
INTERNATIONAL STANDARD



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Petroleum oils — Determination of cloud point

Huiles de pétrole — Détermination du point de trouble

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3015 was drawn up by Technical Committee ISO/TC 28, *Petroleum products*, and circulated to the Member Bodies in March 1973.

It has been approved by the Member Bodies of the following countries :

Australia	Germany	Portugal
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iran	Spain
Bulgaria	Israel	Sweden
Canada	Mexico	Thailand
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
Egypt, Arab Rep. of	Norway	U.S.A.
France	Poland	U.S.S.R.

No Member Body expressed disapproval of the document.

Petroleum oils – Determination of cloud point

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the cloud point of petroleum oils which are transparent in layers 40 mm in thickness, and have a cloud point below 49 °C.

2 DEFINITION

cloud point: The temperature at which a clear liquid product becomes hazy or cloudy due to the appearance of wax crystals when the test is conducted under standardized conditions.

3 PRINCIPLE

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

4 APPARATUS (see figure)

4.1 Test jar, cylindrical, of clear glass, flat bottomed, 30 to 33,5 mm in inside diameter and 115 to 125 mm in height. The jar should be marked with a line to indicate a sample volume of 45 ml. Jars marked with upper and lower permitted levels, ± 3 mm of the sample volume line, are allowed.

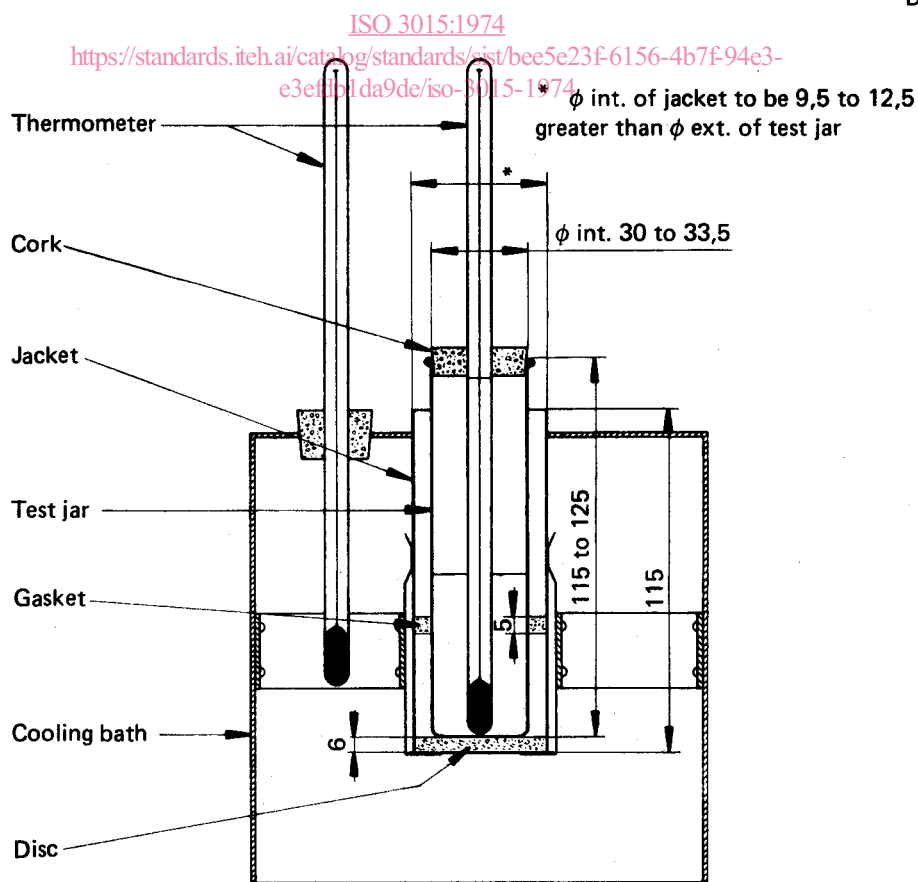


FIGURE – Apparatus for cloud point test

4.2 Thermometers, partial immersion type conforming to the following specifications :

Specification	High cloud and pour	Low cloud and pour
Range	-38 to + 50 °C	-80 to + 20 °C
Immersion	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 permitting heating to	100 °C	60 °C
Overall length	231 ± 5 mm	232 ± 5 mm
Stem diameter	7 to 8 mm	7 to 8 mm
Bulb length	7,0 to 9,5 mm	8,0 to 9,5 mm
Bulb diameter	5,5 to 7,0 mm	5,0 to 6,5 mm
Distance from bottom of bulb to line at	- 38 °C : 120 to 130 mm	- 57 °C : 120 to 130 mm
Distance from bottom of bulb to line at	49 °C : 195 to 205 mm	20 °C : 182 to 196 mm

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

4.4 Jacket, watertight, cylindrical, of glass or metal, flat-bottomed, about 115 mm in depth, with inside diameter 9,5 to 12,5 mm greater than the outside diameter of the test jar.

4.5 Disc, of cork or felt, 6 mm in thickness and of the same diameter as the inside of the jacket.

4.6 Gasket, ring form, about 5 mm in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. This gasket may be made of cork, felt, 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.

4.7 Cooling bath, of a type suitable for obtaining the required temperatures. The size and shape of the bath are optional, but a support to hold the jacket firmly in a vertical position is essential. 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 — The freezing mixtures commonly used are as follows :

For 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 naphtha¹⁾

5 PROCEDURE

5.1 Bring the oil to be tested to a temperature at least 14 °C above the approximate cloud point. Remove any moisture present by any suitable method, such as by filtration through dry lintless filter paper, until the oil is perfectly clear, but make such filtration at a temperature of at least 14 °C above the approximate cloud point.

5.2 Pour the clear oil into the test jar to the level mark, or to a level between the two etched lines according to type.

5.3 Close the test jar tightly by the cork carrying the test thermometer (see note). Use the high cloud and pour thermometer if the expected cloud point is above - 38 °C and the low cloud and pour thermometer if the expected cloud point is below - 38 °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.

NOTE — Since separation of the mercury or toluene thread of cloud and pour thermometers occasionally occurs, and since such separation may otherwise escape immediate detection, it is suggested that the ice points of the thermometers be checked immediately prior to the test. Any thermometer that shows an ice point differing from 0 °C by more than 1 °C should be further examined or recalibrated, or both, before use.

5.4 Place the disc in the bottom of the jacket. Place the ring gasket around the test jar, 25 mm from the bottom. The disc, gasket, and inside and outside of the jacket shall be clean and dry. Insert the test jar in the jacket.

5.5 Maintain the temperature of the cooling bath at - 1 to + 2 °C. Support the jacket containing the test jar firmly in a vertical position in the cooling bath so that not more than 25 mm of the jacket projects out of the cooling medium.

1) This 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. If necessary, it may be prepared as follows : invert a cylinder of liquid carbon dioxide and draw off carefully into a chamois skin bag the desired amount of carbon dioxide which, through rapid evaporation, becomes solid.

5.6 At each test thermometer reading showing a drop of 1 °C, remove the test jar from the jacket quickly but without disturbing the oil, inspect for cloud, and replace in the jacket. This complete operation shall require not more than 3 s. If the oil does not show a cloud when it has been cooled to 10 °C, transfer the test jar to another jacket in a second bath maintained at a temperature of -18 to -15 °C. If the oil does not show a cloud when it has been cooled to -7 °C, transfer the test jar to another jacket in a third bath maintained at a temperature of -34,5 to -31,5 °C.

For the determination of very low cloud points, additional baths are required, each bath to be maintained at 17 °C below the temperature of the preceding bath. In each case transfer the test jar when the temperature of the oil reaches a point 28 °C above the temperature of the new bath. At no time place the cold test jar directly in the cooling medium.

5.7 When such inspection first reveals a distinct cloudiness or haze in the oil at the bottom of the test jar, record the reading of the test thermometer as the cloud point (see note).

NOTE — A 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 oil. 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 papers 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 should be dried by shaking 100 ml with 5 g of anhydrous sodium sulphate 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 should be done always at a temperature at least 14 °C above the approximate cloud point but not in excess of 49 °C unless the expected cloud point is above 35 °C.

6 PRECISION

The following criteria shall be used for judging the acceptability of results (95 % confidence level) :

6.1 Repeatability

Duplicate results by the same operator shall be considered suspect if they differ by more than 2 °C in the case of distillate oils, and by more than 6 °C in the case of other oils.

6.2 Reproducibility

The results submitted by each of two laboratories shall be considered suspect if the two results differ by more than 4 °C in the case of distillate oils and by more than 6 °C in the case of other oils.

7 TEST REPORT

Report the temperature recorded in 5.7 as the cloud point and make reference to this International Standard.

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