



Standard Test Method for Density of Solid Pitch (Helium Pycnometer Method)¹

This standard is issued under the fixed designation D 4892; 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.

^{ε1} NOTE—Editorial corrections were made in Section 12 in December 1999.

1. Scope

1.1 This test method covers the determination of pitch density by helium pycnometer. It is applicable at a range of room temperatures of 15 to 35°C.

1.2 *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 71 Test Method for Relative Density of Solid Pitch and Asphalt (Displacement Method)²

D 2320 Test Method for Density (Specific Gravity) of Solid Pitch (Pycnometer Method)²

D 2962 Method for Calculating Volume-Temperature Correction for Coal-Tar Pitches³

D 4296 Practice for Sampling Pitch³

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁴

3. Terminology

3.1 Definition of Term Specific to this Standard:

3.1.1 *density, as determined by this test method*— the mass per unit volume and shall be reported as follows:

$$\text{density, } 25^{\circ}\text{C, g/cm}^3 \text{ (grams per cubic centimeter)} \quad (1)$$

4. Summary of Test Method

4.1 The sample is pulverized and screened to a given fraction, then the volume of a weighed portion of the fraction is determined using a helium pycnometer.

5. Significance and Use

5.1 This test method is useful in characterizing pitches as

one element in establishing uniformity of shipments and sources of supply. With this method, the density is determined to two decimal places, which is sufficient for most applications. If a more precise measurement is required (three decimal places), use Test Methods D 2320 or D 71.

6. Apparatus

6.1 *Helium Pycnometer.*⁵

6.2 *Balance,* capable of weighing a 150-g specimen to within 1 mg.

6.3 *Sieves,* U.S. Standard 2.36-mm (No. 8), 600- μm (No. 30), conforming to Specification E 11.

6.4 *Thermometer,* for measuring room temperature.

7. Bulk Sampling

7.1 Samples from shipments shall be taken in accordance with Practice D 4296 and shall be free of foreign substances.

8. Dehydration

8.1 All bulk samples suspected of having free moisture should be air-dried or oven-dried at 50°C in a convection or forced-air oven before analyzing.

9. Preparation of Test Specimen

9.1 Crush a 50 to 200-g representative portion of the dry pitch until all of it passes the 2.36 mm (No. 8 sieve). Avoid grinding the pitch. Remove the fines by screening through the 600 μm (No. 30 sieve) and use the pitch retained on this sieve as the test specimen.

10. Calibration of Helium Pycnometer

10.1 Calibrate daily or before using, following instrument manufacturer's instructions.

11. Procedure

11.1 Place 10 to 150 g (80 to 90 % of the maximum capacity of the particular holder being used) of the prepared crushed and sized fraction into the required specimen holder provided with

⁵ Helium pycnometers available from the following suppliers have been found satisfactory for this purpose: (a) Beckman 2500 Harbor Blvd., Fullerton, CA, (b) Micromeritics, 5680 Goshen Springs Rd., Norcross, GA, and (c) Quantachrome, 5 Aerial Way, Syosset, NY.

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05.OF on Industrial Pitches.

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² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 05.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

the instrument. Weigh to the nearest milligram.

11.2 Determine volume of pitch following manufacturer's instructions.

11.3 Weigh specimen container after determination; if this weight is lower than the initial weight, the specimen has lost moisture or volatiles during the density determination. Use the lower weight in determining the density. Also, if the specimen appears to have free moisture, dry the specimen as specified in 8.1, and repeat the density determination.

11.4 Record the room temperature during the density determination.

12. Calculation

12.1 The procedure described in this test method determines the volume of a given mass of pitch at the ambient temperature (room temperature) of the helium pycnometer. Since the volume of a given mass of pitch is temperature-dependent, the density of the given specimen will vary with the ambient temperature. The variation of pitch volume with temperature has been previously established (Method D 2962 and the literature⁵), and the following calculation corrects all density measurements to 25°C.

12.2 The density of pitch, corrected to 25°C, is given by the general formula:

$$d_{25^{\circ}\text{C}} = \frac{W}{V_t [1 \pm 0.00047 \Delta t]} \quad (2)$$

where:

W = specimen mass, g,

V_t = volume of specimen, cm³, at measurement temperature,

Δt = difference between 25°C and the measurement temperature, and

$d_{25^{\circ}\text{C}}$ = pitch density at 25°C, g/cm³.

The factor 0.00047/°C is obtained from the literature⁶ and Method D 2962.

12.3 Calculate the density of the pitch (to three decimal places) using the following equation:

$$d_{25^{\circ}\text{C}} = V_t \frac{W}{[\text{correction factor}]} \quad (3)$$

The correction factors for measurement temperatures of 15 to 35°C are given in Table 1.

12.4 *Example*—A specimen of pitch having a mass of 20.027 g was found to have a volume of 15.26 cm³ at a temperature of 30°C; the density calculation is as follows:

$$d_{25^{\circ}\text{C}} = \frac{20.027 \text{ g}}{15.26 \text{ cm}^3 [0.998]} = 1.315 \text{ g/cm}^3 \quad (4)$$

⁶ Mapstone, G. E., "Specific Gravity of Coal Tar Fractions," *Journal of Applied Chemistry*, October 5, 1955, pp. 582–588.

TABLE 1 Volume Correction Factors Versus Measurement Temperature

| Measurement Temperature, °C | Correction Factor |
|-----------------------------|-------------------|
| 15 | 1.005 |
| 16 | 1.004 |
| 17 | 1.004 |
| 18 | 1.003 |
| 19 | 1.003 |
| 20 | 1.002 |
| 21 | 1.002 |
| 22 | 1.001 |
| 23 | 1.001 |
| 24 | 1.000 |
| 25 | 1.000 |
| 26 | 1.000 |
| 27 | 0.999 |
| 28 | 0.999 |
| 29 | 0.998 |
| 30 | 0.998 |
| 31 | 0.997 |
| 32 | 0.997 |
| 33 | 0.996 |
| 34 | 0.996 |
| 35 | 0.995 |

12.5 Average duplicate results and round off to two decimal places.

13. Report

13.1 Report density of the pitch to two decimal places as follows: density, 25°C, g/cm³.

14. Precision and Bias ⁷

14.1 *Precision*—The following criteria shall be used for the acceptability of results (95 % probability):

14.1.1 *Repeatability*—Duplicate values by the same operator shall not be considered suspect unless they differ by more than 0.02.

14.1.2 *Reproducibility*—The values reported by each of two laboratories, representing the arithmetic average of duplicate determinations, shall not be considered suspect unless the reported values differ by more than 0.03.

14.2 *Bias*—During the interlaboratory study to determine the precision of this test method, the density was also determined by Test Method D 2320 (water pycnometer). It was found that the density determined by helium pycnometer was higher than that determined by water pycnometer by an average value of 0.004.

15. Keywords

15.1 density; helium pycnometer; pitch

⁷ Supporting data is available from ASTM Headquarters. Request RR: D08-1003.

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