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Petroleum and related products from natural or synthetic sources — Determination of cloud point

Produits pétroliers et connexes d'origine naturelle ou synthétique — Détermination du point de trouble

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

This third edition cancels and replaces the second edition (ISO 3015:1992), which has been technically revised. The main changes compared to the previous edition are as follows:

— extension of the scope to diesel fuels with up to 30 % (*V/V*) FAME and inclusion of paraffinic diesel fuels;

— inclusion of digital contact thermometer;

— normative references in Clause 2 have been updated;

— bath and sample temperature ranges have been aligned with ASTM D2500[1], changes in bath temperature and the temperatures at which the test jars are moved to the batch with the next lower temperature have over the years (1992 up to the time of publication of this document) not led to observation of a bias versus test results obtained with the former edition;

— the precision for 'other products' has been removed as data to support it could not be obtained for comparison;

— a bibliography has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Petroleum and related products from natural or synthetic sources — Determination of cloud point

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to application of this document, and to determine the applicability of any other restrictions.

1 Scope

This document specifies a method for the determination of the cloud point of petroleum products which are transparent in layers 40 mm in thickness and have a cloud point below 49 °C, amongst which are diesel fuels with up to 30 % (*V/V*) of fatty acid methyl ester (FAME)[2], paraffinic diesel fuels with up to 7 % (*V/V*) FAME[3], 100 % FAME[5] and lubricants.

NOTE For the purposes of this document, the term "% (*V/V*)" is used to represent the volume fraction (φ) of a material.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ASTM D7962, *Standard Practice for Determination of minimum Immersion Depth and Assessment of Temperature Sensor Measurement Drift*

ASTM E644-11, *Standard Test Methods for Testing Industrial Resistance Thermometers*

ASTM E2877, *Standard Guide for Digital Contact Thermometers*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
cloud point
temperature at which a cloud of wax crystals first appears in a liquid when it is cooled under specified conditions

4 Principle

A sample is cooled at a specified rate and examined periodically. The temperature at which a cloud is first observed at the bottom of the test jar is recorded as the cloud point.

5 Apparatus

5.1 Test jar, cylindrical, of clear glass, flat bottomed, 33,2 mm to 34,8 mm in outside diameter and 115 mm to 125 mm in height; the inside diameter of the jar may range from 30,0 mm to 32,4 mm, within the constraint that the wall thickness be no greater than 1,6 mm.

See Figure 1 for more details. The jar shall be marked with a line to indicate a sample height 54 mm ± 3 mm above the inside bottom.

5.2 Temperature measuring device, one of the following.

5.2.1 Liquid-in-glass thermometers, as described in A.2.

5.2.2 Digital contact thermometer (DCT), meeting the requirements specified in A.1.

5.3 Cork, to fit the test jar, bored centrally to take the test thermometer.

5.4 Jacket, watertight, cylindrical, metal, flat bottomed, about 115 mm in depth, with an inside diameter of 44,2 mm to 45,8 mm. It shall be supported in a vertical position in a cooling bath (5.7) so that not more than 25 mm projects out of the cooling medium, and it shall be capable of being cleaned.

5.5 Disc, of cork or felt, 6 mm in thickness, to fit loosely inside the jacket.

5.6 Gasket, ring form, about 5 mm in thickness, to fit snugly on the outside of the test jar and loosely inside the jacket. This gasket may be made of rubber, leather or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape.

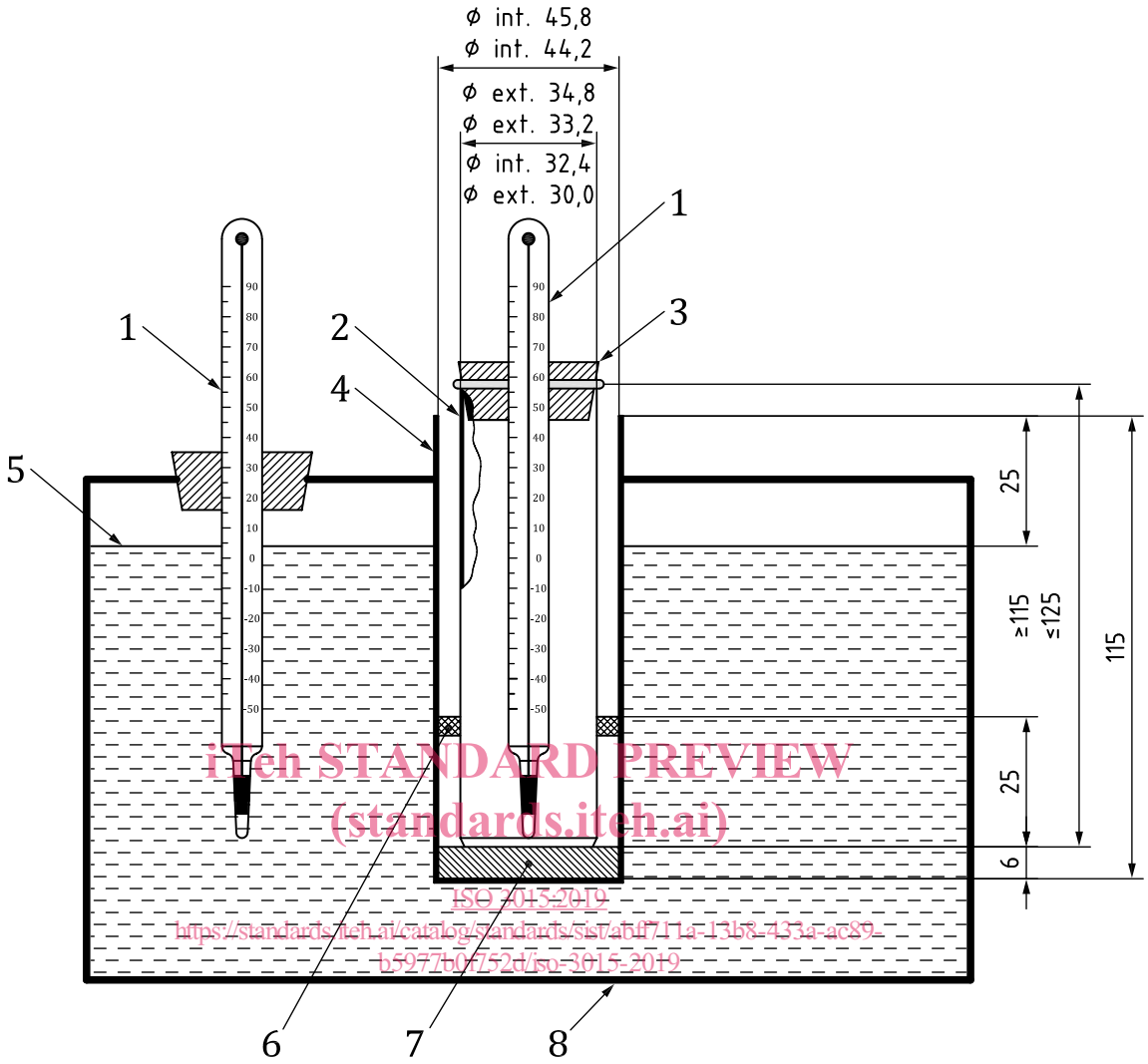
NOTE The purpose of the ring gasket is to prevent the test jar from touching the jacket.

5.7 Cooling baths, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be obtained by refrigeration if available, otherwise by suitable cooling mixtures. Recommended cooling mixtures commonly used for bath temperatures are given in Annex B.

6 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in ISO 3170 or ISO 3171.

Dimensions in millimetres



Key

1 thermometer (5.2)
2 test jar (5.1)
3 cork (5.3)
4 jacket (5.4)
5 coolant level
6 gasket (5.6)
7 disc (5.5)
8 cooling bath (5.7)

Figure 1 — Overview of cloud point apparatus

7 Procedure

7.1 Bring the sample to be tested to a temperature at least 14 °C above the approximate cloud point, but not above 49 °C. Remove any moisture present by any suitable method, such as filtration through dry lint-less filter paper, until the sample is perfectly clear, working at a temperature of at least 14 °C above the approximate cloud point, but not above 49 °C.

7.2 Pour the clear sample into the test jar (5.1) to the level mark.

7.3 If using a liquid-in-glass thermometer and the expected cloud point is above −36 °C, then use the high cloud and pour point thermometer; otherwise use the low cloud and pour point thermometer. Close the test jar tightly by the cork (5.3) carrying the appropriate test thermometer (see 5.2) and adjust the

position of the cork and the thermometer so that the cork fits tightly, the thermometric device and the jar are coaxial, and the thermometer bulb or probe is resting on the bottom of the jar.

The liquid-column separation of thermometers occasionally occurs and may escape detection. Thermometers shall therefore be checked immediately prior to the test and used only if the ice point is 0 °C ± 1 °C, measured with the thermometer immersed to the immersion line in an ice bath and with the emergent-stem temperature not differing significantly from 21 °C. Alternatively, immerse the thermometer to the reading level and correct for the resultant lower stem temperature.

7.4 Ensure that the disc (5.5), the gasket (5.6) and the inside of the jacket (5.4) are clean and dry. Place the disc in the bottom of the jacket. The disc and jacket shall have been placed in the cooling medium (5.7) a minimum of 10 min before the test jar is inserted. Place the gasket round the test jar, 25 mm from the bottom. Insert the test jar in the jacket. Never place a jar directly into the cooling medium.

The use of a jacket cover while the empty jacket is cooling is permitted.

NOTE Failure to keep the disc, the gasket and the inside of the jacket clean and dry, can lead to frost formation which can cause erroneous results.

7.5 Maintain the temperature of the cooling bath at 0 °C ± 1,5 °C.

7.6 At each test thermometer reading that is a multiple of 1 °C, remove the test jar from the jacket quickly but without disturbing the sample.

Inspect for cloud and replace in the jacket. Ensure that this complete operation takes no more than 3 s.

If the sample does not show a cloud when it has been cooled to 9 °C, transfer the test jar to a jacket in a second bath maintained at a temperature of −18 °C ± 1,5 °C (see Table 1). Do not transfer the jacket. If the sample does not show a cloud when it has been cooled to −6 °C, transfer the test jar to a jacket in a third bath maintained at a temperature of −33 °C ± 1,5 °C.

For determination of very low cloud points, additional baths are required, each bath to be maintained in accordance with Table 1. In each case, transfer the jar to the next bath, if the sample does not exhibit cloud point and the temperature of the sample reaches the lowest sample temperature in the range identified for the current bath in use, based on the ranges stated in Table 1.

Table 1 — Bath and sample temperature ranges

Bath #	Bath temperature setting °C	Sample temperature range °C
1	0 ± 1,5	Start to +9
2	−18 ± 1,5	+9 to −6
3	−33 ± 1,5	−6 to −24
4	−51 ± 1,5	−24 to −42
5	−69 ± 1,5	−42 to −60

7.7 Record as the cloud point the temperature to the nearest 1 °C, at which any cloud is observed at the bottom of the test jar, which is confirmed by continued cooling.

The wax cloud or haze is always noted first at the bottom of the test jar, where the temperature is lowest. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature is lowered, is usually due to traces of water in the sample. Generally, this water haze will not interfere with the determination of the wax cloud point. In most cases of interference, filtration through dry lint-less filter paper such as described in 7.1 is sufficient.

In the case of diesel fuels, however, if the haze is very dense, a fresh portion of the sample shall be dried by shaking 100 ml with 5 g of anhydrous sodium sulfate (see B.6) for at least 5 min and then filtering through dry lint-less filter paper. Given sufficient contact time, this procedure will remove or

sufficiently reduce the water haze so that the wax cloud can be readily discerned. Drying and filtering shall always be carried out at a temperature at least 14 °C above the approximate cloud point, but not in excess of 49 °C.

8 Expression of results

Report the cloud point to the nearest 1 °C.

9 Precision

9.1 General

The precision of this test method is determined by statistical examination of interlaboratory results[5] in line with ISO 4259-1[6] and further validation studies[7][8][9], is as in 9.2 and 9.3

The precision statements were developed using liquid-in-glass thermometers corresponding to those in ASTM E1 or IP specifications for IP standard thermometers[10][11][12].

9.2 Repeatability

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of this test method, exceed 2 °C only in one case in 20.

9.3 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would in the long run, in the normal and correct operation of this test method, exceed 4 °C only in one case in 20.

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this document, i.e. ISO 3015:2019;

c) the result of the test (see Clause 8);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of test.