Designation: C1039 – 85 (Reapproved 2020)ε1

Standard Test Methods for Apparent Porosity, Apparent Specific Gravity, and Bulk Density of Graphite Electrodes[1]

This standard is issued under the fixed designation C1039; 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.

ε1 NOTE—Editorially updated 3.1, 4.3.1, Note 1, and 9.1 in December 2020.

1. Scope

1.1 These test methods cover the determination of apparent porosity, apparent specific gravity, and bulk density of cores taken from graphite electrodes manufactured for use in electric arc furnaces. (See also C559 and C838.)

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 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:*[2]

C559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles

C783 Practice for Core Sampling of Graphite Electrodes

C838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes

3. Significance and Use

3.1 The results of these test methods can be used as a quality control or quality assurance check of electrodes either during their manufacture or at the user's location. The results of these methods tend to be operator-sensitive; therefore, care must be taken in the execution of the test in order to obtain reproducible results.

4. Apparatus

4.1 *Drying Oven.*

4.2 *Analytical Balance,* capable of weighing to 0.1 g.

4.3 *Autoclave or Pressure Vessel,* capable of withstanding one atmosphere externally and designed to withstand at least 448 kPa to 483 kPa internal pressure.

4.3.1 The pressure vessel shall be provided with an open-top container to hold the specimens and a means of introducing water around the specimens while specimens are being held at low pressure.

4.4 *Vacuum Pump.*

4.5 *Wire Loop, Halter or Stirrup,* fabricated with 22 AWG (9.643 mm) copper wire shall be provided for determining suspended weight.

4.6 *Smooth Linen or Cotton Cloth.*

5. Test Specimens and Sampling

5.1 Electrodes can be sampled using Practice C783 that result in a specimen with approximately 50 mm diameter and 191 mm long or a specimen of equivalent volume.

5.2 If sizes and shapes which are different from those described in 5.1, these shall be included in the report.

5.3 For each test, select at least five electrodes, at random, to represent a lot. The lot size will be determined by agreement of the parties desiring the tests.

6. Calibration

6.1 Prior to obtaining the suspended weights of the specimens, the balance shall be adjusted to zero with the wire stirrup suspended from the balance and immersed into a container of the liquid to the same depth in the liquid as occurs when a specimen is in place.

[1] These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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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.

7. Procedure

7.1 *Determinations of Dry Weight, D:*

7.1.1 Dry the test specimens to constant weight by heating to 100 °C to 110 °C. Cool and determine the dry weight, *D*, in grams to the nearest 0.1 g. If the time between drying and weighing exceeds 8 h, the specimens must be stored in a desiccator.

7.1.2 The determination of dry weight may be done either before or after the saturation operation. If the specimen is friable or there is evidence that particles have broken loose during the saturation operation, the dry weight shall be obtained after the suspended and saturated weights have been determined. Drying as described in 7.1.1 must be carried out.

7.2 *Saturation:*

7.2.1 Using the pressure vessel described in 4.3, place the specimens in the open-top container inside the vessel. Close and seal the vessel and pump down at least to 133 Pa (1 mm Hg) and maintain this pressure for 30 min.

7.2.2 Slowly introduce water (preferably distilled water) until the specimens are covered with at least 40 mm of water. Continue to pump for an additional 5 min.

NOTE 1—Sufficient water must be provided above the specimens to supply enough water to fill the open pores of the specimens during pressurization. If after pressurization, one or more specimens are exposed to air above the water, these specimens must be dried and the process repeated (7.1.1).

7.2.3 After the evacuation process is completed, close the vacuum line and introduce air pressure. Pressurize to 207 kPa ± 7 kPa gauge pressure and hold for 4 h. Following this period of time return the vessel to atmospheric pressure.

7.3 *Determination of Suspended Weight, S:*

7.3.1 Within 1 h of the time the vessel returns to atmospheric pressure, the suspended weights and saturated weights must be obtained.

7.3.2 Place the container used in the vacuum-pressure vessel near the immersion container to facilitate rapid transfer to the weighing stirrup (see 4.5).

7.3.3 Obtain the suspended weight, *S*, to the nearest 0.1 g, of each specimen. The specimens can be transferred through air but the transfer must be made quickly. Following the weighing, each specimen should be returned to a separate container of water or the saturated weight, *W*, should be obtained immediately according to 7.4.

7.4 *Determination of Saturated Weight, W:*

7.4.1 After determining the suspended weight, blot each specimen lightly with a moistened smooth linen or cotton cloth to remove all drops of water from the surface and weigh in air, to the nearest 0.1 g, to obtain the saturated weight, *W*. The cloth must be prepared by previously saturating it with water and then pressing only enough to remove the water which would drip from the cloth. Avoid excessive blotting or rubbing which will remove water from the pores of the specimen.

8. Calculation

8.1 *Exterior Volume*—Obtain the exterior volume by subtracting the suspended weight from the saturated weight.

$$V,\ cm^3 = W - S \qquad (1)$$

where:

V = exterior volume, cm^3,
W = saturated weight, g, and
S = suspended weight, g.

NOTE 2—This assumes that one cubic centimetre of water weighs 1 g. This is true within about 3 parts in 1000 for water at 20 °C to 25 °C.

8.2 *Volume of Open Pores*—Obtain the volume of open pores by subtracting the dry weight, *D*, from the saturated weight, *W*.

$$\text{Volume of open pores, } cm^3 = W - D \qquad (2)$$

8.3 *Volume of Impervious Portion*—Obtain the volume of impervious portion by subtracting the suspended weight, *S*, from the dry weight, *D*.

$$\text{Volume of impervious portion, } cm^3 = D - S \qquad (3)$$

8.4 *Apparent Porosity, P*—The apparent porosity, *P*, is expressed as a percentage of the volume of open pores to the exterior volume, and may be calculated as follows:

$$P,\ \% = [(W - D)/V] \times 100 = [(W - D)/(W - S)] \times 100 \qquad (4)$$

8.5 *Bulk Density, B*—The bulk density of a specimen, *B*, in grams per cubic centimetre, is its mass per unit volume, including pores. Calculate as follows:

$$B,\ g/cm^3 = D/V = D/(W - S) \qquad (5)$$

8.6 *Apparent Specific Gravity, T*—Calculate the apparent specific gravity, *T*, of that portion of the specimen which is impervious to water under these test conditions as follows:

$$T = D/(D - S) \qquad (6)$$

9. Report

9.1 Report the sizes of the specimens tested.

9.2 Report the values of all properties for individual specimens and the averages.

9.3 Calculate apparent porosity values to one decimal place.

9.4 Calculate bulk density and apparent specific gravity values to two decimal places.

10. Precision and Bias

10.1 *Interlaboratory Tests*—An interlaboratory study using the vacuum-pressure method, was conducted among six laboratories who tested three specimens of each of three graphite materials representing a range of apparent porosities and bulk densities. The three materials were chosen from premium grade electrodes, regular grade electrodes, and connecting nipples.

10.2 *Precision*—Tables 1-3 show the applicable data for precision in terms of percent apparent porosity (Table 1), grams per cubic centimetre of bulk density (Table 2) and apparent specific gravity (Table 3).

TABLE 1 Precision of Apparent Porosity, Percent

Material	Average	Standard Deviations	
		Within Labs	Between Labs
1	23.1	0.70	0.71
2	18.6	1.21	0.91
3	13.1	1.37	0.33