INTERNATIONAL STANDARD ISO 3016

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Petroleum oils — Determination of pour point

Huiles de pétrole — Détermination du point d'écoulement

First edition — 1974-05-01

UDC 665.7.035.6

Ref. No. ISO 3016-1974 (E)

Descriptors : petroleum products, oils, tests, low temperature tests, measurement, solidification points, test equipment.

Price based on 3 pages

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 3016 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	India	South Africa, Rep. of
Austria	Iran	Spain
Belgium	Israel	Sweden
Brazil	Mexico	Thailand
Bulgaria	Netherlands	Turkey
Chile	New Zealand	United Kingdom
Czechoslovakia	Norway	U.S.A.
Egypt, Arab Rep. of	Poland	U.S.S.R.
Germany	Portugal	
Hungary	Romania	

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Canada
France

Printed in Switzerland

Petroleum oils — Determination of pour point

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the pour point of any petroleum oil. A procedure suitable for black oils, cylinder stock, and non-distillate fuel oil is described in 5.9.

2 DEFINITION

pour point : The lowest temperature at which an oil will continue to flow when it is cooled under standardized prescribed conditions.

3 PRINCIPLE

After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3 °C for flow characteristics. The lowest temperature at which movement of the oil is observed is recorded as the pour 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.

Dimensions in millimetres

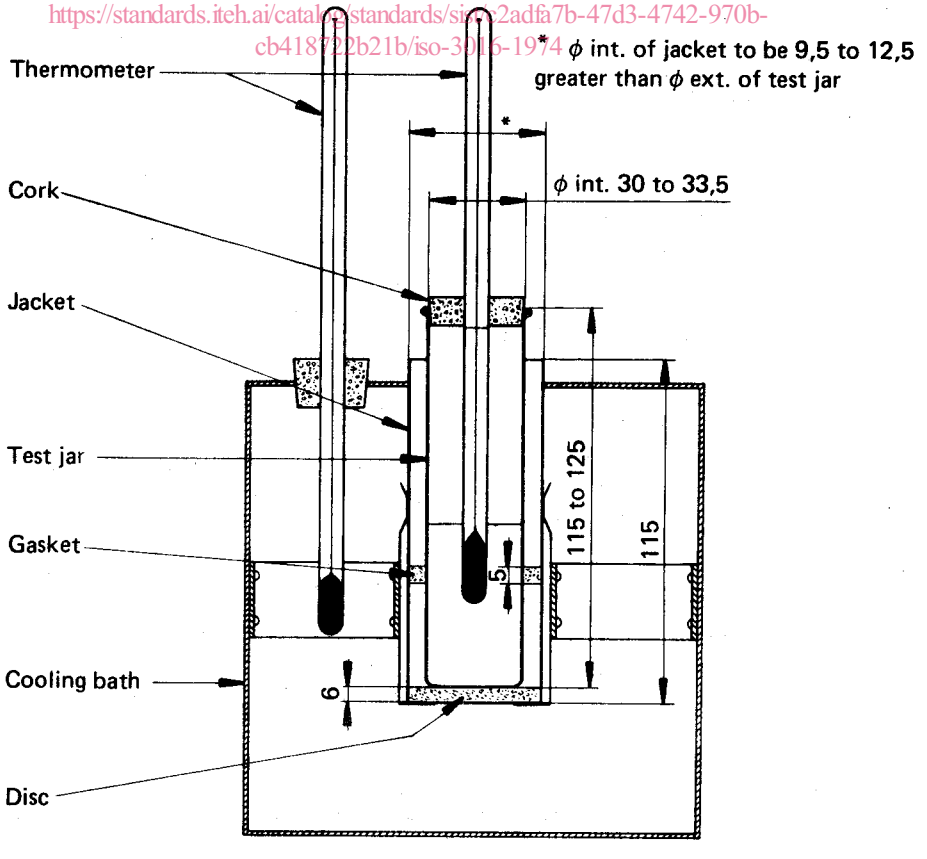

FIGURE — **Apparatus for pour point test**

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 pour 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 naphtha.[1)]

5 PROCEDURE

5.1 Pour the clear oil into the test jar to the level mark or to a level between the two etched lines according to type (see note). When necessary heat the oil in a water bath until it is just sufficiently fluid to pour into the test jar.

NOTE – When it is known that a sample has been heated to some temperature higher than 45 °C during the preceding 24 h or when the thermal history of the sample is not known, keep the sample at room temperature for 24 h before testing it.

5.2 Close the test jar tightly by the cork carrying the high cloud and pour thermometer (4.2), or in the case of pour points above 39 °C, a thermometer as described in note 1. 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 immersed so that the beginning of the capillary is 3 mm below the surface of the oil (see note 2).

NOTES

1 For tests above 39 °C it is permissible to use any thermometer that includes the range from 32 to 105 °C. A total immersion thermometer with graduations of 0,5 °C is suggested.

2 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.

5.3 Subject the oil in the test jar to the following preliminary treatment :

5.3.1 *Oils having pour points between + 33 °C and − 33 °C*

Heat the oil, without stirring, to 45 °C in a bath maintained at 48 °C. Cool the oil to 36 °C in air or in a water bath at approximately 25 °C. Proceed as directed in 5.4.

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.

5.3.2 *Oils having pour points above + 33 °C*

Heat the oil in a water bath, without stirring, to 45 °C or to a temperature approximately 9 °C above the expected pour point (see note 1 to 5.2), whichever temperature is the higher. Proceed as directed in 5.4.

5.3.3 *Oils having pour points below − 33 °C*

Heat the oil as directed in 5.3.1 and cool to 15 °C in a water bath maintained at 7 °C. Remove the high cloud and pour thermometer and place the low cloud and pour thermometer in position. Proceed as directed in 5.4.

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.

5.6 After the oil has cooled enough to allow the formation of paraffin wax crystals, take great care not to disturb the mass of the oil nor to permit the thermometer to shift in the oil; any disturbance of the spongy network of wax crystals will lead to low and fictitious results.

5.7 Beginning at a temperature 9 °C above the expected pour point for oils having pour points above 33 °C, or for other oils, at a temperature 12 °C above the expected pour point, at each test thermometer reading that is a multiple of 3 °C, remove the test jar from the jacket carefully and tilt it just enough to ascertain whether there is a movement of the oil in the test jar. The complete operation of removal and replacement shall require not more than 3 s. If the oil has not ceased to flow when its temperature has reached 9 °C, transfer the test jar to another jacket in a second bath maintained at a temperature of − 18 to − 15 °C (see note). If the oil has not ceased to flow when its temperature has reached − 6 °C transfer the test jar to another jacket in a third bath maintained at a temperature of − 34,5 °C to − 31,5 °C.

For the determination of very low pour 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 27 °C above the temperature of the new bath (see note). As soon as the oil in the test jar does not flow when the jar is tilted, hold the test jar horizontally and carefully observe the surface of the oil. If there is any movement within 5 s (as measured by a stopwatch or other accurate timing device) immediately replace the test jar in the jacket and repeat the test for flow at the next temperature 3 °C lower.

NOTE — The jacket may be left in the bath or transferred with the test jar. Never place the cold test jar directly into the cooling medium.

5.8 Continue the test in this manner until a point is reached at which the oil in the test jar shows no movement when the test jar is held in a horizontal position for 5 s. Record the observed reading of the test thermometer.

5.9 For black oil, cylinder stock, and nondistillate fuel oil the result obtained by the procedure described in 5.1 to 5.8 is the upper (maximum) pour point (see note). If required, determine the lower (minimum) pour point by heating the sample, while stirring, to 105 °C, pouring it into the jar, cooling it to 36 °C as before, and determining the pour point as described in 5.1 to 5.8.

NOTE — When it is known that a sample has been heated to some temperature higher than 45 °C during the preceding 24 h or when the thermal history of the sample is not known, heat the sample to 100 °C and then keep it at room temperature for 24 h before testing.

6 EXPRESSION OF RESULTS

6.1 Calculation

Add 3 °C to the temperature recorded in 5.8.

6.2 Precision

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

6.2.1 *Repeatability*

Duplicate results by the same operator shall be considered suspect if they differ by more than 3 °C.

6.2.2 *Reproducibility*

The results submitted by each of two laboratories shall be considered suspect only if the two results differ by more than 6 °C.

6.2.3 *Special case* (see 5.9)

For oils tested by the procedure described in 5.9, reproducibility of this order cannot be expected, as these oils show anomalous pour points depending on their thermal history.

NOTE — It is a recognized property of these oils that the temperature to which they have been subjected before testing influences their pour points. Although the lower pour points as determined by the special procedure will show approximately the reproducibility given, the upper pour points will show greater variations depending on the previous thermal history of the oils.

7 TEST REPORT

Report the result as the pour point. For black oil, etc., report the results as upper pour point and/or lower pour point, as required. Indicate the method used by referring to this International Standard.