5 Water.

Distilled water or water of equivalent purity shall be used.

5 Apparatus

5.1 Karl Fischer apparatus (coulometric or volumetric), using electrometric end point.

A number of automatic coulometric and volumetric Karl Fischer titration assemblies consisting of titration cell, platinum electrodes, magnetic stirrer, and a control unit are available on the market. Instructions for operation of these devices are provided by the manufacturers and are not described herein. A sample is introduced using a stainless steel needle for gas bubbling; the length can be about 200 mm and the diameter can be about 1 mm.

5.2 Pressure gas cylinder.

Samples are most easily added to the titration vessel by means of double-valve pressurized gas cylinders (size is up to 100 ml, tested pressure not less than 3,0 MPa) that the mass could be measurable. The sample should be delivered from the bottom of the cylinder.

5.3 Electronic balance.

The mass of the samples are determined by means of a top loading electronic balance with an accuracy of at least 1,0 mg and with capacity that covers the mass of double-valve pressurized gas cylinders filled with the sample. The sample mass is concluded by the difference between the cylinder mass before and after the test (after excluding the purge sample quantity).

6 Sampling and sample handling

6.1 Samples shall be taken as described in ISO 29945.

6.2 Test Specimen: the aliquot obtained from the laboratory sample for analysis by this test method. Once drawn, use the entire portion of the test specimen in the analysis.

6.3 Select a test specimen size from 10 g to 20 g.

7 Preparation of apparatus

7.1 Follow the manufacturer's directions for preparation and operation of the titration apparatus.

7.2 Seal all joints and connections to the vessel to prevent atmospheric moisture from entering the apparatus.

7.3 Add the sample solvent into the titration vessel to the level that is recommended by the manufacturer. Prepare the Karl Fischer reagent as described in 4.2 or 4.3.

For coulometric titration, add the Karl Fischer anode solution to the anode (outer) compartment. Add the solution to the level recommended by the manufacturer. Add the Karl Fischer cathode solution to the cathode (inner) compartment. Add the solution to a level 2 mm to 3 mm below the level of the solution in the anode compartment.

7.4 Turn on the apparatus and start the magnetic stirrer for a smooth stirring action. Allow the residual moisture in the titration vessel to be titrated until the end point is reached. Do not proceed beyond this stage until the background current (or background titration rate) is constant and less than the maximum recommended by the manufacturer of the instrument.

7.5 Install the needle to the double-valve gas cylinder. Stick the needle of the cylinder to the titration flask and fix the cylinder. The needle shall be rolled with tissue paper or threaded into a silicone rubber plate to prevent dew condensation water from mixing into the titration flask.

7.6 Open the liquid valve of the sample cylinder slowly and adjust the gas flow rate in the range of 0,5 g/min to 2,0 g/min. Be sure that the gas doesn't generate foam or overflow. The needle shall be below the liquid surface, with at least 3,0 cm to allow the water content in the bubbled gas to be extracted in the solvent.

7.7 Allow the residual moisture in the titration vessel to be titrated until the end point is reached again. The system now is ready for sample titration. Disconnect the sample cylinder and measure the mass and record as the "initial sample mass".

8 Calibration and standardization

8.1 Standardize the Karl Fischer reagent at least once daily.

