Designation: C 914 – 95 (Reapproved 1999)

Standard Test Method for Bulk Density and Volume of Solid Refractories by Wax Immersion[1]

This standard is issued under the fixed designation C 914; 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 basic procedure for determining bulk density and volume of refractory shapes. This test is applicable to all refractory shapes or monoliths, burned or unburned, independent of composition or forming method, including materials that slake and hydrate. It is particularly suitable for determining bulk density and volume of complex shapes after forming, since results may be obtained in a matter of minutes.

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.* For a specific hazard statement, see Note 2.

2. Significance and Use

2.1 This test method may be used to quickly determine volume and bulk density of a refractory of any shape, provided it has sufficient structural integrity to permit handling. Thus it may be used on unfired or fired, strong or friable shapes.

2.2 Since the test may be performed quickly, it has found application as manufacturing in-process control as well as in characterizing finished refractory products. Also it may be used to examine specimens after other test or service exposure.

3. Apparatus

3.1 *Paraffin Wax*, fully refined, that has a known constant density, *K*, that does not change after repeated melting and cooling cycles.

NOTE 1—The paraffin waxes generally used are commercially available and have density values in the range 0.87 to 0.91 g/cm^3. Also, these waxes melt at approximately 135°F (57°C).

3.2 *Wax-Melting Container*, used to melt the wax but should not allow the wax to overheat. A container heated by hot water, preferably thermostatically controlled, is satisfactory. The wax should be heated to only slightly above the melting point to avoid flashing of the wax vapors and to permit quickly forming a uniform surface coating of wax.

NOTE 2—**Caution:** Vapors given off by molten wax ignite spontaneously at above 400°F (205°C) and should not be allowed to come in contact with the heating element or open flame.

3.3 *Balance*, capable of determining the weights of the specimens to four significant figures. Thus, specimens weighing from 100 to 999 g should be weighed to one decimal place, those from 10 to 99 g should be weighed to two decimal places, and so forth.

4. Sampling

4.1 At least five representative specimens should be chosen of the refractory to be characterized. These may be whole shapes or broken pieces, depending on the purpose of the test.

5. Procedure

5.1 *Preparation of Specimens*—The test specimens shall be dried to a constant weight by heating to 220 to 230°F (105 to 110°C) to remove entrapped moisture, which would affect the bulk density determination. This drying process may be omitted when specimens are known to be dry or when it is desired to make density determinations on moisture-containing specimens, such as brick shapes, immediately after forming.

5.2 *Initial Weight,W*—Determine the initial weight, *W*, of each test specimen in grams to four significant figures.

5.3 *Coating the Test Specimen*:

5.3.1 Coat the specimen with wax by dipping the specimen into the container of melted wax. The coating is easily applied by holding one end of the specimen and immersing one half to two thirds of it. Then, hold the waxed end, and immerse the unwaxed portion plus a small overlap into the wax to provide a complete coating.

5.3.2 Take care not to entrap air bubbles under the wax. If found, press these bubbles out so the wax conforms exactly to the surface of the specimen. Close holes in the wax coating by additional dipping in wax so the surface can be completely sealed.

5.4 *Wax-Coated Weight,P*—Determine the weight of the wax-coated specimen, *P*, in grams to four significant figures.

5.5 *Suspended Weight, S*:

5.5.1 Determine the weight of the wax-coated specimen suspended in water, *S*, in grams to four significant figures.

5.5.2 Previously counterbalance the balance with the wire

[1] This test method is under the jurisdiction of ASTM Committee C-8 on Refractories and is the direct responsibility of Subcommittee C08.03 on Physical Tests and Properties.

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or other device used to suspend the specimen in place, and immerse in water to the same depth as used when the refractory specimen is in place. This weighing may be 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.

6. Calculation

6.1 *Volume, V*:

6.1.1 Obtain the volume, V_1, of the test specimens (including the wax) in cubic centimetres as follows:

$$V_1 = P - S \quad (1)$$

NOTE 3—This assumes that 1 cm^3 of water weighs 1 g. This is true within 3 parts in 1000 for water at room temperature.

6.1.2 Obtain the volume, V_2, of the wax coating on the test specimen in cubic centimetres as follows:

$$V_2 = (P - W)/K \quad (2)$$

where:

K = density of the wax, g/cm^3.

6.1.3 Obtain the volume, V, of the test specimen by subtracting the volume of the wax coating from the total volume as calculated in 6.1.1 as follows:

$$V = V_1 - V_2 \quad (3)$$

6.2 *Bulk Density,B*—The bulk density, B, of a specimen in grams per cubic centimetre is the quotient of its initial weight divided by volume of the test specimen, excluding the volume of wax. Calculate B as follows:

$$B = W/V \quad (4)$$

7. Report

7.1 For each property, report the average of the values obtained with at least five specimens, and preferably, the individual values as well.

7.2 Report the bulk density results to two decimal places.

8. Precision and Bias

8.1 *Volume Measurement*:

8.1.1 *Interlaboratory Test Program*—Interlaboratory study was conducted by five laboratories using three replications and two duplicate runs on the same specimen. The specimen was 2½ in. (63.5 mm) series 9 in. (229 mm) straights of oxynitride bonded silicon carbide cut into quarter bricks approximately 4.5 by 2.25 by 1.5 in. (114 by 57 by 38 mm).

8.1.2 *Precision*:

8.1.2.1 *Repeatability*— Two test results, each composed of five specimens from one laboratory, should be considered significantly different at the 95 % confidence level, if their difference exceeds the Repeatability Interval, Ir, for the grand average in Table 1.

8.1.2.2 *Reproducibility*— Two test results, each composed of five specimens from two laboratories, should be considered significantly different at the 95 % confidence level, if their difference exceeds the Reproducibility Interval, IR, for the grand average in Table 1.

8.1.3 *Bias*—No justifiable statement can be made since there is no accepted reference material and the true values of volume cannot be established by an accepted reference method.

8.2 *Bulk Density Measurement*:

8.2.1 *Interlaboratory Test Program*—Interlaboratory study was conducted by five laboratories using three replications and two duplicate runs on the same specimen. The specimen was 2½ in. (63.5 mm) series 9 in. (229 mm) straights of oxynitride bonded silicon carbide cut into quarter bricks approximately 4.5 by 2.25 by 1.5 in. (114 by 57 by 38 mm).

8.2.2 *Precision*:

8.2.2.1 *Repeatability*— Two test results, each composed of five specimens from one laboratory, should be considered significantly different at the 95 % confidence level, if their difference exceeds the Repeatability Interval, Ir, for the grand average in Table 2.

8.2.2.2 *Reproducibility*— Two test results, each composed of five specimens from two laboratories, should be considered significantly different at the 95 % confidence level, if their

TABLE 1 Volume Measurement

Precision					
		Standard Deviation			
Material	Average	Within Laboratories, Sr	Between Laboratories, SL	Repeatability Interval, Ir	Reproducibility Interval, IR
A	397.17	0.695	0.377	1.965	1.066
B	408.51	0.592	0.245	1.674	0.693
C	408.82	0.859	0.307	2.429	0.868
D	410.56	0.801	0.370	2.265	1.046
E	411.80	0.885	0.494	2.503	1.397
Grand Average	407.37	0.766	0.359	2.167	2.933
Relative Precision					
		Coefficient of Variance			
Material	Average	Within Laboratories, % Vr	Between Laboratories, % VL	Relative Repeatability Interval, % Ir	Relative Reproducibility Interval, % IR
A	397.17	0.175	0.095	0.495	0.269
B	408.51	0.145	0.060	0.410	0.170
C	408.82	0.210	0.075	0.594	0.212
D	410.56	0.195	0.090	0.552	0.255
E	411.80	0.215	0.120	0.608	0.339
Grand Average	407.37	0.188	0.088	0.532	0.249

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TABLE 2 Bulk Density Measurement

Precision					
		Standard Deviation			
Material	Average	Within Laboratories, Sr	Between Laboratories, SL	Repeatability Interval, Ir	Reproducibility Interval, IR
A	2.585	0.0033	0.0066	0.0094	0.0210
B	2.569	0.0033	0.0026	0.0094	0.0120
C	2.619	0.0041	0.0009	0.0115	0.0120
D	2.622	0.0033	0.0020	0.0094	0.0110
E	2.599	0.0024	0.0024	0.0067	0.0094
Grand Average	2.599	0.0033	0.0029	0.0093	0.0131
Relative Precision					
		Coefficient of Variance			
Material	Average	Within Laboratories, % Vr	Between Laboratories, % VL	Relative Repeatability Interval, % Ir	Relative Reproducibility Interval, % IR
A	2.585	0.129	0.257	0.36	0.81
B	2.569	0.130	0.103	0.37	0.47
C	2.619	0.156	0.033	0.44	0.45
D	2.622	0.127	0.077	0.36	0.42
E	2.599	0.091	0.091	0.26	0.36
Grand Average	2.599	0.127	0.112	0.36	0.50

difference exceeds the Reproducilibity Interval, IR, for the grand average in Table 2.

8.2.3 *Bias*—No justifiable statement can be made since there is no accepted reference material and the true values of bulk density cannot be established by an accepted reference method.

9. Keywords

9.1 bulk density; refractory shapes; solid refractories; volume; wax immersion

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