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Dimethyl ether (DME) for fuels — Determination of impurities — Gas chromatographic method

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National foreword

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Dimethyl ether (DME) for fuels — Determination of impurities — Gas chromatographic method

*Diméthylether (DME) pour carburants et combustibles —
Détermination des impuretés — Méthode par chromatographie en
phase gazeuse*



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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).

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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

Throughout the manufacturing process, impurities can generate in the dimethyl ether (DME). In addition, there is a possibility that DME becomes contaminated during loading and transportation by sea and/or various land transportations. Examples of such impurities include methanol, water, carbon dioxide, ethyl methyl ether, sulfur, residues, and so on.

Gas chromatography is recommended to analyse many kinds of vaporizable impurities.

Dimethyl ether (DME) for fuels — Determination of impurities — Gas chromatographic method

WARNING — The use of this International Standard can 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 methanol, CO, CO₂, methyl formate, ethyl methyl ether, and hydrocarbons up to C₄, in DME used as fuel by the gas chromatography method. This procedure is applicable to determine the amount of methanol, CO, CO₂, methyl formate, ethyl methyl ether, and hydrocarbons up to C₄, up to the value specified in ISO 16861.

NOTE The precision of this method has been studied for a limited set of samples and content levels by a limited number 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 6975, *Natural gas — Extended analysis — Gas-chromatographic method*

ISO 16861, *Petroleum products — Fuels (class F) — Specifications of Dimethylether (DME)*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions given in ISO 6975 and the following apply.

3.1

components

DME, methanol, ethyl methyl ether, methyl formate, carbon dioxide, carbon monoxide, hydrocarbons up to C₄

3.2

hydrocarbons (up to C₄)

components such as ethane (C₂H₆), ethylene (C₂H₄), propane (C₃H₈), propylene (C₃H₆), i-butane (i-C₄H₁₀), n-butane (n-C₄H₁₀), i-butene (i-C₄H₈), 1-butene (1-C₄H₈), trans-2-butene (trans-2-C₄H₈), cis-2-butene (cis-2-C₄H₈), and 1,3-butadiene (1,3-butadiene)

3.3 working reference gas mixture WRM

mixtures which are used as working standards for regular calibration of the measuring system

Note 1 to entry: WRM can be prepared by gravimetric method in accordance with ISO 6142 or certified and validated by comparison with gas mixtures in accordance with ISO 6143.

4 Principle

The components to be determined in a gaseous sample are separated by gas chromatography and compared with calibration data obtained under the same set of conditions.

The components are separated using packed or open tubular columns in a gas chromatograph and detected by TCD or FID or FID with methanizer.

5 Analysis and analytical requirements

5.1 Apparatus and materials

5.1.1 Analytical system

The analytical system shall consist of a gas chromatograph and an appropriate data handling system.

NOTE Examples of analytical systems and conditions are informatively proposed in this International Standard. See [Annex A](#) and [Annex B](#).

The gas-chromatographic unit can consist of one or more gas chromatographs capable of isothermal and/or temperature-programmed operation and equipped with a TCD and an FID and a sample heated transfer and introduction system. A methanizer is optional.

The sample is preferably transferred as a gas phase to the injection system through a constant volume system according to the annexes.

If the sample is transferred as a gas phase, a specific procedure to allow full vaporization of liquid sample is required (see [6.3.1](#)).

A liquid sampling valve can also be used to inject the sample in a liquid phase.

5.1.2 Reference gas mixtures

5.1.2.1 Working reference gas mixture

The concentration of each component in the WRM shall be within the tolerances given in [Table 1](#) relative to the maximum value allowed in the specification in ISO 16861.

WRM for the following components should be prepared: CO, CO₂, methanol, methyl formate, propane, butane, and ethyl methyl ether.

WRM for other hydrocarbons up to C₄ can be prepared.

Standard reagents or standard gas for ethyl methyl ether might not be widely available in the market. However, it is necessary to obtain such a mixture to perform test methods described in this International Standard.

Example of a WRM is shown in [Annex C](#).

Table 1 — Tolerance between concentrations of components in the WRM and the sample

Component concentration allowed in specification ISO 16861 mass %	Deviation of component concentration in WRM (% relative to value specified in ISO 16861)
Up to 0,1	±50
0,1 to 1	±25

5.1.2.2 Control gas

A control gas is a high-pressure gas mixture containing all the components present in the working-reference gas mixture. A sample gas mixture having a composition closely related to the WRM can be used.

A control gas is used for the determination of the mean (p) and standard deviation (a) of the concentrations of the components detected.

5.2 Resolution

The resolution between two adjacent peaks for each component shall not be less than 2.

In the event that a valve switching is performed in multicolumn analysis, the resolution between the peak of the component eluted before switching the valve and the peak of the component that would be eluted without the valve switching shall not be less than 4.

If the resolution is unsatisfactory, the selection of chromatograph columns and/or the analytical conditions should be optimized until the expected resolution is obtained.

6 Procedures

6.1 Setting up the analytical system

Set up all the analytical system in accordance with the manufacturer's instructions and the analytical methods chosen.

Condensation and sorption in the sampling system shall be avoided. The sample cylinder and the transfer line and all the lab equipment shall be in a well air-conditioned room or the sample cylinder and transfer line shall be heated to at least 10 °C above the greater of the sampling temperature or ambient temperature. If necessary, use a heated vaporizer to ensure complete vaporization. In case of gas phase transfer and injection, special precautions shall be taken at any spot in the system where pressure reduction occurs.

6.2 Sample preparation

Samples shall be taken as described in ISO 29945.

NOTE Since DME is a liquefied gas, depending on the filling level and pressure, CO and CO₂ might be more concentrated in the vapour phase of the sample container. The CO and CO₂ concentration in the vapour phase can be calculated if the distribution coefficient of CO and CO₂ in liquid phase and vapour phase is known at given pressure. See [Annex E](#) for values of the distribution coefficient.

6.3 Injection

Either of the two following procedures can be used.