Designation: D1321 – 10

Standard Test Method for Needle Penetration of Petroleum Waxes[1]

This standard is issued under the fixed designation D1321; 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 Department of Defense.

1. Scope*

1.1 This test method covers the empirical estimation of the consistency of waxes derived from petroleum by measurement of the extent of penetration of a standard needle. This test method is applicable to waxes having a penetration of not greater than 250.

NOTE 1—This test method is similar to the needle method for determining the penetration of bituminous material, Test Method D5. Cone methods applicable to greases and to petrolatum are described in Test Methods D217 and Test Method D937, respectively.

1.2 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D5 Test Method for Penetration of Bituminous Materials

D87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)

D217 Test Methods for Cone Penetration of Lubricating Grease

D937 Test Method for Cone Penetration of Petrolatum

D938 Test Method for Congealing Point of Petroleum Waxes, Including Petrolatum

E1 Specification for ASTM Liquid-in-Glass Thermometers

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions:*

3.1.1 *penetration*, *n*—*of petroleum wax*, the depth in tenths of a millimetre to which a standard needle penetrates into the wax under defined conditions.

3.1.1.1 *Discussion*—As an example, a penetration reading of 85 from the indicator scale corresponds to a penetration depth of 8.5 mm.

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 needle (6.3) is used to determine the hardness of petroleum wax. The penetration force is determined by the total mass (100 g) of the needle, plunger, and 50 g weight.

4. Summary of Test Method

4.1 The sample is heated to at least 17°C (30°F) above its expected congealing point or melting point, poured into a container, and then air cooled under controlled conditions. The sample then is conditioned at test temperature in a water bath. Penetration is measured with a penetrometer, which applies a standard needle to the sample for 5 s under a load of 100 g.

5. Significance and Use

5.1 Petroleum waxes differ in hardness. Needle penetration is a measurement of hardness. Hardness may have a significant effect upon other physical properties.

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

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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.

***A Summary of Changes section appears at the end of this standard.**

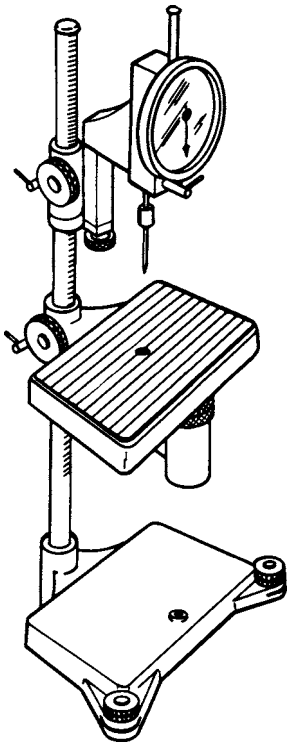

FIG. 1 Penetrometer

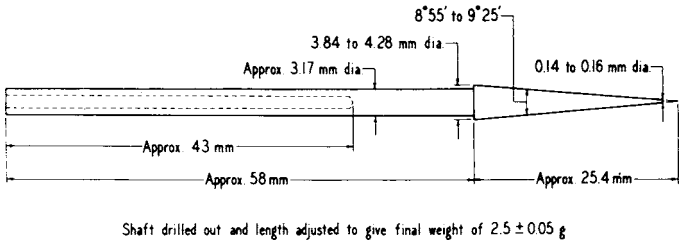

FIG. 2 Standard Needle

6. Apparatus

6.1 *Penetrometer*, for applying the standard needle to the surface of the sample specimen and for measuring the extent of penetration at the conclusion of the test. The penetrometer shall be constructed in such a manner that the accurate placement of the tip of the needle at the level surface of the specimen may be made while maintaining a "zero" reading on the indicator. The apparatus shown in Fig. 1 represents a composite drawing illustrating the two available types of instrument, one with an adjustable table and the other with an adjustable needle assembly; the use of either type of instrument is permissible. The loaded needle must fall, when released, without appreciable friction. The instrument shall be provided with leveling screws and a spirit level to maintain the plunger shaft in a true vertical position. The indicator scale shall be calibrated in tenths of a millimetre division and shall have a range of at least 250 tenths of millimetres.

6.2 *Timing Device*—An automatic timing release mechanism attached to the penetrometer may be used. Alternatively, a stop watch graduated in 0.1-s intervals may be used.

6.3 *Needle and Plunger*—The needle shall be approximately 83 mm in length and conform to the dimensions shown in Fig. 2. It shall be symmetrically tapered at one end to a cone whose angle shall be within the range from 8°, 55 min to 9°, 25 min over the entire length of the cone. The axis of the cone shall be coincident with the shaft axis within 0.13-mm (0.005-in.) maximum runout (total indicator reading). The tapered section of the needle shall be made from fully hardened and tempered stainless steel, Grade 440-C or equal, Rockwell hardness C57 to 60. After tapering, the point shall be ground off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm in diameter. The truncation shall be square with the needle axis within 2°, and the edge shall be sharp and free from burrs. The conical surface and the truncation shall be finished to a smoothness of 0.2 µm (8 µin.) (rms). The final weight of the needle shall be 2.5 ± 0.05 g. The total weight of the plunger shall be 47.5 ± 0.05 g; a weight of 50 ± 0.05 g is required for mounting on the plunger.

NOTE 2—The National Institute of Standards and Technology will measure and certify the accuracy of penetration needles in accordance with these permissible variations.

6.4 *Test Specimen Container*, consisting of a brass cylinder open at both ends, having a 25.4 ± 1.6-mm (1 ± 1/16-in.) inside diameter, 31.8 ± 1.6-mm (1¼ ± 1/16-in.) height, and 3.2 ± 1.6-mm (⅛ ± 1/16-in.) wall thickness. To prevent slippage of very hard wax, a few screw threads or grooves shall be cut into the center part of the inside wall of the cylinder. The cylinder shall be placed on a base plate of brass, wetted with an equal volume mixture of glycerin and water, when casting a test specimen.

6.5 *Test Room or Cabinet*, capable of being maintained at 23.9 ± 2.2°C (75 ± 4°F).

6.6 *Water Bath*, of at least 10-L capacity, capable of being maintained at the test temperature within ±0.1°C (±0.2°F) (Note 4). The water bath should be made of glass or other suitable transparent material, or have a window to permit a horizontal view of the specimen. It shall be possible to immerse the test specimen in the bath to a depth of not less than 102 mm (4 in.) and to support it on a perforated conditioning shelf not less than 51 mm (2 in.) from the bottom of the bath. The bath also shall be equipped with a rigid perforated test shelf about 51 mm below the water level to support the specimen during the penetration by the needle.

6.7 *Thermometer*, for use in the water bath. An ASTM Precision Thermometer, total immersion, having a range from 25 to 55°C or 77 to 131°F and conforming to the requirements for Thermometer 64C or 64F as prescribed in Specification E1 or Thermometer S64C or S64F as prescribed in Specification E2251.

6.8 *Brass Plate*, 63.5 ± 1.6 mm by 38 ± 1.6 mm by 6.4 ± 1.6 mm (2½ ± 1/16 in. by 1½ ± 1/16 in. by ¼ ± 1/16 in.) for supporting test specimen during preparation of the sample. The specimen support is placed on an insulating material, such as corks or rubber stoppers during the cooling period.

7. Preparation of Test Specimen

7.1 Heat the wax sample to at least 17°C (30°F) above its expected congealing point or melting point (as determined by Test Method D938 or Test Method D87, respectively), using care to prevent local overheating. Make sure the sample is

homogeneous and free from air bubbles. In the test room or cabinet maintained at 23.9 ± 2.2°C (75 ± 4°F), place the brass plate on a stable support, such as stoppers or corks, and wet the upper surface of the plate with a mixture of equal volumes of glycerin and water. Place the test specimen container on the plate and then pour the melted wax into it in such a way that a convex meniscus is formed. Allow the container and contents to cool in the room at 23.9 ± 2.2°C for 1 h. Then shave any excess wax from the top of the container and remove the brass plate. Place the smooth wax surface up. Condition the specimen in the bath at the test temperature within 0.1°C (0.2°F) for 1 h.

NOTE 3—Very hard waxes occasionally will shrink away from the walls of the test specimen container; in such cases, it is permissible to wedge the specimen in the container.

8. Procedure

8.1 Reverse the penetrometer base and place the penetrometer head over the edge of the water bath and above the perforated test shelf used for supporting the specimen. It may be necessary to place a weight on the base of the penetrometer to counterbalance the head (Note 5). Level the penetrometer and the perforated shelf in the water bath.

NOTE 4—Alternatively, the penetrometer may be placed in the water bath. Likewise, a small bath may be placed on the penetrometer stand provided the test temperature (within 0.1°C (±0.2°F)) and the required water circulation above and below the test specimen are maintained and provided further that the temperature of the small bath is measured immediately before testing each specimen using the thermometer specified in 6.7. Emergent stem corrections shall be applied when the correction equals or exceeds 0.05°C (0.1°F). One of the above alternatives will be required if the penetrometer is the adjustable table type.

8.2 Place the specimen container on the perforated test shelf with the smooth wax surface that had contacted the brass plate at the top. Make certain that the container or test shelf cannot teeter during testing. Adjust the water level so that it is at least 25 mm (1 in.) above the top surface of the specimen and maintain it at the test temperature.

NOTE 5—The test may be performed at any temperature in the range from 25 to 55°C (77 to 130°F). Temperatures 25, 35, 45, or 50°C (77, 95, 113, or 122°F) normally are used.

8.3 Place a 50-g weight above the penetrometer needle, making a total load of 100 ± 0.15 g for the needle and all attachments. Observe that the release mechanism does not drag on the shaft and that the indicator on the scale is in the "zero" position. Adjust either the indicator assembly or the table, depending upon the type of instrument, until the tip of the needle nearly touches the surface of the specimen. Securely lock the movable assembly in this position.

8.4 Then, by means of the slow-motion adjustment, bring the needle tip to just touch the surface of the specimen, watching the reflection of the needle tip as an aid to accurate setting. After ensuring the bath temperature is within the proper specifications, release the needle shaft and hold it free for 5.0 ± 0.1 s, timing this interval automatically or with a stop watch graduated to 0.1 s. Then gently depress the indicator shaft until it is stopped by the needle shaft and read the penetration from the indicator scale.

TABLE 1 Repeatability and Reproducibility Calculated for Different Values of Penetration

Penetration, $\bar{x}$	Repeatability	Reproducibility
10	2	5
20	2	6
30	2	7
40	3	7
50	3	8
60	4	9
70	4	10
80	5	11
90	5	12
100	6	13
125	8	17
150	11	22
175	14	29
200	19	37

8.5 Make four tests at points about equally spaced (not less than 12.7 mm (½ in.) apart) on a circumference at least 3.2 mm (⅛ in.) from the side of the container. Before each test, wipe the needle carefully toward its point with a clean, dry cloth to remove all adhering wax, position the needle as described in 8.4, and proceed with the test.

9. Report

9.1 Record as a single test value the average scale reading for the four penetrations on the prepared specimen and report to the nearest penetration reading (see 3.1.1). Also report the actual test temperature used.

10. Precision and Bias [3]

10.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.1.1 *Repeatability*—The difference between successive 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 in only one case in twenty.

$$\text{Repeatability} = 1.72\,[10^{0.005\,24\,(\bar{x})}] \quad (1)$$

where:

$\bar{x}$ = penetration.

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, exceed the following values in only one case in twenty.

$$\text{Reproducibility} = 4.81\,[10^{0.004\,42\,(\bar{x})}] \quad (2)$$

where:

$\bar{x}$ = penetration.

10.1.3 The repeatability and reproducibilities for the different penetration values calculated from the above equations are shown in Table 1.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1205.

10.2 *Bias*—The procedure in Test Method D1321 for measuring the needle penetration of petrolatum waxes has no bias because the value of the needle penetration can be defined only in terms of a test method.

11. Keywords

11.1 hardness; needle penetration; penetration; petroleum wax

SUMMARY OF CHANGES

Subcommittee D02.10 has identified the location of selected changes to this standard since the last issue (D1321–04) that may impact the use of this standard.

(*1*) Added Specification E2251.

(*2*) Revised 6.7.