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Petroleum waxes — Determination of melting point (cooling curve)

Cires de pétrole — Détermination du point de fusion (courbe de refroidissement)

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FOREWORD

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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 3841 was drawn up by Technical Committee ISO/TC 28, *Petroleum products*, and was circulated to the Member Bodies in July 1975.

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

Australia	India	Romania
Austria	Iran	South Africa, Rep. of
Belgium	Ireland	Spain
Brazil	Italy	Sweden
Bulgaria	Japan	Turkey
Canada	Mexico	United Kingdom
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Germany	Poland	
Ghana	Portugal	

No Member Body expressed disapproval of the document.

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Petroleum waxes — Determination of melting point (cooling curve)

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies a method for the determination of the melting point (cooling curve) of petroleum wax. It is unsuitable for waxes of the petrolatum group, the microcrystalline waxes, or blends of such waxes with paraffin wax or scale wax.

1.2 Melting point (cooling curve) is a test that is widely used by wax suppliers and consumers. It is particularly applicable to petroleum waxes that are rather highly paraffinic or crystalline in nature. A plateau occurs with specimens containing appreciable amounts of hydrocarbons that crystallize at the same temperature, giving up heat of fusion, thus temporarily retarding the cooling rate. In general, petroleum waxes with large amounts of non-normal hydrocarbons or with amorphous solid forms will not show the plateau.

NOTE — For additional methods used for testing petroleum waxes, see ISO 2207, *Petroleum waxes — Determination of congealing point*, and ISO . . ., *Petroleum waxes — Determination of drop-melting point*.[1] Results may differ, depending on the method used. For pharmaceutical petrolatum, ISO . . . is usually used.

2 PRINCIPLE

A specimen of molten wax in a test tube fitted with a thermometer is placed in an air bath, which in turn is surrounded by a water bath maintained at 16 to 28 °C. As the molten wax cools, periodic readings of its temperature are taken. When solidification of the wax occurs, the rate of temperature change decreases, yielding a plateau in the cooling curve.

3 DEFINITION

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

melting point (cooling curve) of petroleum wax : The temperature at which a melted petroleum wax first shows a specified plateau in its cooling curve when allowed to cool under prescribed conditions.

4 APPARATUS

4.1 Test tube, of soda-lime glass type, of outside diameter 25 mm and length 100 mm. It may be marked with a filling line, 50 mm above the bottom, and a reference line for positioning the bottom of the thermometer at 10 mm above the bottom.

4.2 Air bath, comprising a cylinder of inside diameter 51 mm and depth 113 mm, provided with a tightly fitting cork having a central opening for holding the test tube (4.1) firmly in a vertical position in the centre of the air bath.

4.3 Water bath, comprising a suitable cylindrical vessel, of inside diameter 130 mm and depth 150 mm, provided with a fitted cover equipped to support the air bath (4.2) vertically so that the sides and bottom of the air bath are surrounded by a layer of water about 38 mm thick. The cover shall have an opening through which the bath thermometer may be suspended 20 mm from the outside wall of the water bath.

NOTE — The air bath, water bath, and water bath cover may be made in one assembly as shown in the figure.

Dimensions in millimetres

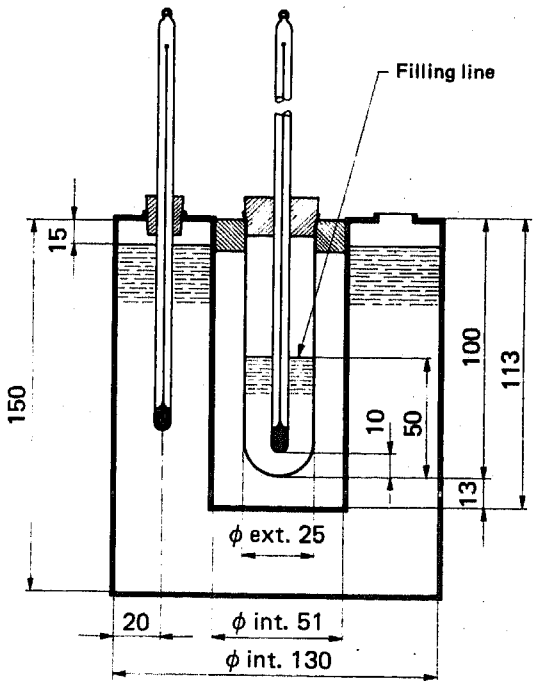

FIGURE — **Apparatus for determination of melting point (cooling curve) of petroleum wax**

1) In preparation.

4.4 **Melting-point thermometer,** partial immersion type, conforming to the following specification :

Range	38 to 82 °C
Immersion	79 mm
Graduation at each	0,1 °C
Longer line at each	0,5 °C
Figured at each	1,0 °C
Maximum scale uncertainty	0,1 °C
Expansion chamber permitting heating to	100 °C
Overall length	377 ± 5 mm
Stem diameter	6,0 to 7,0 mm
Bulb length	18 to 28 mm
Bulb diameter	5,0 to 6,0 mm
Distance from bottom of bulb to 40 °C line	116 to 125 mm
Distance from bottom of bulb to 80 °C line	315 to 335 mm
Distance from bottom of bulb to top of contraction chamber	41 mm max.

4.5 **Bath thermometer,** of suitable partial immersion type, accurate to 1 °C throughout the required range.

4.6 **Oven** or **water bath,** capable of being controlled at temperatures up to 93 °C.

5 PROCEDURE

5.1 Support the air bath (4.2) in its proper position in the water bath (4.3). Fill the water bath to within 15 mm of the top with water at a temperature of 16 to 28 °C. The bath temperature shall be kept within these limits throughout the test.

5.2 Heat the wax sample to at least 8 °C above its expected melting point (see note). To heat the wax sample, use a suitable container in the oven or water bath (4.6) which is controlled at a temperature not exceeding 93 °C. Avoid direct heat such as a flame or hot-plate. Do not keep the sample in the molten state longer than 1 h.

NOTE – If no estimate of the melting point is available, heat the wax sample to 10 °C above the temperature at which the wax is completely molten, or to a temperature of 90 to 93 °C, before proceeding to the next step.

5.3 Fill the test tube (4.1) to a height of 50 mm with the melted sample. Insert the melting-point thermometer (4.4) through the centre of a cork so that the 79 mm immersion line is at the lower surface of the cork. Insert the cork into the test tube so that the bottom of the thermometer bulb is 10 mm from the bottom of the test tube. Support the test tube assembly in the air bath as shown in the figure while the temperature of the molten wax is still at least 8 °C above its expected melting point (see note to 5.2).

5.4 Read the melting point thermometer every 15 s. Record each reading to the nearest estimated 0,05 °C. Observe the progress of these sequential readings to determine the appearance of the plateau. Identify the plateau as the first five consecutive readings all of which agree within 0,1 °C. Discontinue the test after obtaining these five plateau readings.

NOTE – If no plateau appears as defined above, continue the reading procedure until either the temperature reaches 38 °C, or the temperature reaches a point 8 °C below a temperature at which the wax has solidified (as may be observed through a transparent bath). In either of these cases, the test shall be discontinued and the method judged NOT APPLICABLE to the sample. In such cases, other methods may be selected (see note to 1.2).

6 EXPRESSION OF RESULTS

Average the first five consecutive thermometer readings of the identified plateau, which agree within 0,1 °C. Correct this average for uncertainty (error) in the thermometer scale where necessary.

7 PRECISION

The precision of the method, as obtained by statistical examination of inter-laboratory test results, is as follows.

7.1 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 the test method, exceed the following value only in one case in twenty :

0,1 °C

7.2 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 the test method, exceed the following value only in one case in twenty :

0,5 °C

8 TEST REPORT

Report the result to the nearest 0,05 °C as the melting point (cooling curve) and make reference to this International Standard.