BS ISO 17786:2015

BSI Standards Publication

Dimethyl ether (DME) for fuels — Determination of high temperature (105°C) evaporation residues — Mass analysis method

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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INTERNATIONAL STANDARD

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Dimethyl ether (DME) for fuels — Determination of high temperature (105°C) evaporation residues — Mass analysis method

Diméthylether (DME) pour carburants et combustibles — Détermination de la température haute (105°C) de résidus d'évaporation — Méthode gravimétrique

Reference number
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Contents

Page

Foreword

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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).

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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

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 of increasing evaporation residues.

The evaporation residues can over time impact the performance of the equipment when DME is used as fuel. Therefore, evaporation residues in the DME shall be determined accurately using procedures recognized by the parties concerned.

INTERNATIONAL STANDARD ISO 17786:2015(E)

Dimethyl ether (DME) for fuels — Determination of high temperature (105°C) evaporation residues — Mass analysis 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 high temperature (105 °C) evaporation residue in DME used as fuel by the mass analysis method. This procedure is applicable to determine the amount of high temperature (105 °C) evaporation residue up to the value specified in ISO 16861.

Several tests can be applied to determine amount of evaporation residue in liquefied products. Among them, this International Standard specifies the method that has detection limit sufficient for a DME specification in ISO 16861, using less resource compared to other methods.

When more precise quantitative test is required, use of ISO 13757 instead of this International Standard is recommended.

Because of the procedure applied, the evaporation residue which has a boiling point lower than 105 °C will not be determined.

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 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 Principle

After approximately 100 g of liquid sample is evaporated by using an evaporation dish, the residue is weighed, to determine the evaporation residue at 105 °C in mass percentage of the total DME.

4 Reagents and materials

4.1 **Ethanol**, methanol and/or isopropanol is used for cooling bath.

4.2 **Dry ice**, used for cooling bath.

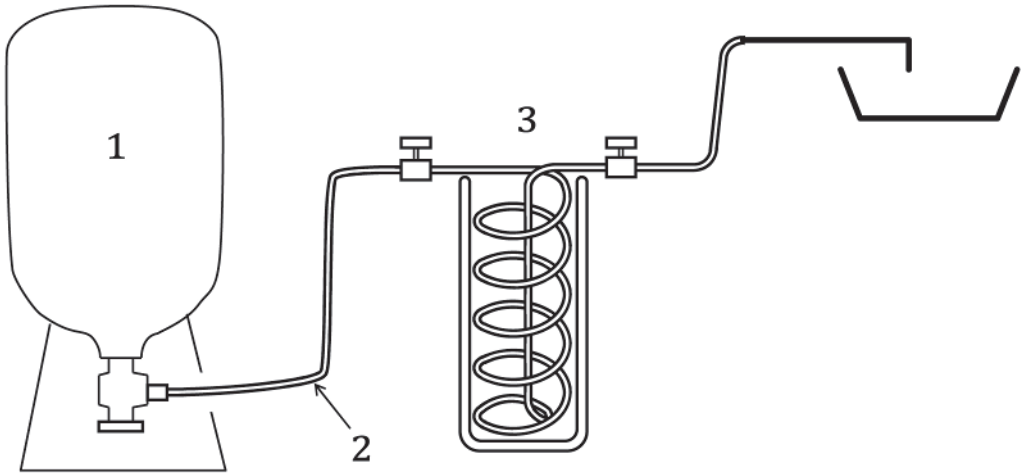
5 Apparatus

5.1 Sample cylinder.

Sample cylinder is made of stainless steel. (size is 1 l, tested pressure not less than 3,0 MPa) that shall be able to be positioned vertically and is weighable. It shall be equipped so that the sample be drawn as a liquid from the liquid phase of the sample.

5.2 Connecting pipe.

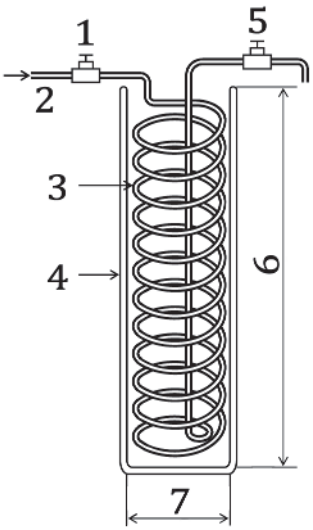
This is a pipe of approximately 3 mm~5 mm in diameter, made of stainless steel. It is provided with a connector to the sample cylinder on one end and with a connector to the cooler on the other end.



Key

1 sample cylinder
2 connecting pipe
3 cooling bath

Figure 1 — Assembly drawing of evaporation residue testing equipment



Key

1 sample valve
2 connecting pipe
3 cooling pipe (stainless steel pipe 5 mm)
4 dewar vessel
5 needle valve
6 below 300
7 approximately 64 mm

Figure 2 — Cooling bath

5.3 Cooling bath.

This is of a type shown in Figure 2.

5.4 Evaporating dish.

The evaporating dish is made of platinum, porcelain, stainless steel, or glass and the volume of the evaporating dish is 100 ml. The diameter of the dish is more than 75 mm and the depth is more than 30 mm.

5.5 Electronic balance.

a) Samples weight are determined by means of top loading electronic balance with precision at least 0,1 g.

b) Evaporation residue weights are determined by means of top loading electronic balance with accuracy at least 0,1 mg.

5.6 Oven.

It shall be capable of holding the temperature at 105 °C ± 2 °C and shall not be provided with an ignition source.

5.7 Desiccator (with silica gel).

It shall be able to store the evaporating dish.

6 Procedures

6.1 Samples shall be taken as described in ISO 29945.

6.2 The clean evaporation dish is placed into the oven at 105 °C for 60 min, and cooled in the desiccator for another 30 min. The mass is measured to the digit of 0,1 mg. [*A*, mass of the evaporating dish (g)].

6.3 Dry ice and methanol are put into the cooling bath (to be under – 50 °C), to cool the spiral tube.

6.4 Sample cylinder, connecting pipe, cooler are connected as shown in Figure 1. Then, the valves are opened in the order of the sample cylinder valve, sample valve and needle valve, and the connecting pipe, spiral tube are prewashed with the liquid sample.

6.5 The sample cylinder valve is opened, and the sample is led into the evaporation dish in liquid state. At this time, the sample is supplied gradually until the evaporation dish cools down. Needle valves should be used to adjust the sample flow.

6.6 When approximately 50 g of liquid sample is led into the evaporating dish, the valve is closed and the mass of sample weighed to the digit of 0,1 g.[*B*, mass of sample DME (g), tare the evaporating dish].

6.7 While ensuring that sudden boiling will not occur, the evaporating dish is exposed to atmosphere and the sample will be almost evaporated.

6.8 Repeat 6.4 and 6.5. [*C*, mass of sample DME (g), tare the evaporating dish]. $B + C$ is approximately 100 g.

6.9 The evaporating dish is placed in the oven at 105 °C for 60 min.

6.10 The evaporating dish is taken out of the oven. After being left cooling in the desiccator for 30 min, the mass of the evaporating dish is weighed to the digit of 0,1 mg [*D*, mass of the evaporating dish after the procedure (g)].

NOTE During these procedures, there is a risk of fire. Extra caution shall be taken, especially procedures including and after 6.3. The procedures including and after 6.3 shall be carried out in a fume food or an equivalent.

7 Calculation

The evaporation residue is calculated in accordance with Formula (1), and the result is rounded to four decimal.

$$E(\text{mass }\%) = \frac{(D - A) \times 10^4}{B + C} \tag{1}$$

where

E is the evaporation residue (mass %);

A is the mass (g) of the evaporating dish, measured in 6.2;

B is the collected amounts of the sample (g), measured in 6.6;

C is the collected amounts of the sample (g), measured in 6.8;

D is the mass (g) of the evaporating dish after evaporation at 105 °C, measured in 6.10.

8 Precision

The provisional precision of this method, established during interlaboratory tests relating to DME sample with residue content of 20,3 mg/kg, and determined on the basis of statistical examination of interlaboratory test results is shown in Table 1. Refer to Annex A for the report of the interlaboratory test.

NOTE The emphasis of the interlaboratory study was to confirm that the sample analysed fulfils ISO 16861 specification or not, rather than to establish an analytical method applicable for a wide range. The reproducibility determined is only indicative and is not considered as one established according to normal statistical procedures as in ISO 4259. The figures are provisional and further work in the future is intended to improve the estimation given.

Repeatability, *r*: The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed the value below in only one case in 20.

Reproducibility, *R*: The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed the value below in only one case in 20.

Table 1 — Provisional precision

Dimension in mass %

Component	**Content**	**Repeatability**, *r*	**Reproducibility**, *R*
Evaporation residue	0 to 0,007 0	0,26*X*	0,71*X*
NOTE *X* is the mean value of the measurements.			

9 Test report

The test report shall include at least the following information:

a) a reference to this International Standard, i.e. ISO 17786:2015;

b) all the necessary information for complete identification of the sample, for example, ISO 29945:

— date of sampling, and

— place in the pipeline system at which the sample was taken;

c) the sampling method used (including the size and type of material of the high-pressure cylinder used);

d) the content of the evaporation residue (mass) in mass % to four decimal;

e) the details of any deviation from the procedure specified.

Annex A
(informative)

The report of the interlaboratory tests

A.1 Precision

a) Outline of the test

A repeatability standard deviation and a reproducibility standard deviation were calculated from results of a Round-robin test which was carried out with one sample by eight laboratories.

b) Round-robin test results

The precision was evaluated by Cochran's test and Grubbs' test prescribed on ISO 5725-2, then, the repeatability standard deviation and the reproducibility standard deviation were calculated. The test results are shown in Table A.1. These values were determined from a level experiment involving 8 laboratories with 0,002 14 mass % of sample, in which one straggler were detected by the Grubbs' test and retained.

Table A.1 — Round-robin test results (Evaporation residues)

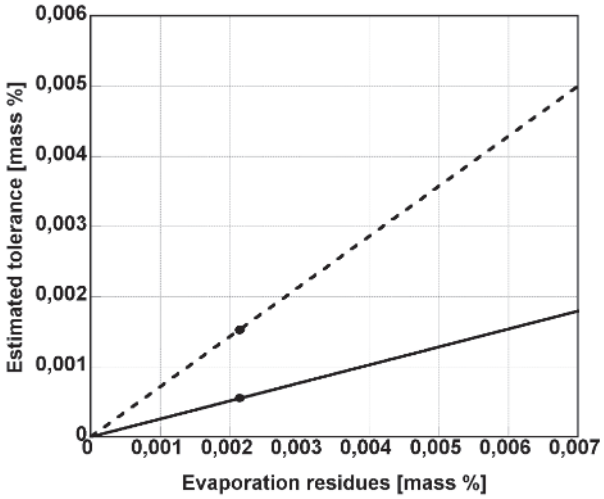
Dimension in mass %

Laboratory	1st	2nd	3rd	4th	5th	Average	Standard deviation	Number of tests
L1	0,000 38	0,001 13	0,001 34	–	–	0,000 95	0,000 50	3
L2	0,000 89	0,001 07	0,000 87	–	–	0,000 94	0,000 11	3
L3	0,002 91	0,002 92	0,002 92	–	–	0,002 92	0,000 01	3
L4	0,005 63	0,006 33	0,004 31	–	–	0,005 42	0,001 02	3
L5	0,003 59	0,002 07	0,001 39	–	–	0,002 35	0,001 13	3
L6	0,001 17	0,001 30	0,001 76	–	–	0,001 41	0,000 31	3
L7	0,000 98	0,001 07	0,000 85	–	–	0,000 97	0,000 11	3
L8	0,002 22	0,002 07	0,002 12	0,001 94	0,002 30	0,002 13	0,000 14	5
The general mean	0,002 14							
All number of tests	26							
Repeatability standard deviation	0,000 55							
Reproducibility standard deviation	0,001 53							

c) Provision of precision

Normally, results measured by plural level samples should be used for calculation of precision, only one level sample was used in this Round-Robin Test because of limited the samples and the time. Therefore, tolerance of repeatability and the reproducibility standard deviation were estimated assuming that the

repeatability and the reproducibility standard deviation calculated by the test results are linear. The estimated tolerance of repeatability and the reproducibility standard deviation is shown in Figure A.1.



Key

— — — — reproducibility standard deviation

———— repeatability standard deviation

Figure A.1 — Estimated tolerance of repeatability and the reproducibility standard deviation

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