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Petroleum products — Determination of cloud point

Produits pétroliers — 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.

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.

International Standard ISO 3015 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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Petroleum products — Determination of cloud point

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this 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 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.

2 Definition

For the purposes of this International Standard, the following definition applies.

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

3 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.

4 Apparatus (see figure 1)

4.1 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 mm to 32,4 mm, within the constraint that the wall thickness be no greater than 1,6 mm. The jar shall be marked with a line to indicate a sample height 54 mm $\pm$ 3 mm above the inside bottom.

4.2 Thermometers, partial-immersion type, conforming to the specifications in table 1.

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

4.4 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 (4.7) so that not more than 25 mm projects out of the cooling medium, and it shall be capable of being cleaned.

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

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

Dimensions in millimetres

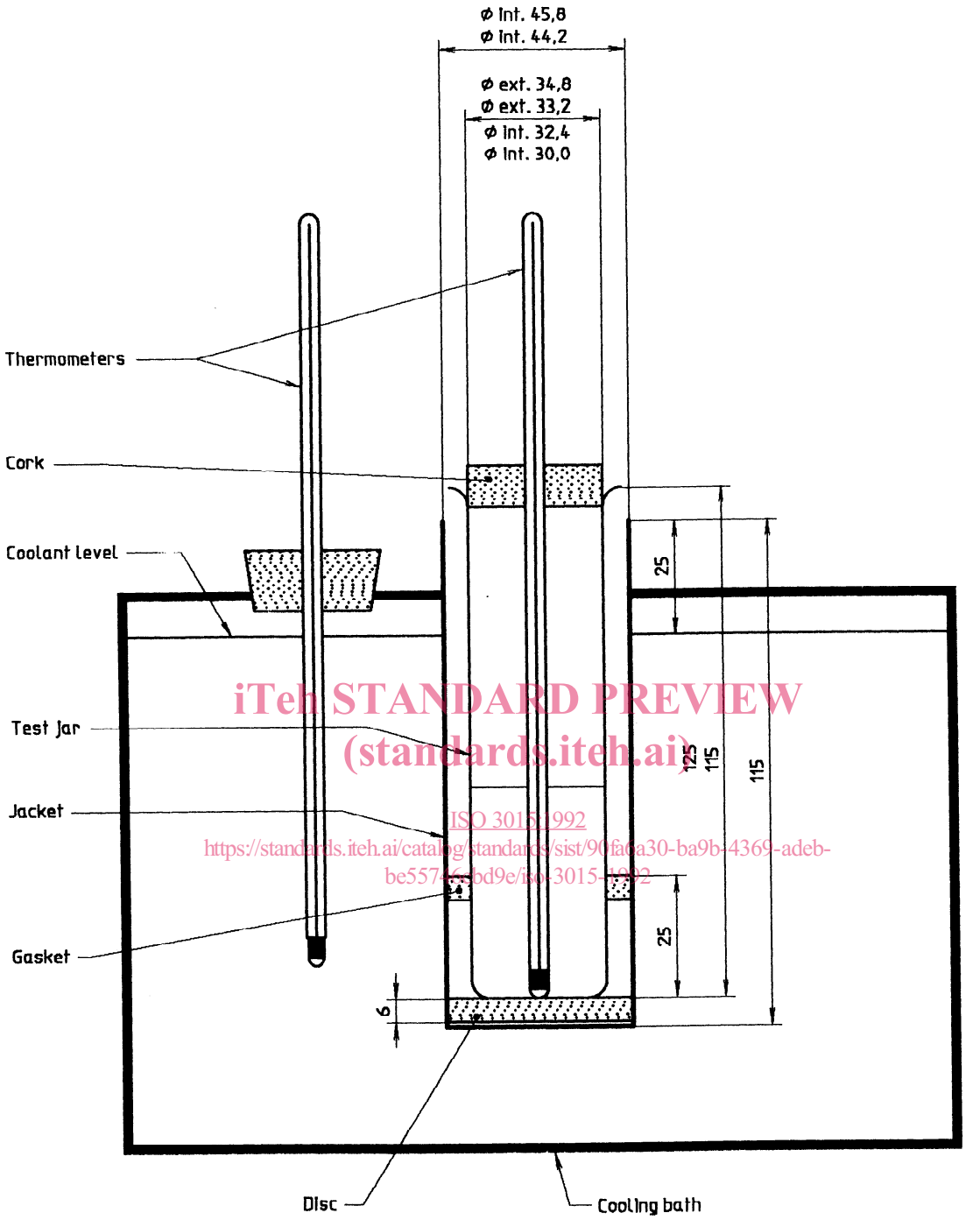

Figure 1 — Apparatus for cloud-point test

Table 1 — Specifications of thermometers

Specification	High cloud and pour	Low cloud and pour
Range	− 38 °C to + 50 °C	− 80 °C to + 20 °C
Immersion length	108 mm	76 mm
Graduation at each	1 °C	1 °C
Longer lines at each	5 °C	5 °C
Figured at each	10 °C	10 °C
Scale error not to exceed	0,5 °C	1 °C down to − 33 °C, 2 °C below − 33 °C
Expansion chamber: heating permitted to	100 °C	60 °C
Overall length	230 mm ± 5 mm	230 mm ± 5 mm
Stem diameter	6 mm to 8 mm	6 mm to 8 mm
Bulb length	7,0 mm to 10 mm	7,0 mm to 10 mm
Bulb diameter	5,5 mm min, but not greater than stem diameter	5,0 mm min, but not greater than stem diameter
Distance from bottom of bulb to line at	− 38 °C : 120 mm to 130 mm	− 70 °C : 100 mm to 120 mm
Length of scale	65 mm to 85 mm	70 mm to 100 mm

NOTE — The emergent-stem temperature is 21 °C throughout the scale range.

4.7 Cooling baths, of a type suitable for obtaining the required temperatures.

The size and shape of the baths are not specified, but a support to hold the jackets firmly in a vertical position is essential. The bath temperature shall be monitored by means of a high or low cloud and pour thermometer immersed to the correct immersion depth.

For the determination of cloud points below 10 °C, two or more baths are needed. The required bath temperatures may be maintained by refrigeration or by suitable freezing mixtures.

NOTE 1 The freezing mixtures commonly used are as follows:

For cloud-point temperatures down to

10 °C: ice and water

− 12 °C: crushed ice and sodium chloride crystals

− 26 °C: crushed ice and calcium chloride crystals

− 57 °C: solid carbon dioxide and acetone or petroleum naphtha.

The CO_2-based mixture may be made as follows: In a covered metal beaker, chill a suitable amount of acetone or petroleum naphtha to − 12 °C, or lower, by means of an ice/salt mixture. Then add enough solid carbon dioxide to the chilled acetone or petroleum naphtha to give the desired temperature. Solid carbon dioxide is commercially available in many areas.

5 Procedure

5.1 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 lintless 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.

5.2 Pour the clear sample into the test jar (4.1) to the level mark.

5.3 Close the test jar tightly by the cork (4.3) carrying the appropriate test thermometer (see 4.2). Use the high cloud and pour thermometer if the expected cloud point is at or above − 36 °C and the low cloud and pour thermometer if the expected cloud point is below − 36 °C. Adjust the position of the cork and the thermometer so that the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is resting on the bottom of the jar.

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.

5.4 Ensure that the disc (4.5), the gasket (4.6) and the inside of the jacket (4.4) 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 (see 4.7) a minimum of 10 minutes 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.

NOTES

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

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

5.5 Maintain the temperature of the cooling bath at − 1 °C to + 2 °C.

5.6 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 10 °C, transfer the test jar to a jacket in a second bath maintained at a temperature of − 18 °C to − 15 °C (see table 2). Do not transfer the jacket. If the sample does not show a cloud when it has been cooled to − 7 °C, transfer the test jar to a jacket in a third bath maintained at a temperature of − 35 °C to − 32 °C.

For determination of very low cloud points, additional baths are required, each bath maintained at 17 °C below the temperature of the preceding bath (see table 2). In each case, transfer the jar to the next bath when the temperature of the sample comes to 28 °C above the temperature of the low end of the temperature range of the next bath.

Table 2 — Bath and sample temperature ranges

Bath	Bath temperature range °C	Sample temperature range °C
1	− 1 to + 2	Start to + 10
2	− 18 to − 15	+ 9 to − 7
3	− 35 to − 32	− 8 to − 24
4	− 52 to − 49	− 25 to − 41
5	− 69 to − 66	− 42 to − 58

5.7 Report 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 lintless filter paper such as described in 5.1 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 for at least 5 min and then filtering through dry lintless 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.

6 Precision

6.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

6.2 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 for distillate products and 6 °C for other products only in one case in twenty.

6.3 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 for distillate products and 6 °C for other products only in one case in twenty.

7 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 International Standard;

c) the result of the test (see 5.7);

d) the date of test.

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