Designation: D 70 – 09$^{\varepsilon 1}$

# Standard Test Method for Density of Semi-Solid Bituminous Materials (Pycnometer Method)[1]

This standard is issued under the fixed designation D 70; 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.*

ε[1] NOTE—Values in Table 1 were editorially corrected in July 2009.

## 1. Scope*

1.1 This test method covers the determination of the relative density and density of semi-solid bituminous materials, asphalt cements, and soft tar pitches by use of a pycnometer.

NOTE 1—An alternate method for determining the density of semi-solid and solid bituminous materials is Test Method D 3289. For materials which are too fluid for use of this test method, use Test Method D 3142.

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

1.3 *This standard does not purport to address 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]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

D 140 Practice for Sampling Bituminous Materials

D 3142 Test Method for Specific Gravity, API Gravity, or Density of Cutback Asphalts by Hydrometer Method

D 3289 Test Method for Density of Semi-Solid and Solid Bituminous Materials (Nickel Crucible Method)

D 4311 Practice for Determining Asphalt Volume Correction to a Base Temperature

E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Other:*

CRC Handbook of Chemistry and Physics

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *density*—the mass per unit volume of a material.

3.1.2 *relative density*—the ratio of the mass of a given volume of a material to the mass of the same volume of water at the same temperature (see Note 2).

NOTE 2—Relative density is also described as specific gravity.

## 4. Summary of Test Method

4.1 The sample is placed in a calibrated pycnometer. The pycnometer and sample are weighed, then the remaining volume is filled with water. The filled pycnometer is brought to the test temperature, and weighed. The density of the sample is calculated from its mass and the mass of water displaced by the sample in the filled pycnometer.

## 5. Significance and Use

5.1 Values of density are used for converting volumes to units of mass, and for correcting measured volumes from the temperature of measurement to a standard temperature using Practice D 4311.

## 6. Apparatus

6.1 *Pycnometer*, glass, consisting of a cylindrical or conical vessel carefully ground to receive an accurately fitting glass stopper 22 to 26 mm in diameter. The stopper shall be provided with a hole 1.0 to 2.0 mm in diameter, centrally located in reference to the vertical axis. The top surface of the stopper shall be substantially plane and have no chips, and the lower

[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

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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.**

surface shall be concave to allow all air to escape through the bore. The height of the concave section shall be 4.0 to 18.0 mm at the center. The stoppered pycnometer shall have a capacity of 24 to 30 mL and shall weigh not more than 40 g. Suitable pycnometers are illustrated in Fig. 1.

6.2 *Water Bath*, constant-temperature, capable of maintaining the temperature within 0.1°C (0.2°F) of the test temperature.

6.3 *Thermometric device*, calibrated liquid in glass, total immersion type, of suitable range with graduations at least every 0.1°C (0.2°F) and a maximum scale error of 0.1°C (0.2°F) as prescribed in Specification E 1. Thermometer commonly used is ASTM 63°C (63F). Any other thermometric device of equal accuracy may be used.

6.4 *Balance*, capable of making the required measurements to an accuracy of at least 0.001g.

6.5 *Beaker*, 600 mL or larger Griffin low-form beaker.

## 7. Materials

7.1 *Water*—Freshly boiled and cooled distilled or deionized water.

## 8. Hazards

8.1 **Warning:** Mercury has been designated by the United States Environmental Protection Agency (EPA) and many state 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—www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury, mercury containing products, or both, into your state may be prohibited by state law.

## 9. Sampling

9.1 Take samples in accordance with Practice D 140. The sample shall be free of foreign substances.

9.2 Thoroughly mix the sample before removing a representative portion for testing.

## 10. Preparation of Apparatus

10.1 Partially fill a 600-mL or larger Griffin low-form beaker with freshly boiled and cooled distilled or deionized water to a level that will allow the top of the pycnometer to be immersed to a depth of not less than 40 mm.

10.2 Partially immerse the beaker in the water bath to a depth sufficient to allow the bottom of the beaker to be immersed to a depth of not less than 100 mm, while the top of the beaker is above the water level of the bath. Utilize some method to ensure that the beaker does not tip over, while making sure that circulation of the water in the conditioning bath around the beaker is not restricted.

10.3 Maintain the temperature of the water bath within 0.1°C (0.2°F) of the test temperature.

## 11. Calibration of Pycnometer

11.1 Thoroughly clean, dry, and weigh the pycnometer to the nearest 0.001 g. Designate this mass as *A*.

11.2 Remove the beaker from the water bath if necessary. Fill the pycnometer with freshly boiled distilled or deionized water, placing the stopper loosely in the pycnometer. Place the pycnometer in the beaker and press the stopper firmly in place. Return the beaker to the water bath if previously removed.

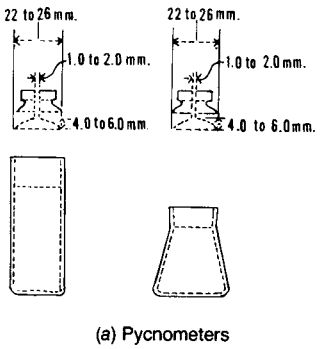
NOTE 3—Calibration must be done at the test temperature. A pycnometer calibrated at one temperature cannot be used at a different temperature without recalibration at that temperature.

11.3 Allow the pycnometer to remain in the water for a period of not less than 30 min. Remove the pycnometer, immediately dry the top of the stopper with one stroke of a dry towel (Note 4), then quickly dry the remaining outside area of the pycnometer and weigh to the nearest 0.001 g. Designate the mass of the pycnometer plus water as *B*.

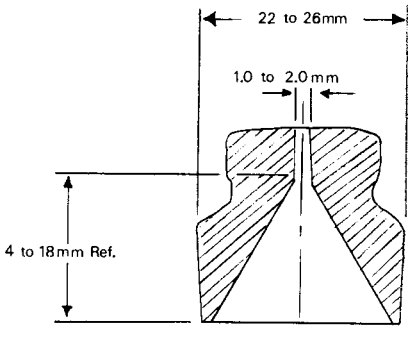
NOTE 4—Do not redry the top of the stopper even if a small droplet of water forms as a result of expansion. If the top is dried at the instant of removing the pycnometer from the water, the proper mass of the contents at the test temperature will be recorded. If moisture condenses on the pycnometer during weighing, quickly redry the outside of the pycnometer (excluding the top) before recording the mass.

## 12. Procedure

12.1 *Preparation of Sample*—Heat the sample with care, stirring to prevent local overheating, until the sample has become sufficiently fluid to pour. In no case should the temperature be raised to more than 55°C (131°F) above the expected softening point for tar, or to more than 110°C (230°F)




(*a*) Pycnometers




(*b*) Stopper

**FIG. 1 Suitable Pycnometers and Stopper**

above the expected softening point for asphalt. Do not heat for more than 60 min, and avoid incorporating air bubbles into the sample.

12.2 Pour enough sample into the clean, dry, warmed pycnometer to fill it about three fourths of its capacity. Take precautions to keep the material from touching the sides of the pycnometer above the final level and prevent the inclusion of air bubbles (Note 5). Allow the pycnometer and its contents to cool to ambient temperature for a period of not less than 40 min and weigh with the stopper to the nearest 0.001 g. Designate the mass of the pycnometer plus sample as *C*.

NOTE 5—If any air bubbles are inadvertently occluded, remove by brushing the surface of the asphalt in the pycnometer with a high "soft" flame of a bunsen burner or torch. To avoid overheating, do not allow the flame to remain in contact with the asphalt more than a few seconds at any one time.

12.3 Remove the beaker from the water bath if necessary. Fill the pycnometer containing the asphalt with freshly boiled distilled or deionized water, placing the stopper loosely in the pycnometer. Do not allow any air bubbles to remain in the pycnometer. Place the pycnometer in the beaker and press the stopper firmly in place. Return the beaker to the water bath if previously removed.

12.4 Allow the pycnometer to remain in the water bath for a period of not less than 30 min. Remove the pycnometer from the bath. Dry and weigh using the same technique and timing as that employed in 11.3. Designate this mass of pycnometer plus sample plus water as *D*.

## 13. Calculation

13.1 Calculate the relative density to the nearest 0.001 as follows:

$$\text{relative density} = (C - A)/[(B - A) - (D - C)] \quad (1)$$

where:

$A$ = mass of pycnometer (plus stopper),
$B$ = mass of pycnometer filled with water,
$C$ = mass of pycnometer partially filled with asphalt, and
$D$ = mass of pycnometer plus asphalt plus water.

13.2 Calculate density to the nearest 0.001 as follows:

$$\text{Density} = \text{relative density} \times W_T \quad (2)$$

where:

$W_T$ = density of water at the test temperature (Note 6).

NOTE 6—Density of water from CRC Handbook of Chemistry and Physics:

| Temperature, °C | Density of Water, kg/m$^3$(kg/L) |
|---|---|
| 15.6 | 999.0 (0.9990) |
| 25.0 | 997.0 (0.9970) |

## 14. Report

14.1 Report density to the nearest 1 kg/m$^3$(0.001 kg/L) and the test temperature.

## 15. Precision and Bias

15.1 *Single Operator Precision*—The single-operator pooled standard deviation for the relative density (specific gravity) of semi-solid bituminous materials has been found to be 0.0013 at 15.6°C (60°F) and 0.00082 at 25.0°C (77°F) (see Table 1). Therefore, results of two properly conducted tests by the same operator on the same material should not differ by more than the following values (see Note 7):

| Test Temperature, °C | Density, kg/m$^3$(kg/L) |
|---|---|
| 15.6 | 3.7 (0.0037) |
| 25.0 | 2.3 (0.0023) |

15.2 *Multilaboratory Precision*—The multilaboratory pooled standard deviation for the relative density (specific gravity) of semi-solid bituminous materials has been found to be 0.0024 at 15.6°C and 0.0019 at 25.0°C (see Table 1). Therefore, results of two properly conducted tests by two laboratories on samples of the same material should not differ by more than the following values (see Note 6):

| Test Temperature, °C | Density, kg/m$^3$(kg/L) |
|---|---|
| 15.6 | 6.8 (0.0068) |
| 25.0 | 5.4 (0.0054) |

NOTE 7—These number represent, respectively, the (1S) and (D2S) limits as described in Practice C 670.

15.3 *Bias*—No information can be presented on the bias of the procedure in this test method for measuring density because no material having an accepted reference value is available.

## 16. Keywords

16.1 density; pycnometer; relative density; specific gravity

**TABLE 1 Precision of Relative Density Data for Semi-Solid Bituminous Materials**

| | Temperature °C | Single-Operator | | | Multilaboratory | | |
|---|---|---|---|---|---|---|---|
| | | Degrees of Freedom | (1S) | (D2S) | Degrees of Freedom | (1S) | (D2S) |
| Asphalt | 15.6 | 54 | 0.0011 | 0.0032 | 24 | 0.0018 | 0.0051 |
| | 25.0 | 54 | 0.00080 | 0.0023 | 24 | 0.0024 | 0.0068 |
| Soft tar pitch | 15.6 | 72 | 0.0013 | 0.0038 | 27 | 0.0029 | 0.0083 |
| | 25.0 | 72 | 0.00083 | 0.0023 | 27 | 0.0017 | 0.0048 |
| Pooled values | 15.6 | 114 | 0.0013 | 0.0037 | 51 | 0.0024 | 0.0068 |
| | 25.0 | 114 | 0.00082 | 0.0023 | 51 | 0.0019 | 0.0053 |

## SUMMARY OF CHANGES

Committee D04 has identified the location of selected changes to this standard since the last issue (D 70 – 08) that may impact the use of this standard. (Approved June 1, 2009.)

(*1*) Updated Note 6, 15.1, and 15.2.