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British Standard 4698

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Standard Test Method for Cone Penetration of Petrolatum¹

This standard is issued under the fixed designation D937; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers measuring with a penetrometer the penetration of petrolatum as an empirical measure of consistency.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D217 Test Methods for Cone Penetration of Lubricating Grease

3. Terminology

3.1 *Definitions:*

3.1.1 *penetration of petrolatum*—the depth, in tenths of a millimetre, that a standard cone will penetrate the sample under fixed conditions of mass, time, and temperature.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.10.0A on Physical/Chemical Properties.

In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP Standard in 1965.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.2 *penetrometer, n*—an instrument that measures the consistency or hardness of semiliquid to semisolid materials by measuring the depth to which a specified cone or needle under a given force falls into the material.

3.1.2.1 *Discussion*—In this test method, a standard penetrometer cone (see Test Methods D217) is used to determine the consistency of petrolatum. The penetration force is determined by the total mass of the cone and shaft.

4. Summary of Test Method

4.1 The sample is completely melted by heating to a minimum temperature of 82 °C (180 °F), and then cooled under controlled conditions to 25 °C (77 °F). The penetration is measured with the cone and the sample at this temperature using a penetrometer by means of which a standard cone is applied to the sample for 5 s under a load of 150 g.

5. Significance and Use

5.1 Petrolatum is a purified mixture of semi-solid hydrocarbons obtained from petroleum and is often described as an unctuous mass. Cone penetration is a means of measuring the firmness or consistency of petrolatum. Such measurements are useful for selecting or specifying, or both, a petrolatum of a particular consistency or firmness. Cone penetration values may or may not correlate with end-use functional properties.

6. Apparatus

6.1 *Penetrometer and Cone*, as specified in Test Methods D217.

6.2 *Oven*, capable of maintaining a minimum temperature of 82 °C \pm 2 °C (180 °F \pm 5 °F) such that it is sufficient for melting the petrolatum samples. The oven shall be capable of controlling the selected temperature to within \pm 2 °C (\pm 5 °F).

6.3 *Water Bath*, constant-temperature, regulated to 25 °C \pm 0.5 °C (77 °F \pm 1 °F).

6.4 *Sample Containers*, cylindrical, having a flat bottom 100 mm \pm 6 mm (4 in. \pm 1/4 in.) in diameter and 65 mm (2 1/2 in.) or more in depth, constructed of at least 1.6 mm (16 gauge) metal and provided with a well-fitting, watertight cover.

NOTE 1—Containers of the “ointment box” type having somewhat flexible sides should not be used, for these permit slight working of the petrolatum, due to flexing of the sides in handling.

7. Preparation of Sample

7.1 Test all samples of petrolatum for original consistency after melting and cooling to the temperature of the test as described in 7.3.

7.2 If the penetration of the sample is over 200, three separate test samples are required.

7.3 Melt the sample in an oven maintained at a minimum temperature of $82\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ($180\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$) or at a higher temperature if needed to completely melt the petrolatum (see Note 2). Place the required number of sample containers in the oven along with the sample to bring them up to the same temperature. When the sample has melted and comes to the selected temperature, remove the sample and the heated containers and fill the required number of containers to within 6 mm ($\frac{1}{4}$ in.) of their rims. Allow the filled containers to cool in a location free from drafts and at a temperature controlled to $25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ($77\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$) for 16 h to 18 h. Then cover and place the samples in the water bath for 2 h to bring the temperature to $25\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$ ($77\text{ }^{\circ}\text{F} \pm 1\text{ }^{\circ}\text{F}$) before testing.

NOTE 2—Some petrolatums containing higher melting point waxes may require higher melting and pouring temperatures. If no estimate of the melting point is available, ensure that the sample is completely molten (appears visually clear) before proceeding. A hot plate may be used instead of an oven, provided that the sample temperature is carefully monitored.

8. Procedure

8.1 The surface of the sample must not be cut level nor worked in any other way, as this may affect the result. If the temperature of the penetrometer cone (that is, the room temperature) varies from $25\text{ }^{\circ}\text{C}$ by $2\text{ }^{\circ}\text{C}$ ($77\text{ }^{\circ}\text{F}$ by $3\text{ }^{\circ}\text{F}$) or more, adjust the cone temperature to $25\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$ ($77\text{ }^{\circ}\text{F} \pm 1\text{ }^{\circ}\text{F}$) immediately before testing the sample. Frequent adjustment of the cone temperature may be necessary if the room temperature varies appreciably from $25\text{ }^{\circ}\text{C}$ ($77\text{ }^{\circ}\text{F}$).

8.2 Place the can of petrolatum on the penetrometer table, so located that the tip of the penetrometer cone is 25 mm to 40 mm (1 in. to $1\frac{1}{2}$ in.) in from the rim of the container (except for petrolatums having a penetration greater than 200 (see 8.4). Observe that the cone is in its “zero” position and adjust either the indicator assembly or the table, dependent on the type of instrument, until the tip of the cone just touches the surface of the sample. Watching the shadow of the tip is an aid to accurate

setting. Finally, quickly release the plunger and hold for $5\text{ s} \pm 0.1\text{ s}$. Read the total penetration from the scale. Make at least three determinations.

8.3 With samples having penetrations less than 200, three tests (and sometimes more) may be made in one container by proper spacing. To prevent one test from being affected by the disturbed area of a previous test, the tip of the cone must not be placed nearer the edge of a previous test than the penetration distance of the sample. Some harder petrolatums tend to form a marked depression in the center on solidifying; such samples should not be tested in this depression, as the results obtained may be different from those obtained in off-center positions on the level surface.

8.4 With samples having penetrations over 200, make only one test in a container and place the cone tip as exactly as possible at the center of the container.

9. Report

9.1 Report the average of all results to the nearest 0.1 mm as the penetration, ASTM Test Method D937.

10. Precision and Bias

10.1 *Precision*—The precision of the test method as determined by statistical examination of interlaboratory results is as follows:

10.1.1 *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 values only in one case in twenty:

$2 + 5\text{ } \%$ penetration value

10.1.2 *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 values only in one case in twenty:

$9 + 12\text{ } \%$ penetration value

10.2 *Bias*—The procedure in this test method has no bias because the value of cone penetration can be defined only in terms of a test method.

11. Keywords

11.1 core penetration; penetrometer; petrolatum

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