International Standard 2207

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Petroleum waxes — Determination of congealing point

Cires de pétrole — Détermination de la température de figeage

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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 2207 was developed by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, and was circulated to the member bodies in December 1979.

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

Australia	Egypt, Arab Rep. of	South Africa, Rep. of
Austria	Germany, F. R.	Spain
Belgium	Hungary	Sweden
Brazil	India	Turkey
Bulgaria	Israel	United Kingdom
Canada	Netherlands	USA
Chile	Peru	USSR
China	Poland	Venezuela
Czechoslovakia	Romania	

No member body expressed disapproval of the document.

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Petroleum waxes — Determination of congealing point

1 Scope and field of application

1.1 This International Standard specifies a method for the determination of the congealing point of petroleum waxes, including petrolatum.

1.2 The congealing point of a wax is a property of interest to many petroleum wax consumers. The procedure specified in this International Standard allows determination of the temperature at which a sample being cooled develops a "set" or resistance to flow. At that temperature, the wax may be at or close to the solid phase, or it may be semi-solid and quite unctuous, depending on the composition of the wax or petrolatum being tested. In the case of petrolatums, congealing is associated with the formation of a gel structure as the sample cools.

NOTE — This International Standard is an alternative to ISO 6244, *Petroleum waxes — Determination of drop melting point*. Results obtained are usually lower than the results obtained by ISO 6244, the amount of deviation varying with the nature of the petroleum wax.

2 Definition

congealing point : The temperature, determined by a standard test, at which molten petroleum wax ceases to flow.

3 Principle

A test portion of wax is melted and a droplet is made to adhere to the bulb of a thermometer. Using a pre-warmed flask as an air-jacket, the droplet on the bulb is allowed to cool at a fixed rate until it congeals. The congealing point is the temperature at which the droplet ceases to flow as the thermometer is turned.

4 Apparatus

4.1 Thermometer, total immersion type conforming to the specification in the annex.

4.2 Conical flask, 100 to 150 ml nominal capacity, glass, to serve as a thermometer jacket.

4.3 Cork or rubber stopper, for attaching the thermometer to the conical flask.

5 Procedure

5.1 Adjust the thermometer through the stopper so that the bottom of the bulb will be 10 to 15 mm above the bottom of the conical flask when the stopper is fitted snugly in the flask. After making this adjustment, remove the thermometer and stopper from the flask, being careful not to change the position of the stopper relative to the thermometer stem.

5.2 Place approximately 50 g of sample representative of the material under inspection, in a porcelain evaporating dish or other suitable container.

5.3 Place the empty conical flask (without the thermometer assembly) and the container holding the sample in a temperature-controlled oven set at 99 ± 3 °C until the sample and flask reach oven temperature.

NOTE — For non-referee, routine testing of samples known to have low congealing points, the oven may be set at a lower temperature, but it should be at least 11 °C above the expected congealing point of the sample.

5.4 Remove the sample from the oven and completely immerse the thermometer bulb in it without covering any part of the thermometer stem with sample. Gently stir the sample with the thermometer until the mercury column has stopped rising.

5.5 While holding the thermometer bulb in the molten wax sample, remove the heated flask from the oven, using a towel or gloves to protect the hands. Now carefully remove the thermometer from the sample, taking care to retain a relatively large drop of sample adhering to the bulb. Holding the thermometer in a horizontal position, firmly fit the thermometer and stopper into the flask. Keep the assembly in a horizontal position.

5.6 While observing the drop on the thermometer bulb at an eye-level position, rotate the thermometer and flask about a horizontal axis. Use a steady and even rate for each continuous full revolution, and complete each revolution in not less than 2 s and not more than 3 s. Do not pause at the completion of each revolution any longer than required to re-position the fingers for the next full and continuous rotation (see note). When the drop is observed to rotate with the bulb, immediately read the thermometer to the nearest 0,5 °C and record this determination. Make a repeat determination on the wax sample by repeating the above procedure, commencing at 5.3. If the difference between the results of these two determinations

does not exceed 1 °C, record the average of these results as the congealing point of the sample under test. If the difference between the results of two determinations is greater than 1 °C, make one additional determination and record the average of the results of the three determinations as the congealing point.

NOTE — Operators should periodically check themselves for compliance with this turning rate. The brief pause time should not be included in the 2 to 3 s rotation time.

6 Expression of results

6.1 Method of calculation

The congealing point is the average of the results of two or more determinations, recorded as specified in 5.6 and expressed to the nearest even-numbered decimal place.

6.2 Precision[1)]

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

6.2.1 Repeatability

The difference between successive test results (each the average of the results of two or more determinations) 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 values shown in the table only in one case in 20.

6.2.2 Reproducibility

The difference between two single and independent test results (each the average of the results of two or more determinations) 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 values shown in the table only in one case in 20.

Table — Precision

Wax type	Repeatability	Reproducibility
Distillate waxes	0,5 °C	1,5 °C
Residual waxes, including petrolatums	1,0 °C	2,5 °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 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.

1) The precision data quoted here were derived through the use of a slightly different method, ASTM D938/IP 76, which specifies reading the thermometer and reporting the results to the nearest 0,25 °C rather than 0,5 °C.

Annex

Thermometer specification

(This annex forms part of the Standard.)

Range	+ 20 to + 100 °C
Immersion	total
Graduation at each	0,5 °C
Longer lines at each	1 °C
Figured at each	5 °C
Scale error not to exceed	0,5 °C
Expansion chamber permitting heating to	110 °C
Overall length	300 ± 10 mm
Stem diameter	6,0 to 7,0 mm
Bulb shape	ellipsoidal (see figure)
Bulb length	10 to 12 mm
Bulb diameter	4,5 to 6,0 mm
Distance from bottom of bulb to 20 °C line	55 to 75 mm
Length of graduated portion	175 to 215 mm
Top finish	ring

NOTES

1 The figures shall be upright when the thermometer is held horizontally.

2 The bulb shall have a circular cross-section at all points.

3 Thermometers ASTM 54C and IP 18C conform to the above specification, except that graduation is at each 0,2 °C, and are suitable alternatives.

Dimensions in millimetres

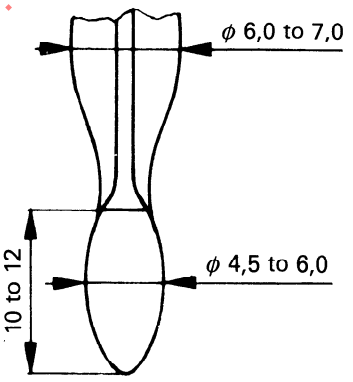

Figure — Bulb of congealing point thermometer

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