International Standard 6244

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Petroleum waxes and petrolatums — Determination of drop melting point

Cires de pétrole et pétrolatums — Détermination du point de fusion

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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 6244 was developed by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, and was circulated to the member bodies in February 1980.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Portugal
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iraq	Spain
Bulgaria	Israel	Sweden
Canada	Italy	United Kingdom
China	Japan	USA
Czechoslovakia	Korea, Rep. of	USSR
Egypt, Arab Rep. of	Netherlands	Venezuela
France	Poland	

No member body expressed disapproval of the document.

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Petroleum waxes and petrolatums — Determination of drop melting point

1 Scope and field of application

This International Standard specifies a method for the determination of the drop melting point of petroleum waxes. Drop melting point can be an indication of the performance properties of the wax. This International Standard may be used to measure the melting characteristics of petrolatums and other high viscosity petroleum waxes.

NOTE — Additional methods used for petroleum waxes are ISO 3841, *Petroleum waxes — Determination of melting point (cooling curve)*, and ISO 2207, *Petroleum waxes — Determination of congealing point*. Results obtained may differ, depending on the method used. For pharmaceutical petrolatum, the use of this International Standard is usually preferred.

2 Principle

A film of wax is deposited on each of two thermometer bulbs by dipping chilled thermometers into the test portion. The thermometers are then placed in test tubes and heated by means of a water bath until the wax melts and the first drop falls from each thermometer bulb. The temperature at which each drop falls is read and the average of these values is the drop melting point of the sample.

3 Definition

drop melting point of petroleum wax : The temperature at which material becomes sufficiently fluid to drop from the thermometer bulb used in making the determination under standardized conditions, and reported as the mean of two determinations.

4 Apparatus

4.1 Set of two test tubes, 25 mm outside diameter and 150 mm long. Each test tube shall be closed with a cork grooved at the sides to permit air circulation and bored in the exact centre to receive a thermometer (4.3).

4.2 Water bath, consisting of a transparent container of not less than 1 500 ml capacity, capable of being heated to at least 94 °C, and that will permit the immersion of the test tubes to a depth of at least 100 mm and still leave a depth of 15 mm of water below the bottoms of the sets of test tubes.

NOTE — Other heat transfer liquids may be used in the bath, for example ethylene glycol.

4.3 Set of two thermometers, conforming to the specification in the annex.

4.4 Bath thermometer, of any suitable type, accurate to 0,5 °C throughout the required range.

4.5 Heating apparatus, of any suitable type, for heating the water bath.

5 Procedure

5.1 Secure a test sample of sufficient size (approximately 250 ml) that is representative of the material under inspection. Use a fresh test portion of the test sample for the preparation of each set of two thermometers (4.3). Melt the sample slowly until the temperature reaches 93 °C, or about 11 °C above the expected drop melting point, whichever is higher. Place sufficient test portion in a flat-bottom container to give a sample depth of 12 ± 1 mm. Adjust the temperature of the test portion to 6 to 11 °C above its drop melting point (see the note in 5.2), using any general laboratory thermometer for measurement. Chill one of the test thermometer bulbs (4.3) to 4 °C. Wipe dry and, quickly but carefully, immerse the chilled bulb vertically into the heated test portion until it touches the bottom of the container (12 mm submerged) and withdraw it immediately. Hold the thermometer vertically away from the heat until the surface dulls, and then place it for 5 min in water having a temperature of 16 °C. Prepare a second thermometer from the same test portion using the described procedure.

NOTE — A dipping temperature of 11 °C above the congealing point determined in accordance with ISO 2207 will be usually 6 to 11 °C above the actual drop melting point.

5.2 Securely fix the thermometers in the test tubes (4.1) by means of the corks so that the tip of each thermometer is 15 mm above the bottom of its test tube. Bring the temperature of the water bath (4.2) to about 16 °C. Place the test tubes in the bath and adjust the depth of immersion so that the immersion marks on the thermometers are level with the water surface. Raise the temperature of the bath at a rate of 2 °C/min to 38 °C, then at a rate of 1 °C/min until the first drop of material falls from each thermometer. Record to the nearest 0,2 °C in each case the temperature at which the first drop falls from the thermometer.

NOTE — If the drop melting point is higher than 85 °C, it is difficult to maintain the required heating rate of 1 °C/min. This does not, however, impair the use of the method up to 94 °C, above which the drop melting point as defined by the method cannot be determined.

6 Expression of results

6.1 Method of calculation

Calculate the drop melting point of the test sample by taking the average of the two determinations.

6.2 Precision

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

6.2.1 Repeatability

The difference between successive 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 0,8 °C only in one case in 20.

6.2.2 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 1,3 °C only in one case in 20.

NOTE — The above precision data have been adopted as the result of a recent re-examination of the method and supersede the following values obtained in 1954 by statistical examination of inter-laboratory results.

Repeatability	Reproducibility
1 °C	1,2 °C

7 Test report

The test report shall contain at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard or to a corresponding national standard;

c) the result of the test (see 6.1);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.

Annex

Specification for thermometers (4.3)[1][2]

Range	32 to 127 °C
Graduation	0,2 °C
Immersion, mm	79
Overall length, mm	375 to 385
Stem diameter, mm	6,0 to 8,0
Bulb shape[3]	—
Bulb length,mm	18 to 28
Bulb diameter, mm	5,0 to 6,0
Length of graduated portion, mm	200 to 240
Distance, bottom of bulb to 32 °C, mm	105 to 115
Distance, bottom of bulb to 125 °C, mm	312 to 342
Distance, top of contraction chamber to top of thermometer, mm	41
Longer lines at each	1 °C
Figured at each	2 °C
Expansion chamber to allow heating to	150 °C
Top finish	Plain
Scale error not to exceed	0,2 °C

NOTES

1 Thermometers ASTM 61C and IP 63C conform to this specification and are suitable.

2 During manufacture and testing, the average temperature of the emergent mercury column shall be 25 °C over the whole range, and any corrections resulting from the difference between the actual and specified temperature should be calculated and allowed for in determining whether or not the thermometer complies with the specification.

3 The bottom of the bulb shall be essentially hemispherical.

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