



Designation: D2320 – 98 (Reapproved 2022)<sup>ε1</sup>

# Standard Test Method for Density (Relative Density) of Solid Pitch (Pycnometer Method)<sup>1</sup>

This standard is issued under the fixed designation D2320; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Editorially updated Section 15 in August 2022.

## 1. Scope

1.1 This test method covers the determination of density and relative density by pycnometer, and can be used for pitch that can be handled in fragments.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D4296 Practice for Sampling Pitch

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

## 3. Terminology

3.1 *Definitions:*

3.1.1 *density, n*—the mass per unit of volume at a standard test temperature.

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

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<sup>2</sup> 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.

3.1.2 *relative density, n*—the ratio of the mass of a given volume of the material at a standard test temperature to that of an equal volume of water at the same temperature.

## 4. Summary of Test Method

4.1 The sample is weighed and its volume determined by the mass of water displaced.

## 5. Significance and Use

5.1 Values of density and relative density are used for converting volumes to units of mass as required in other ASTM standards and in sales transactions.

## 6. Apparatus

6.1 *Glass Pycnometer*, capacity about 25 mL, with accurately fitting glass stopper with a capillary tube 1.0 mm to 2.0 mm in diameter.

6.2 *Water Bath*, maintained at 25.0 °C ± 0.1 °C and provided with mechanical stirring.

6.3 *Vacuum Pump or Aspirator*, capable of producing a vacuum of 2.7 kPa (20 torr).

NOTE 1—20 torr = 20 mm Hg = 2.66 kPa.

6.4 *Manometer*, suitable for measuring the specified vacuum.

6.5 *Vacuum Desiccator with Guard*.

6.6 *Sieves*, U.S. Standard 2.36 mm (No. 8) and 600 μm (No. 30) conforming to Specification E11.

## 7. Reagents and Materials

7.1 *Wetting Agent*, 0.1 g/mL Aerosol OT.

7.2 *Wetting Agent*, 0.1 g/mL BRU 35 (Alyoxyethylene dodecyl ether, detergent).

## 8. Bulk Sampling

8.1 Samples from shipments shall be taken in accordance with Practice D4296 and shall be free of foreign substances. Thoroughly mix the sample immediately before removing a representative portion for the determination or for dehydration.

## 9. Dehydration

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

## 10. Preparation of Test Sample

10.1 Crush a 50 g to 100 g representative portion of the dry pitch until all of it passes through 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 sample. If desired, small lumps of pitch may be used without crushing, provided the lumps pass the 2.36 mm sieve and are retained on the 600 μm sieve. In either case, follow the evacuation steps described in 12.2. In case of dispute use the crushing method.

## 11. Calibration of Pycnometer

11.1 Weigh the thoroughly clean, dry, and assembled pycnometer to the nearest 0.5 mg and record the mass.

11.2 Fill the pycnometer with freshly boiled distilled water that has been cooled to about 20 °C. Insert the stopper with a rotary motion to secure a firm seat, making sure that no air is entrapped, then completely immerse the pycnometer in the water bath at 25 °C for a minimum of 30 min. Remove and wipe the filled pycnometer as described in 12.4; then immediately weigh to the nearest 0.5 mg. Remove the stopper and repeat the operations until three successive weighings agree within 0.5 mg. Record the average mass of the pycnometer with water.

## 12. Procedure

12.1 Place not less than 5 g of the prepared crushed or lump sample in the dry, tared pycnometer and weigh with the stopper to the nearest 0.5 mg. Record the mass. Cover the pitch to a depth of about 10 mm with freshly boiled distilled water cooled to a temperature of about 20 °C. Add several drops of a stock solution of Aerosol OT containing 0.1 g/mL of solution or other wetting agent.

12.2 Place the unstoppered pycnometer in the vacuum desiccator and evacuate the air at such a rate that no sudden or violent ebullition occurs. Maintain a pressure in the desiccator of 2.7 kPa to 3.3 kPa (20 torr to 25 torr), as measured by the manometer, for at least 15 min to remove all entrapped air bubbles. If air bubbles persist, add more wetting agent and continue the process until no air bubbles are visible.

12.3 Remove the pycnometer from the desiccator and fill with freshly boiled distilled water that has been cooled to about 20 °C. Insert the stopper with a rotary motion to secure a firm seat, making sure that no air is entrapped. Then completely immerse the pycnometer in the water bath at 25 °C for a minimum of 30 min.

12.4 After 30 min, raise the pycnometer until the top of the stopper is above the water level, then quickly and carefully wipe the water off the flat surface of the stopper, taking care not to remove water from the capillary tube. Remove the pycnom-

eter from the bath and dry with a clean, dry cloth or with soft, absorbent paper. Weigh immediately to the nearest 0.5 mg and record the mass.

## 13. Calculation

13.1 Calculate the relative density of the pitch as follows:

$$\text{sp gr} = (W_3 - W_1) / [(W_2 - W_1) - (W_4 - W_3)] \quad (1)$$

$$= W / [W - (W_4 - W_2)]$$

where:

$W$  = mass of sample ( $W_3 - W_1$ ),  
 $W_1$  = mass of pycnometer,  
 $W_2$  = mass of pycnometer filled with water at 25 °C,  
 $W_3$  = mass of pycnometer with sample, and  
 $W_4$  = mass of pycnometer with sample filled with water at 25 °C.

13.2 Calculate the density of the pitch as follows:

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

where:

relative density = calculation from 13.1, and  
 $W_T$  = density of water at test temperature in desired units.

Test Temperature	Density of Water, <sup>3</sup>	
	g/cm <sup>3</sup>	kg/m <sup>3</sup>
25 °C	0.9970	997.0

## 14. Report

14.1 Report the relative density of the pitch to the third decimal place, expressed as specific gravity, 25 °C/25 °C.

14.2 Report the density to the nearest third decimal place in g/cm<sup>3</sup> and with four significant figures in kg/m<sup>3</sup>, expressed as density at 25 °C.

## 15. Precision and Bias

15.1 The following criteria shall be used for judging the acceptability of results (95 % probability):

15.1.1 *Repeatability*—The difference between two independent results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test materials within short intervals of time would exceed the following value about 5 % of the time (one case in 20 in the long run) in the normal and correct operation of the test method:

$$\text{Relative density: } 0.011$$

$$\text{Density: } 0.007 \text{ g/cm}^3 \text{ or } 7.0 \text{ kg/m}^3$$

15.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would exceed the following value about 5 % of the time (one case in 20 in the long run) in the normal and correct operation of the test method:

<sup>3</sup> Density of water from *CRC Handbook of Chemistry and Physics*, 76th ed., CRC Press, 1995.