



Designation: **D1321—16 D1321 – 16a**

Standard Test Method for Needle Penetration of Petroleum Waxes¹

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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

1.3.1 *Exception*—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.

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.

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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.

3.1.1.1 *Discussion*—

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

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

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.

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

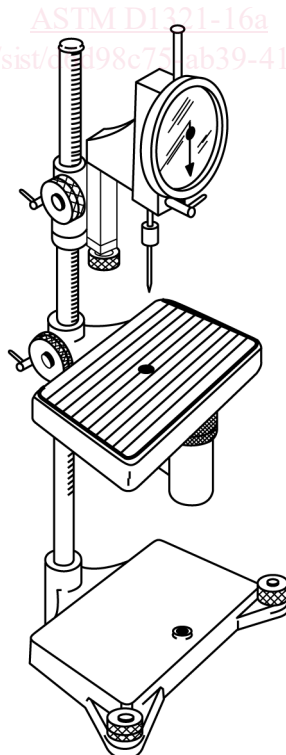
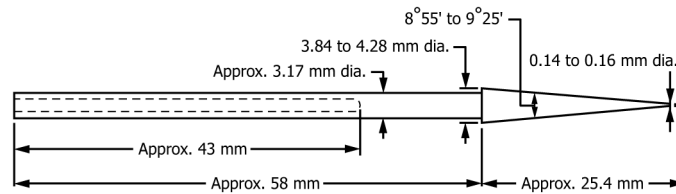


FIG. 1 Penetrometer



Shaft drilled out and length adjusted to give final weight of 2.5 ± 0.05 g

FIG. 2 Standard Needle

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 mm 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 $0.2 \mu\text{m}$ ($8 \mu\text{in.}$) (rms). The final weight of the needle shall be $2.5 \text{ g} \pm 0.05 \text{ g}$. The total weight of the plunger shall be $47.5 \text{ g} \pm 0.05 \text{ g}$; a weight of $50 \text{ g} \pm 0.05 \text{ g}$ 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 $25.4 \text{ mm} \pm 1.6 \text{ mm}$ (1 in. $\pm 1/16$ in.) inside diameter, $31.8 \text{ mm} \pm 1.6 \text{ mm}$ ($1\frac{1}{4}$ in. $\pm 1/16$ in.) height, and $3.2 \text{ mm} \pm 1.6 \text{ mm}$ ($1/8$ in. $\pm 1/16$ 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 $23.9 \text{ }^\circ\text{C} \pm 2.2 \text{ }^\circ\text{C}$ ($75 \text{ }^\circ\text{F} \pm 4 \text{ }^\circ\text{F}$).

6.6 *Water Bath*, of at least 10 L capacity, capable of being maintained at the test temperature within $\pm 0.1 \text{ }^\circ\text{C}$ ($\pm 0.2 \text{ }^\circ\text{F}$) (Note 45). 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 $25 \text{ }^\circ\text{C}$ to $55 \text{ }^\circ\text{C}$ or $77 \text{ }^\circ\text{F}$ to $131 \text{ }^\circ\text{F}$ 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.

6.7.1 Thermometric devices such as resistance temperature detectors (RTDs), thermistors, or other liquid-in-glass thermometers of equal or better accuracy, and covering the appropriate temperature range, may be used.

6.8 *Brass Plate*, $63.5 \text{ mm} \pm 1.6 \text{ mm}$ by $38 \text{ mm} \pm 1.6 \text{ mm}$ by $6.4 \text{ mm} \pm 1.6 \text{ mm}$ ($2\frac{1}{2}$ in. $\pm 1/16$ in. by $1\frac{1}{2}$ in. $\pm 1/16$ in. by $1/4$ in. $\pm 1/16$ 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 $17 \text{ }^\circ\text{C}$ ($30 \text{ }^\circ\text{F}$) 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 $23.9 \text{ }^\circ\text{C} \pm 2.2 \text{ }^\circ\text{C}$ ($75 \text{ }^\circ\text{F} \pm 4 \text{ }^\circ\text{F}$), 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 $23.9 \text{ }^\circ\text{C} \pm 2.2 \text{ }^\circ\text{C}$ for 1 h. Then After 1 h of cooling, 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 $0.1 \text{ }^\circ\text{C}$ ($0.2 \text{ }^\circ\text{F}$) 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.

NOTE 4—The sample temperature prior to pouring into the test specimen container can impact the wax tunneling severity. For good practice, it is suggested that sample is cooled to $\sim 17 \text{ }^\circ\text{C}$ to $20 \text{ }^\circ\text{C}$ above its melting point prior to pouring into the test specimen container.