DRAFT INTERNATIONAL STANDARD ISO/DIS 17197

ISO/TC **28**/SC **4** Secretariat: **AFNOR**

Voting begins on **2013-04-08** Voting terminates on **2013-07-08**

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

ICS 71.080.60; 75.160.20

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Published in Switzerland

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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ISO 17197 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 4, *Classifications and specifications*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

Introduction

In general, large amount of DME in the international trade and domestic transportation may be executed using sea and/or various land transportations. Throughout the loading and transportation there may be a risk increasing water contents.

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

Accordingly, water contents in the DME shall 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 contents is standardized.

DME (Dimethyl ether) for fuel – 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 method to determine whether the amount of water contained in DME (Dimethyl ether) satisfies 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 may be used for the determination of water from mg/kg (ppm) to percent level concentrations

2 Normative references

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

ISO 760:1978, *Determination of water – Karl Fischer method (General method)*

ISO 29945: *Refrigerated non-petroleum-based liquefied gaseous fuels – Dimethyl ether (DME) – Method of manual sampling onshore terminals*

ISO 5725-2: Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard

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 mole of iodine reacts with 1,0 mole 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

Methanol (anhydrous)—Minimum purity 99,9 mass % and water content maximum 0,1 mass % (and preferable 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 litres of methanol. Reflux for 5 hours. Distil 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.

a) *Anode Solution*—Use standard commercially available anode Karl Fischer solution. Newly made solution should be used.

b) *Cathode Solution*—Use standard commercially available cathode Karl Fischer solution. 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 component).

a) *One Component*—Use commercial volumetric Karl Fischer reagent. Fresh Karl Fisher reagent must be used (alternatively, the solution should be standardized or calibrated each time used).

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

c) *Methanol (anhydrous)*

Use as described in 4.1.

4.4 Molecular Sieve

5Å—8 to 12 mesh, ball or cylindrical shaped Molecular Sieve could be used. All vented ends of the system should be through drying tubes filled with this molecular sieve. Alternatively, anhydrous calcium chloride could 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. Sample is introduced using a stainless steel *Needle* for gas bubbling, the length may be about 200 mm and the diameter may 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

Mass of the samples are determined by means of top loading electronic balance with accuracy at least 1,0 mg and with capacity that cover the mass of double-valve pressurized gas cylinders filled with the sample. The sample mass is concluded by difference between 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 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.