

INTERNATIONAL STANDARD



3016

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Petroleum oils — Determination of pour point

Huiles de pétrole — Détermination du point d'écoulement

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

Canada
France

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

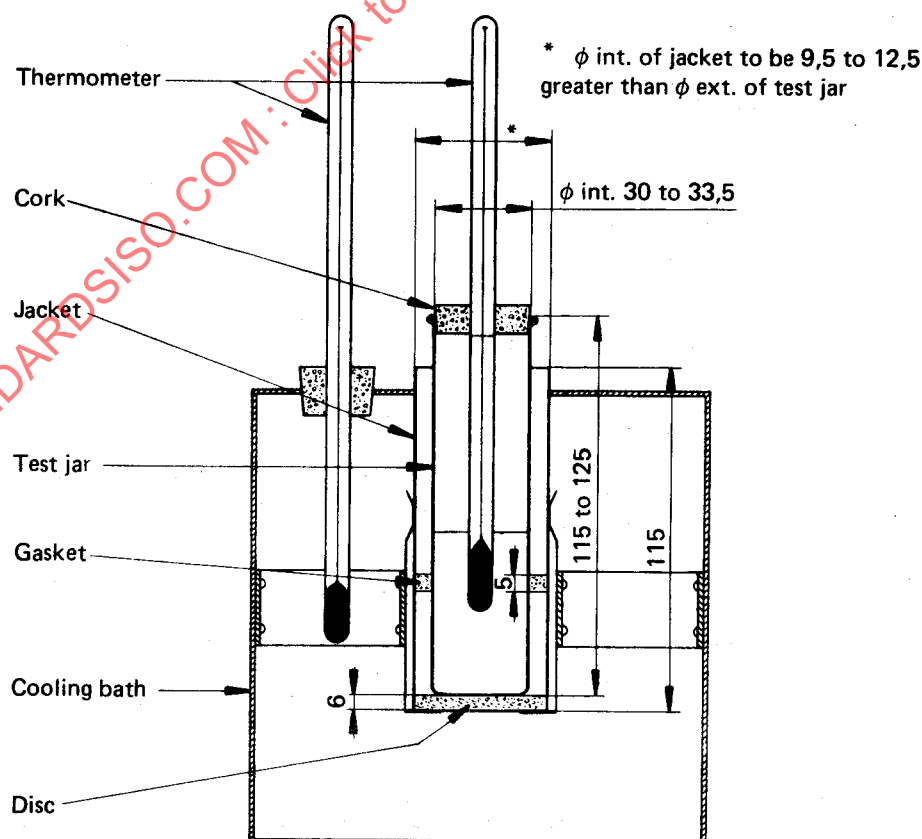


FIGURE — Apparatus for pour point test

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.