Standard Test Methods for Apparent Porosity, Liquid Absorption, Apparent Specific Gravity, and Bulk Density of Refractory Shapes by Vacuum Pressure¹

This standard is issued under the fixed designation C 830; 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 These test methods cover the determination of the following properties of refractory shapes:
 - 1.1.1 Apparent porosity,
 - 1.1.2 Liquid absorption,
 - 1.1.3 Apparent specific gravity, and
 - 1.1.4 Bulk density.
- 1.2 These test methods are applicable to all refractory shapes except those that chemically react with both water and mineral spirits. When testing a material capable of hydration or other chemical reaction with water but which does not chemically react with mineral spirits, mineral spirits is substituted for water and appropriate corrections for the density differences are applied when making calculations.
- 1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are provided for information only.
- 1.4 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.

Note 1—Test Methods C 20 cover procedures for testing properties of refractories that are not attacked by water.

2. Referenced Documents

- 2.1 ASTM Standards:
- C 20 Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water²
- C 134 Test Methods for Size, Dimensional Measurements, and Bulk Density of Refractory Brick and Insulating Firebrick²
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

3. Significance and Use

- 3.1 Apparent porosity, water absorption, apparent specific gravity, and bulk density are primary properties of refractory shapes. These properties are widely used in the evaluation and comparison of product quality and as part of the criteria for selection and use of refractory products in a variety of industrial applications. These test methods are used for determining any or all of these properties and are particularly useful for testing hydratable products.
- 3.2 These test methods are primary standard methods that are suitable for use in quality control, research and development, establishing criteria for and evaluating compliance with specifications, and providing data for design purposes.
- 3.3 Fundamental assumptions inherent in these test methods are:
- 3.3.1 The test specimens conform to the requirements for size, configuration, and original faces,
- 3.3.2 The open pores of the test specimens are fully impregnated with liquid during the vacuum-pressure treatment, and
- 3.3.3 The blotting of the saturated test specimens is performed as specified in a consistent and uniform manner to avoid withdrawing liquid from the pores.
- 3.3.4 Deviation from any of these assumptions adversely affects the test results.
- 3.4 In laboratory studies involving castable specimen, a bias was noted between formed 2 X 2 X 2-in (50 X 50 X 50-mm) and specimens quartered from larger 9 X 4.5 X 2.5-in (228 X 114 X 64-mm) cast specimens. Additionally, an error in the apparent porosity determination was found on castables whenever the specimens were heated to 1500°F (816°C) and then exposed to water as a saturation media. The error was attributed to reactivity of cement with water and subsequent re-hydration of cement phases. The higher the cement level of the castable, the greater the error noted. It was concluded that an error in porosity values could occur for refractory materials having a potential to form hydrated species with water. Testing under the same conditions in kerosene produced results that were believed to be more accurate, but the data suggested that the kerosene might not have saturated the open pores of cast specimen as readily as water. Supporting data were filed at ASTM headquarters and can be obtained by requesting research report 1014.

¹ These test methods are under the jurisdiction of ASTM Committee C-8 on Refractories and are the direct responsibility of Subcommittee C08.03 on Physical Total

Current edition approved April 10, 2000. Published June 2000. Originally published as C 830 – 76. Last previous edition C 830 – 93 (1998).

² Annual Book of ASTM Standards, Vol 15.01.

³ Annual Book of ASTM Standards, Vol 14.02.



- 3.5 Certain precautions must be exercised in interpreting and using results from these test methods. All four property values are interrelated by at least two of the three base data values generated during testing. Thus, an error in any base data value will cause an error in at least three of the property values for a given test specimen. Certain of the properties, that is, apparent specific gravity and bulk density, are functions of other factors such as product composition, compositional variability within the same product, impervious porosity, and total porosity. Generalizations on or comparisons of property values should be judiciously made between like products tested by these test methods or with full recognition of potentially inherent differences between the products being compared or the test method used.
- 3.6 When a liquid other than water is used, such as types of kerosene or mineral spirits, specific gravity must be known by either determination or monitoring on a controlled basis. Specific gravity will change due to different grades of liquids, evaporation, or contamination with dirt or foreign material. The test should not be run if the liquid becomes dirty, foamy, or changes color, because foreign particles can block pores and prevent impregnation of the sample.

4. Test Specimens

- 4.1 When testing 9-in. (228-mm) straight brick, use a quarter-brick specimen obtained by halving the brick along a plane parallel to the 9 by 2½ or 3-in. (228 by 64 or 76-mm) face and along a plane parallel to the 4½ by 2½ or 3-in. (114 by 64 or 76-mm) face. Four of the surfaces of the resultant quarter-brick specimen include part of the original molded faces.
- 4.2 When testing other refractory shapes, cut drill, or break from each shape a specimen having a volume of approximately 25 to 30 in.³ (410 to 490 cm³). The specimen shall include interior and exterior portions of the shape.
- 4.3 Remove all loosely adhering particles from each specimen.

5. Procedures

- 5.1 Determination of Dry Weight, D:
- 5.1.1 Dry the test specimens to constant weight by heating to 220 to 230°F (105 to 110°C) and determine the dry weight, D, in grams to the nearest 0.1 g.
- 5.1.2 The drying procedure may be omitted only when the test specimens are known to be dry, as may be the case with samples taken directly from kilns.
- 5.1.3 The drying of the specimens to constant weight and the determination of their dry weight may be done either before or after the saturation operation (5.2). Usually, the dry weight is determined before saturation; if, however, the specimens are friable or evidence indicates that particles have broken loose during the saturating operation, dry and weigh the specimens after the suspended weight, *S*, and the saturated weight, *W*, have been determined as described in 5.3 and 5.4. Use this second dry weight in all appropriate calculations.
- 5.2 Saturation—Place the test specimens in a suitable vacuum-pressure vessel (Note 2) which shall be closed, secured, and pumped down to an absolute pressure of not more than 1.9 in. Hg (6.4 kPa). Hold this pressure for 30 min. Allow

the water or mineral spirits (see 1.2) to enter the vessel while maintaining the vacuum for 5 min. Then close the vacuum line and pressurize the vessel by means of compressed air or a pressure pump. Maintain this pressure at 30 psi (207 kPa) or more for 60 min. Then release the pressure; the saturated specimens are now ready for weighing.

Note 2—The vacuum-pressure vessel should be capable of withstanding an absolute pressure of 1.0 in. Hg (3.4 kPa) or a pressure of 65 to 70 psi (448 to 483 kPa) without deforming or rupturing. It should be provided with gages or manometers for indicating vacuum or pressure and a relief valve, as well as vacuum, pressure, and liquid lines. The liquid may be introduced at the bottom, in which case a dual-acting valve will suffice for both filling and draining the vessel.

- 5.3 Determination of Suspended Weight, S:
- 5.3.1 Determine the weight, S, of each test specimen in grams to the nearest 0.1 g after saturation and while suspended in liquid.
- 5.3.2 This weighing is usually accomplished by suspending the specimen in a loop or halter of AWG Gage-22 (0.643-mm) copper wire hung from one arm of the balance. The balance shall be previously counter-balanced with the wire in place and immersed in liquid to the same depth as is used when the refractory specimens are in place.
- 5.4 Determination of Saturated Weight, W—After determining the suspended weight, blot each specimen lightly with a moistened smooth linen or cotton cloth to remove all drops of liquid from the surface, and determine the saturated weight, W, in grams to the nearest 0.1 g by weighing in air. Perform the blotting operation by rolling the specimen lightly on the wet cloth, which has previously been saturated with liquid, and then press only enough to remove such liquid as will drip from the cloth. Excessive blotting will induce error by withdrawing liquid from the pores of the specimen.
- 5.5 Determination of Exterior Volume, V—Obtain the volume, V, of the test specimens in cubic centimetres by subtracting the suspended weight from the saturated weight, both in grams, as follows:

$$V, \text{cm}^3 = W - S$$

(1)

Note 3—This assumes that 1 cm³ of water weighs 1 g. This is true within about 3 parts in 1000 for water at room temperature.

Note 4—When substituting mineral spirits for water, make the following correction:

$$V$$
, cm³ = $(W - S)$ /density of liquid

(2)

5.6 Determination of Volume of Open Pores and Impervious Portions—Calculate the volume of both the open pores and the impervious portions of the specimen as follows:

Volume of open pores,
$$cm^3 = W - D$$
 (3)

Volume of impervious portion,
$$cm^3 = D - S$$
 (4)

Note 5—When substituting mineral spirits for water, make the following corrections:

Volume of open pores,
$$cm^3 = (W - D)/density of liquid$$
 (5)

Volume of impervious portion, cm³ = (D - S)/density of liquid

(6)



6. Calculation

- 6.1 As noted in 1.2, when mineral spirits is substituted for water, appropriate corrections for the density difference between the liquids are required in the calculations. Thus, the equations used when the liquid is mineral spirits differ from those used when the liquid is water. To avoid confusion and intermingling of equations, the calculations used for each liquid are presented in separate sections (see Sections 7 and 8).
- 6.2 When the liquid is water, calculation of the various volumes and properties is straightforward, as in Test Methods C 20. The assumption is taken that 1 cm³ of water weighs 1 g. This is true within about 3 parts per 1000 at room temperature. Therefore, no corrections for change in water density with change in temperature are applied in any calculations. However, use of the 1 g/cm³ factor is implicit in all calculations where direct weight measurements are converted to volumes. This affects expression of the results in the proper unit of measure, that is, cm³.
- 6.3 When the liquid is mineral spirits, correction for the density difference between mineral spirits and water is required in all calculations where direct weight measurements are converted to volumes. The correction factor is:

density of liquid (g/cm³)

Use of this factor in calculations also affects expression of the results in the proper unit of measure, that is, g/cm³.

7. Calculation When Liquid is Water

- 7.1 *Volume Calculations*:
- 7.1.1 Determination of Exterior Volume, V—The exterior volume of the test specimen is its bulk volume, including all solid material, open pores, and impervious portions. Calculate *V* in cubic centimetres by subtracting the suspended weight from the saturated weight, both in grams, as follows:

$$V. \text{ cm}^3 = W - S$$

(7)

7.1.2 Determination of Volume of Open Pores and Volume of Impervious Portions—Calculate the volume of open pores and the volume of impervious portions in the test specimen in cubic centimetres as follows:

Volume of open pores,
$$cm^3 = W - D$$
 (8)

Volume of impervious portions,
$$cm^3 = D - S$$
 (9)

- 7.2 Property Calculations:
- 7.2.1 Apparent Porosity, P—The apparent porosity expresses as a percentage the relationship of the volume of open pores in the test specimen to its exterior volume. Calculate P as follows:

$$P, \% = [(W - D)/V] \times 100$$
 (10)

7.2.2 Water Absorption, $A_{\rm w}$ — The water absorption expresses as a percentage the relationship of the weight of water absorbed to the weight of the dry test specimen. Calculate $A_{\rm w}$ as follows:

$$A_{w}, \% = [(W - D)/D] \times 100$$
 (11)

7.2.3 Apparent Specific Gravity, T—The apparent specific

gravity of the test specimen is the quotient of its dry weight divided by its volume of impervious portions. Since the result expresses a type of specific gravity value, it is given that the value obtained for T has already been divided by the assumed density of water, thereby rendering the result unitless. Calculate T as follows:

$$T = D/D - S$$

(12)

7.2.4 *Bulk Density, B*—The bulk density of the test specimen in grams per cubic centimetre is the quotient of its dry weight divided by its exterior volume. Calculate *B* as follows:

$$B$$
, $g/cm^3 = D/V$

(13)

Note 6—This test method for determining bulk density is useful for checking bulk density values obtained by the direct measurement method described in Test Methods C 134. While this test method is more accurate than the direct measurement method, the latter is better suited for plant and field testing since it is a less involved technique. The present test method is preferable for specimens that are deeply branded or are irregular in contour.

8. Calculation When Liquid Is Mineral Spirits

- 8.1 The calculations when the liquid is mineral spirits are presented below without commentary or explanation except where these are essential to understand a particular calculation. However, the commentary, explanations, and Note 6 given in Section 7 for calculations when the liquid is water are fully applicable here also.
- 8.1.1 When the term *liquid* appears in any of the calculations below, it means mineral spirits.
- 8.1.2 When the density of water is used in any of the calculations below, it is still assumed to be 1 g/cm³.
 - 8.2 Volume Calculations:
- 8.2.1 *Determination of Exterior Volume, V*—Calculate *V* as follows:

$$V$$
, cm³ = $(W - S)$ / density of liquid

(14)

8.2.2 Determination of Volume of Open Pores and Volume of Impervious Portions—Calculate as follows:

Volume of open pores,
$$cm^3 = (W - D)/density of liquid$$
 (15)

Volume of impervious portions, cm³ = (D - S)/density of liquid (16)

- 8.3 Property Calculations:
- 8.3.1 *Apparent Porosity, P*—Calculate *P* as follows:

$$P, \% = [(W - D)/(W - S)] \times 100$$
 (17)

Note 7—Since this calculation involves division of the volume of open pores, (W-D)/density of liquid, by the exterior volume, (W-S)/density of liquid, the correction factor (density of liquid) cancels out and therefore does not appear in the equation.

8.3.2 *Liquid Absorption*, A_1 — Because of the density difference between mineral spirits and water, the value calculated for liquid absorption of the test specimen will not be equal to the value for water absorption (7.2.2) of the same specimen.



Calculate A_i as follows:

$$A_l = [(W - D)/D] \times 100$$
 (18)

Note 8—If conversion of liquid absorption to water absorption is desired, multiply the value for liquid absorption by the following factor:

density of water/density of liquid

8.3.3 *Apparent Specific Gravity, T*—Calculate *T* as follows:

$$T = \frac{D}{D - S} \times \frac{\text{density of liquid}}{\text{density of water}}$$

(19)

8.3.4 Bulk Density, B—Calculate B as follows:

$$B, g/\text{cm}^3 = D/V$$

(20)

9. Report

9.1 For each property report the individual values obtained.

10. Precision and Bias

10.1 Interlaboratory Test Data—An interlaboratory round-robin test was conducted in 1982 between six laboratories on three different types of refractories. The same four specimens of each material were sent from laboratory to laboratory, thereby eliminating sample variation. Each laboratory conducted two separate tests using two different operators, Operators A and B. The components of variance expressed as standard deviation and relative standard deviation (coefficient of variation) for absorption, apparent porosity, bulk density, and apparent specific gravity (ASG) were as given in Table 1.

Note 9—All statistical calculations are in accordance with Practice E 691.

- 10.2 *Precision*—For the components of variance listed in Table 1, a test result on any given sample should be considered significantly different at a confidence level of 95 % if the repeatability or reproducibility, or both, exceed the precision data listed in Table 2.
- 10.3 *Bias*—No justifiable statement on bias is possible since the true physical property values of refractories cannot be established by an accepted reference material.

11. Keywords

11.1 apparent porosity; apparent specific gravity; bulk density; hydration; refractory shapes; vacuum pressure; water absorption

TABLE 1 Interlaboratory Test Data

| | | | Material | | |
|---|----------------------------------|---------|----------|---------|-----------------|
| | | A | В | С | — Grand Average |
| Absorption, avg, % | Xi | 12.46 | 6.77 | 4.86 | |
| Standard deviation within | S _r | 0.0732 | 0.0899 | 0.0815 | 0.0815 |
| Standard deviation between | S_L | 0.1201 | 0.0231 | 0.0073 | 0.050 |
| Relative standard deviation, % | V_{r}^{-} | 0.59 | 1.33 | 1.68 | 1.20 |
| | VL | 0.96 | 0.34 | 0.15 | 0.48 |
| Apparent porosity, avg, % | X_{j} | 22.39 | 14.69 | 11.31 | |
| | S _r | 0.1038 | 0.1762 | 0.1941 | 0.158 |
| | SL | 0.1805 | 0.031 | 0.0108 | 0.0741 |
| | V_{r}^{-} | 0.46 | 1.20 | 1.72 | 1.13 |
| | V _L | 0.81 | 0.21 | 0.095 | 0.37 |
| Bulk density, avg | Xi | 1.798 | 2.172 | 2.327 | |
| , · · · · · · · · · · · · · · · · · · · | X _j S _r | 0.0021 | 0.0047 | 0.0049 | 0.0039 |
| | SL | 0.0036 | 0.0025 | 0.0011 | 0.0024 |
| | V_r | 0.12 | 0.22 | 0.21 | 0.18 |
| | V _L | 0.20 | 0.12 | 0.05 | 0.12 |
| Apparent specific gravity, avg | X_{j} | 2.317 | 2.546 | 2.624 | |
| | S _r | 0.00236 | 0.00818 | 0.00670 | 0.00575 |
| | SĹ | 0.00308 | 0.0000 | 0.00147 | 0.00152 |
| | V_r | 0.10 | 0.32 | 0.26 | 0.23 |
| | VL | 0.13 | 0.00 | 0.056 | 0.062 |

TABLE 2 Precision and Relative Precision

| | Precision | | Relative Precision | |
|---------------------------|------------------------------|-----------------------|--------------------|------------------|
| Test Property | Repeatability I _r | Reproducibility I_R | % I _r | % I _R |
| Absorption, % | 0.23 | 0.27 | 3.40 | 3.66 |
| Apparent porosity, % | 0.45 | 0.49 | 3.20 | 3.36 |
| Bulk density | 0.011 | 0.013 | 0.51 | 0.61 |
| Apparent specific gravity | 0.016 | 0.017 | 0.65 | 0.67 |

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