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An American National Standard

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

This standard is issued under the fixed designation D4892; 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 pitch density by helium pycnometer. It is applicable at a range of room temperatures of 15 to 35°C.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:*[2]

D71 Test Method for Relative Density of Solid Pitch and Asphalt (Displacement Method)

D2320 Test Method for Density (Relative Density) of Solid Pitch (Pycnometer Method)

D2962 Test Method for Calculating Volume-Temperature Correction For Coal-Tar Pitches

D4296 Practice for Sampling Pitch

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

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:

density, 25°C, g/cm^3 (grams per cubic centimeter) (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 D2320 or D71.

6. Apparatus

6.1 *Helium Pycnometer.* [3]

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 E11.

6.4 *Thermometer*, for measuring room temperature.

7. Bulk Sampling

7.1 Samples from shipments shall be taken in accordance with Practice D4296 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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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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.

[3] 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. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

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 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 (Test Method D2962 and the literature[4]), 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:

$$d_{25°C} = \frac{W}{V_t\,[1 \pm 0.00047\,\Delta t]} \tag{2}$$

where:

W = specimen mass, g,
V_t = volume of specimen, cm^3, at measurement temperature,
Δt = difference between 25°C and the measurement temperature, and
$d_{25°C}$ = pitch density at 25°C, g/cm^3.

The factor 0.00047/°C is obtained from the literature[4] and Test Method D2962.

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

$$d_{25°C} = V_t \frac{W}{[\text{correction factor}]} \tag{3}$$

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

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

12.4 *Example*—A specimen of pitch having a mass of 20.027 g was found to have a volume of 15.26 cm^3 at a temperature of 30°C; the density calculation is as follows:

$$d_{25°C} = \frac{20.027\ \text{g}}{15.26\ \text{cm}^3\ [0.998]} = 1.315\ \text{g/cm}^3 \tag{4}$$

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/cm^3.

14. Precision and Bias [5]

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 D2320 (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

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

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D08-1003.