Designation: D 70 – 97

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.

1. Scope

1.1 This test method covers the determination of the specific gravity 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 in SI units are to be regarded as the standard.

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:*

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[2]

D 140 Practice for Sampling Bituminous Materials[3]

D 3142 Test Method for Density of Liquid Asphalts (Hydrometer Method)[3]

D 3289 Test Method for Density of Semi-Solid and Solid Bituminous Materials (Nickel Crucible Method)[3]

D 4311 Practice for Determining Asphalt Volume Correction to a Base Temperature[3]

E 1 Specification for ASTM Thermometers[4]

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 smooth and substantially plane, and the lower 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 of the test temperature.

6.3 *Thermometers*, calibrated liquid in glass, total immersion type, of suitable range with graduations at least every 0.1°C and a maximum scale error of 0.1°C as prescribed in Specification E 1. Thermometers commonly used are 63°F or 63°C. Any other thermometer device of equal accuracy may be used.

7. Materials

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

8. Sampling

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

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[1] This test method is under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

Current edition approved Aug. 10, 1997. Published April 1998. Originally published as D 70 – 20. Last previous edition D 70 – 82 (1990).

[2] *Annual Book of ASTM Standards*, Vol 04.02.

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

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

FIG. 1 Suitable Pycnometers and Stopper

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

9. Preparation of Apparatus

9.1 Partially fill a 600-mL 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.

9.2 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. Clamp the beaker in place.

9.3 Maintain the temperature of the water bath within 0.1°C of the test temperature.

10. Calibration of Pycnometer

10.1 Thoroughly clean, dry, and weigh the pycnometer to the nearest 1 mg. Designate this mass as *A*.

10.2 Remove the beaker from the water bath. 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.

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.

10.3 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 1 mg. Designate the mass of the pycnometer plus water as *B*.

NOTE 4—Do not redry the top of the stopper even 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.

11. Procedure

11.1 *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 56°C above the expected softening point for tar, or to more than 110°C above the expected softening point for asphalt. Do not heat for more than 30 min, and avoid incorporating air bubbles into the sample.

11.2 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 1 mg. 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. To avoid overheating, do not allow the flame to remain in contact with the asphalt more than a few seconds at any one time.

11.3 Remove the beaker from the water bath. 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.

11.4 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 10.3. Designate this mass of pycnometer plus sample plus water as *D*.

12. Calculation

12.1 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.

12.2 Calculate density to the nearest 0.001 as follows:

$$\text{Density} = \text{specific gravity} \times W_T \quad (2)$$

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

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

Temperature, °C	Density of Water, kg/m^3
15.0	999.1
25.0	997.0

13. Report

13.1 Report density to the nearest 1 kg/m^3 and the test temperature.

14. Precision and Bias

14.1 *Single Operator Precision*—The single-operator standard deviation for the relative density of semi-solid bituminous materials has been found to be 0.0013 at 15.6°C and 0.00082 at 25.0°C. 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 6):

Test Temperature, °C	Density, kg/m^3
15.0	3.7
25.0	2.3

14.2 *Multilaboratory Precision*—The multilaboratory standard deviation for the relative density of semi-solid bituminous materials has been found to be 0.0024 at 15.6°C and 0.0019 at 25.0°C. 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
15.0	6.8
25.0	5.4

TABLE 1 Precision of Specific Gravity Data for Semi-Solid Bituminous Materials

	Temperature °F [°C]	Single-Operator			Multilaboratory		
		Degrees of Freedom	(1S)	(D2S)	Degrees of Freedom	(1S)	(D2S)
Asphalt	60 [15.6]	54	0.0011	0.0032	24	0.0018	0.0051
	77 [25.0]	54	0.00080	0.0023	24	0.0024	0.0068
Soft tar pitch	60 [15.6]	72	0.0013	0.0038	27	0.0029	0.0083
	77 [25.0]	72	0.00083	0.0023	27	0.0017	0.0048
Pooled values	60 [15.6]	114	0.0013	0.0035	51	0.0024	0.0067
	77 [25.0]	114	0.00082	0.0023	51	0.0019	0.0053