Designation: D 3289 – 03

Standard Test Method for Density of Semi-Solid and Solid Bituminous Materials (Nickel Crucible Method)[1]

This standard is issued under the fixed designation D 3289; 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.

1. Scope

1.1 This test method covers the determination of the density of semi-solid and solid bituminous materials by weighing in air and in water.

NOTE 1—An alternate method for determining the density of semi-solid and solid bituminous materials is Test Method D 70. For materials which are too fluid for use of this method, use Test Method D 3142.

1.2 *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 70 Test Method for Density of Semi-Solid and Solid Bituminous Materials (Pycnometer Method)[3]

D 140 Practice for Sampling Bituminous Materials[3]

D 3142 Test Method for Density of Liquid Asphalts (Hydrometer 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 *density*—the mass per unit volume of a material.

3.2 *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. (Note 2)

NOTE 2—Relative density is also called specific gravity.

4. Summary of Test Method

4.1 The sample is placed in a nickel crucible and weighed in air, and then in water at the test temperature. The density is calculated from the mass of the sample and its apparent mass when weighed in water.

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 *Crucible*, nickel, high-form, 30-mL capacity, about 43 mm in height by 41 mm in diameter.

6.2 *Bath*, constant-temperature, capable of maintaining the test temperature within ±0.1°C.

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

6.4 *Balance*, capable of weighing to 0.001 g. It shall be fitted with a pan straddle and a wire basket, as shown in Fig. 1.

6.5 *Basket and Pan Straddle*, capable of holding the crucible in air or in water by suspending from the balance beam, as shown in Fig. 1. The basket may be made out of soft 0.81-mm copper wire or equivalent. The straddle may be made out of about 0.81-mm aluminum sheeting formed to provide support of the beaker while permitting free movement of the pan.

7. Material

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 D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

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[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.

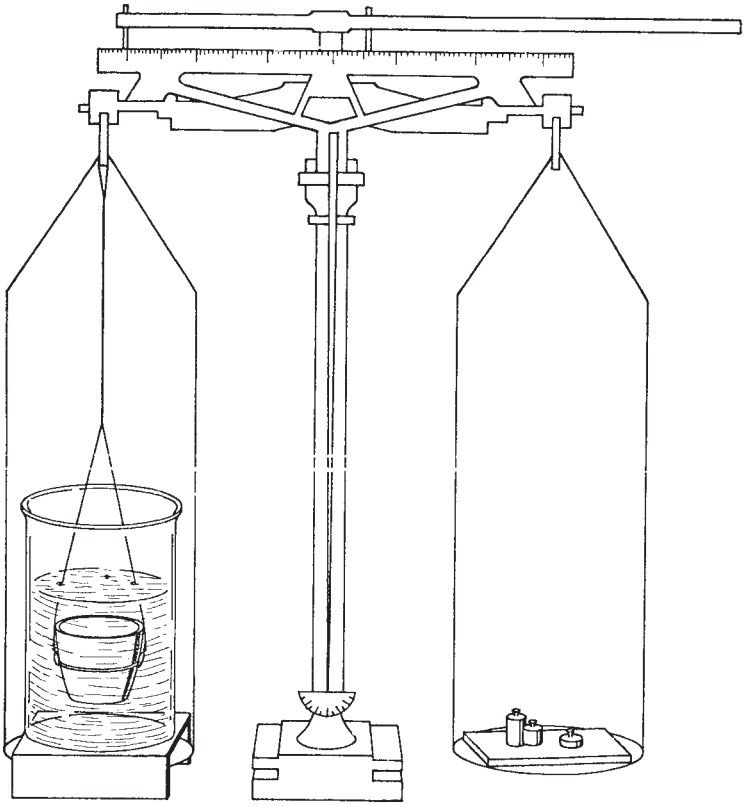

FIG. 1 Analytical Balance Equipped with Crucible Holder, Immersion Beaker, and Pan Straddle

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

9. Procedure

9.1 Place the clean, dry nickel crucible in the wire basket and suspend the basket from one arm of the balance. Weigh to the nearest 1 mg and record the combined mass as W_1.

9.2 Fill a 400-mL Griffin, low-form beaker with distilled water, which is freshly boiled and cooled to test temperature and place it on the pan straddle. Suspend the basket containing the empty crucible from the balance arm so that the crucible is immersed in the water. Weigh to the nearest 1 mg and record the apparent mass as W_2.

9.3 Remove the crucible from the basket and dry.

9.4 *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 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 60 min., and avoid incorporating air bubbles into the sample.

9.5 Warm the crucible in a 120°C oven for 10 min, then fill the dried crucible nearly full with the sample. Allow to cool to ambient temperature for a period of not less than 40 min, suspend in the basket and weigh to the nearest 1 mg. Record the mass of the crucible and basket plus the sample as W.

9.6 Remove the crucible from the basket and immerse it in the water bath maintained within ±1°C of the test temperature. Allow to remain in the water for at least 30 min.

9.7 At the end of 30 min, remove the crucible from the bath and insert it in the basket. Place the beaker filled with distilled water at the test temperature ±0.1°C on the pan straddle. Suspend the basket containing the crucible from the balance arm so that the crucible is immersed in the water in the beaker. Weigh to the nearest 1 mg and record the apparent mass as W_3.

10. Calculation

10.1 Calculate the relative density as follows:

$$\text{relative density} = (W - W_1)/[(W - W_1) - (W_3 - W_2)] \quad (1)$$

where:

W = mass of the crucible containing the sample suspended in the basket in the air, g,

W_1 = apparent mass of the empty crucible suspended in the basket in the air, g,

W_2 = mass of the empty crucible suspended in the basket in the water, g, and

W_3 = apparent mass of the crucible containing the sample suspended in the basket in water, g.

10.2 Calculate density as follows:

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

where:

W_T = density of water at the test temperature (Note 3).

NOTE 3—Density of water from CRC Handbook of Chemistry and Physics[5]:

Test Temperature, °C	Density of Water, kg/m^3

15.0	999.1
25	997.0

11. Report

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

12. Precision and Bias

12.1 *Single-Operator Precision*—The single-operator standard deviation of the relative density has been found to be 0.00058. Therefore, the results of two properly conducted tests by the same operator on the same asphalt should not differ by more than 1.6 kg/m^3.

12.2 *Multilaboratory Precision*—The multilaboratory standard deviation of the relative density has been found to be 0.00072. Therefore, the results of two properly conducted tests by two laboratories on the same material should not differ by more than 2.0 kg/m^3.

NOTE 4—These numbers represent the 1S and D2S limits as described in Practice C 670. The precision of tests at 15.0°C has been taken to be the same as at 15.6°C (60°F).

12.3 *Bias*—The bias of this test method has not been determined.

13. Keywords

13.1 asphalt; bituminous material; density

[5] *CRC Handbook of Chemistry and Physics*, 74th Edition, 1993–1994, pp. 6–12.