Designation: C 604 – 02

Standard Test Method for True Specific Gravity of Refractory Materials by Gas-Comparison Pycnometer[1]

This standard is issued under the fixed designation C 604; 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 This test method covers the determination of the true specific gravity of solid materials, and is particularly useful for materials that easily hydrate which are not suitable for test with Test Method C 135. This test method may be used as an alternate for Test Method C 135, Test Method C 128, and Test Method C 188 for determining true specific gravity.

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:*

C 128 Test Method for Specific Gravity and Absorption of Fine Aggregate[2]

C 135 Test Method for True Specific Gravity of Refractory Materials by Water Immersion[3]

C 188 Test Method for Density of Hydraulic Cement[4]

3. Summary of Test Method

3.1 The sample is powdered to ensure permeation of gas into all pores. For practical purposes this is assumed to be true when the sample passes a No. 325 (45-µm) sieve. The volume of a carefully weighed powdered sample which has first been heated to drive off moisture and undesired combined water is measured by the gas-comparison pycnometer. Density is calculated from the sample weight in grams divided by its volume in cubic centimetres. This is also the specific gravity of the sample at room temperature compared to water at 4°C.

3.2 The principle of the gas-comparison pycnometer is as follows: There are two chambers and two pistons as sketched in Fig. 1. For purposes of illustration, the chambers are assumed to be equal in volume, and there is no sample in either cylinder. Under these conditions, with the coupling valve closed, any change in the position of one piston must be duplicated by an identical stroke in the other in order to maintain the same pressure on each side of the differential pressure indicator.

3.3 If a sample, V_x, is inserted into chamber *B*, the coupling valve closed and both pistons advanced the same amount from position *1* to position *2*, the pressures will not remain the same. However the pressures can be maintained equal if piston *B* instead is moved only to position *3*. Then the remaining displacement d_x, from position *3* to position *2*, is equal to the volume of the sample, V_x. If piston *A* always is advanced exactly the same distance each time a measurement is made, the distance that piston *B* differs from position *2*, when the pressures in both cylinders are equal, will always be proportional to the volume, V_x. The distance (d_x) between positions *2* and *3* can be calibrated and made to read directly in terms of cubic centimetres, employing a digital counter.

4. Significance and Use

4.1 The true specific gravity of a material is the ratio of its true density, determined at a specific temperature, to the true density of water, determined at a specific temperature. Thus, the true specific gravity of a material is a primary property which is related to chemical and mineralogical composition.

4.2 This test method is particularly useful for hydratable materials which are not suitable for test with Test Method C 135.

4.3 For refractory raw materials and products the true specific gravity is a useful value for: classification, detecting differences in chemical composition between supposedly like samples, indicating mineralogical phases or phase changes, calculating total porosity when the bulk density is known, and for any other test method that requires this value for the calculation of results.

4.4 This test method is a primary standard method which is suitable for use in specifications, quality control, and research and development. It can also serve as a referee test method in purchasing contracts or agreements.

4.5 Fundamental assumptions inherent in this test method are the following:

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[1] This test method is under the jurisdiction of ASTM Committee C08 on Refractories, and is the direct responsibility of Subcommittee C08.03 on Physical Test and Properties.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 15.01.

[4] *Annual Book of ASTM Standards*, Vol 04.01.

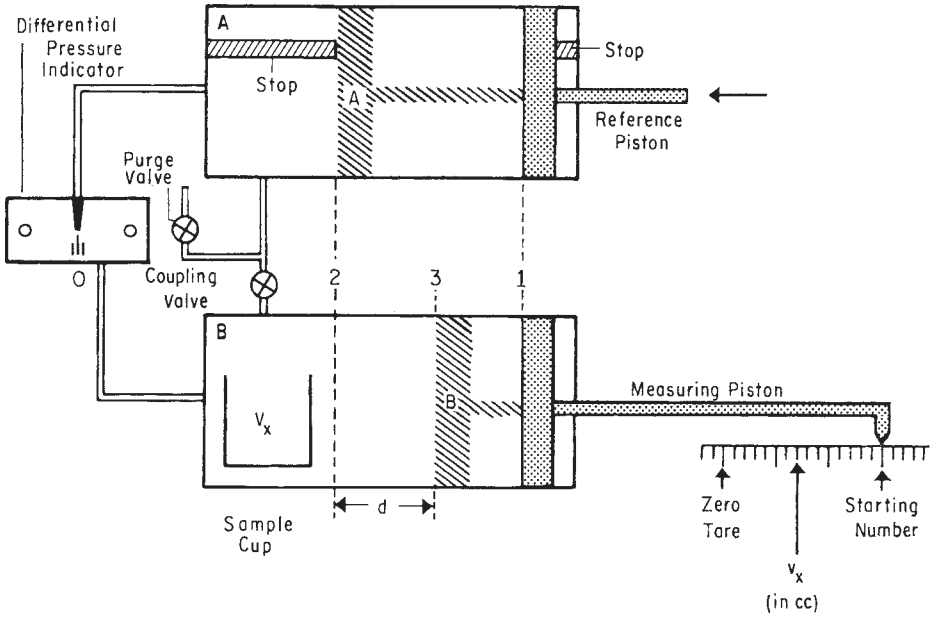

FIG. 1 Simplified Schematic Diagram

4.5.1 The sample is representative of the material in general,

4.5.2 The total sample has been reduced to the particle size specified,

4.5.3 No contamination has been introduced during processing of the sample,

4.5.4 The ignition of the sample has eliminated all free or combined water without inducing sintering or alteration,

4.5.5 An inert gas (helium) has been used in the test, and

4.5.6 The test method has been conducted in a meticulous manner.

4.5.7 Deviation from any of these assumptions negates the usefulness of the results.

4.6 In interpreting the results of this test method it must be recognized that the specified sample particle size is significantly finer than specified for Test Method C 135. Even this finer particle size for the sample does not preclude the presence of some closed pores, and the amount of residual closed pores may vary between materials or even between samples of the same or like materials. The values generated by this test method may, therefore, be very close approximations rather than accurate representations of true specific gravities. Thus, comparisons of results should only be judiciously made between like materials tested by this test method or with full recognition of potentially inherent differences between the materials being compared or the test method used.

5. Apparatus

5.1 *Analytical Balance*, 200-g capacity, minimum sensitivity 10 mg.

5.2 *Desiccator*, charged with magnesium perchlorate.

5.3 *Muffle Furnace*, capable of heating to 1000°C.

5.4 *Cylinder of Dry Helium Gas*, with regulator and gage.

5.5 *Equipment for Grinding Sample*, to pass a No. 325 (45-μm) sieve without contamination.

5.6 *Gas-Comparison Pycnometer*, [5] equipped with external purge manifold.

6. Sample Preparation

6.1 Grind a sufficient representative sample for three determinations to pass a No. 325 (45-μm) sieve. With the Beckman instrument the quantity needed is approximately 150 cm^3.

6.2 After grinding, ignite the total sample at a temperature sufficient to drive off free moisture and any undesired combined water, organic matter, etc., without inducing sintering of the powder. In the case of refractory materials that hydrate, the ignition temperature is a minimum of 600°C for 3 h.

6.3 After ignition, place the powdered sample in a desiccator charged with magnesium perchlorate and allow to cool to room temperature.

7. Procedure

7.1 Check the gas-comparison pycnometer for zero measurement and calibration as specified in the instruction manual for the instrument.

7.2 Take the cooled sample from the desiccator and rapidly fill the previously tared sample cup nearly full. Weigh to 10 mg. The sample and sample cup must be within ± 2°C of instrument temperature. With materials that hydrate, once the sample is removed from the desiccator the succeeding steps must be taken as rapidly as possible to prevent hydration.

7.3 Place the sample cup with sample in the pycnometer sample compartment and lock firmly into place. Purge the pycnometer system with dry helium gas at pressures not exceeding 2 psi (13.8 kPa).

7.4 Measure the sample volume by the standard procedure given in the instruction manual for purge atmosphere volume

[5] A suitable instrument is the Beckman Air Comparison Pycnometer manufactured by Beckman Instruments, Inc., 2500 Harbor Blvd., Fullerton, CA 92634.

measurement except that the wait period for temperature equilibration is increased to 60 s.

7.5 Repeat the volume measurement for the same sample, and take the sample volume as the average of the two measurements, which must agree within 0.05 cm^3.

8. Calculation

8.1 Calculate the true specific gravity of the sample at room temperature as compared to water at 4°C as follows:

$$S = W/V$$

where:
S = true specific gravity,
W = sample weight, g, and
V = sample volume, cm^3(average of two measurements).

9. Report

9.1 Report the true specific gravity to two decimal places as the average of the values determined on three separate samples of the material, which individual values must agree within 0.01.

10. Precision and Bias

10.1 An interlaboratory study was run in which randomly drawn test specimens of two materials (tabular alumina and Mulcoa 47) were tested for true specific gravity by gas-comparison pycnometer. Both materials were tested in 4 labs. Replicates per lab in the tabular alumina testing ranged from 3 to 9 averaging 6. Replicates per lab in the Mulcoa 47 testing ranged from 3 to 6 averaging 4.75. Number of operators ranged from 1 to 3 for both materials. Instruments used in the study were Beckman Air Comparison Pycnometers at 2 labs and a Micromeritics AccuPyc 1330 at 1 lab. The instrument for the 4th lab was not identified. Except for the lack of uniformity in number of replicate tests and the use of only two materials, Practice E 691 was followed for the design and analysis of the data, the details are given in ASTM Research Report No. C08:1013.[6]

10.2 *Test Result*—The precision information given below in the unit of measurement (g/cc) is for the comparison of the two test results. If the difference in the two test results is greater than or equal to the applicable 95 % limit there is a 95 % probability that the materials are measurably different. If the difference in results are less than the applicable 95 % limit, it cannot said with certainty that the materials are measurably different.

	Tabular Alumina	Mulcoa 47
Average Test Value	3.952	2.790
95 % repeatability limit (within laboratory)	0.026	0.016
95 % reproducibility limit (between laboratories)	0.053	0.048

The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E 177. The respective standard deviations among test results may be obtained by dividing the above limit values by 2.8.

10.3 *Bias*—No justifiable statement can be made on the bias of the procedure in this test method for measuring true specific gravity because no reference material was readily available.

11. Keywords

11.1 gas comparison pycnometer; hydratable materials; refractory materials; true specific gravity

[6] Details are given in a research report available from ASTM International Headquarters. Request RR:C08–1013.