Designation: D 1321 – 02

An American National Standard

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

This standard is issued under the fixed designation D 1321; 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 D 5. Cone methods applicable to greases and to petrolatum are described in Test Methods D 217 and Test Method D 937, respectively.

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

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 5 Test Method for Penetration of Bituminous Materials[2]

D 217 Test Methods for Cone Penetration of Lubricating Grease[3]

D 937 Test Method for Cone Penetration of Petrolatum[3]

D 938 Test Method for Congealing Point of Petroleum Waxes, Including Petrolatum[3]

E 1 Specification for ASTM Thermometers[4]

## 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.

3.1.2 *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 melted, heated to 17°C (30°F) above its congealing 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

[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 on Properties of Petroleum Wax.

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[2] *Annual Book of ASTM Standards*, Vol 04.03.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

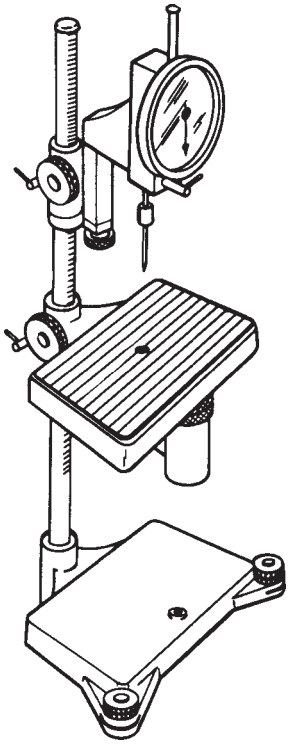

FIG. 1 Penetrometer

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-mm (1-in.) inside diameter, 31.8-mm (1¼-in.) height, and 3.2-mm (⅛-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.

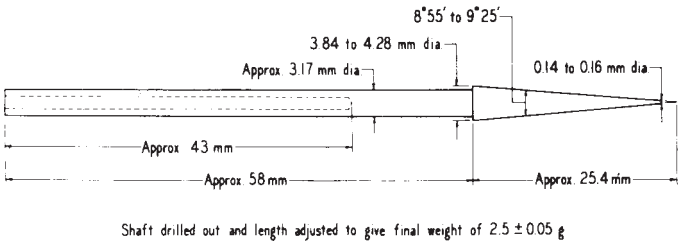


FIG. 2 Standard Needle

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 6). 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 E 1.

6.8 *Brass Plate*, 63.5 by 38 by 6.4 mm (2½ by 1½ by ¼ in.) for supporting test specimen during preparation of the sample. The specimen support is placed on corks during the cooling period.

## 7. Preparation of Test Specimen

7.1 Heat the wax sample to approximately 17°C (30°F) above its congealing point (as determined by Test Method D 938), using care to prevent local overheating. Stir until 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 two corks (No. 16 or 18 recommended) 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