Designation: C 544 – 03 (Reapproved 2008)

Standard Test Method for Hydration of Dead Burned Magnesite or Periclase Grain[1]

This standard is issued under the fixed designation C 544; 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 measurement of the relative resistance of magnesia grain to hydration.

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:* [2]

C 92 Test Methods for Sieve Analysis and Water Content of Refractory Materials

C 357 Test Method for Bulk Density of Granular Refractory Materials

C 456 Test Method for Hydration Resistance of Basic Bricks and Shapes

C 493 Test Method for Bulk Density and Porosity of Granular Refractory Materials by Mercury Displacement (Discontinued 2002)[3]

3. Significance and Use

3.1 This test method determines relative hydration resistance of magnesia grain.

3.2 This test method is used in industry to evaluate grain samples and is used for specification purposes in some cases.

3.3 Care must be taken in interpreting the data.

4. Apparatus

4.1 *Autoclave*, suitable for operation at 80 psi (552 kPa) at 324°F (162°C) and equipped with pressure- and temperature-measuring devices and safety equipment.

NOTE 1—A suitable apparatus is shown in Fig. 1 of Test Method C 456.

TABLE 1 Material Properties

Material Number	1	2	3	4
Chemical Analysis (*Calcined Basis*), %:				
Silica (SiO_2)	1.0	3.3	0.7	0.55
Alumina (Al_2O_3)	0.5	1.7	0.2	0.2
Iron Oxide (Fe_2O_3)	0.5	3.8	0.2	0.2
Chromic Oxide (Cr_2O_3)	. . .	2.2	. . .	. . .
Lime (CaO)	3.0	11.0	0.7	2.2
Boron Oxide (B_2O_3)	. . .	. . .	0.03	<0.01
Magnesia (MgO)	95.0	78.0	98.2	96.75
Total, %	100.0	100.0	100.0	99.9
Bulk Specific Gravity:				
By Water Displacement (ASTM C 357)	. . .	. . .	3.21	3.32
By Mercury Displacement (ASTM C 493)	3.36	3.15	3.27	3.37

4.2 *Standard Sieves*, ASTM No. 6 (3.35 mm), No. 12 (1.70 mm), No. 20 (850 µm), No. 40 (425 µm), and No. 50 (300 µm).

NOTE 2—The equivalent Tyler Standard Series sieves described in Test Methods C 92 may be substituted for the ASTM sieves.

5. Procedure

5.1 Remove the material retained on a No. 6 (3.35-mm) sieve, and crush it to pass the No. 6 sieve to obtain the maximum amount of coarse material. Recombine with the portion passing the No. 6 sieve, and screen the resultant sample to remove all material passing a No. 40 (425-µm) sieve. If necessary, dry at 220 to 230°F (105 to 110°C).

5.2 Separate this sample into the following three fractions:

Passing Sieve No.	Retained on Sieve No.
6 (3.35 mm)	12 (1.70 mm)
12 (1.70 mm)	20 (850 µm)
20 (850 µm)	40 (425 µm)

5.3 Prepare a 100-g specimen by using equal parts by weight of the three sizes listed in 5.2.

5.4 Add sufficient water to the autoclave to maintain 80 psi (552 kPa) at 324°F (162°C) for the duration of each 5-h test, but not enough to permit contact with any of the specimens.

5.5 Place each weighed specimen in a No. 3 porcelain crucible, and cover with a loosely crimped piece of aluminum foil to protect the specimen from drip or condensate. Dry the covered crucibles in a forced-air dryer at 220 to 230°F (105 to 110°C) for at least 2 h.

[1] This test method is under the jurisdiction of ASTM Committee C08 on Refractories and is the direct responsibility of Subcommittee C08.04 on Chemical Behaviors.

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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.

[3] Withdrawn.

TABLE 2 Repeatability and Reproducibility Data

Material Number	Average, $\bar{X}$	Standard Within, S_r	Deviation Between, S_R	Repeatability Interval, r	Reproducibility Interval, R	Coefficient of Variation		Relative Repeatability, % r	Relative Reproducibility, % R
						Within Lab, V_r	Between Labs, V_R		
1	63.61	2.818	4.447	7.891	12.45	4.430	6.991	12.41	19.57
2	37.06	3.082	3.811	8.628	10.67	8.316	10.28	23.28	28.79
3	21.04	3.108	5.947	8.703	16.65	14.77	28.27	41.36	79.13
4	60.27	6.254	7.085	17.51	19.84	10.38	11.76	29.05	32.92

5.6 Place the preheated rack containing the specimens in the autoclave. Heat the autoclave with the pressure-release valve open; after a steady flow of steam is obtained through the valve, continue to purge for 3 min to remove all air, close the valve, and bring the autoclave to 80 psi (552 kPa) at 324°F (162°C) in a total time of 1 h. Maintain the autoclave at 80 ± 5 psi (552 ± 35 kPa) and 324 ± 4°F (162 ± 2°C) for 5 h.

5.7 Allow sufficient cooling to lower the autoclave to 20 to 30 psi (138 to 207 kPa) with the release valve closed, and then carefully open the relief valve to reduce the autoclave to atmospheric pressure in a total time between 30 and 60 min. Remove the specimens and dry to constant weight at 220 to 230°F (105 to 110°C) in a forced-air dryer. Record the individual specimen weight, *G*.

5.8 Place each specimen with the cover and the pan on a No. 50 (300-µm) sieve, and screen according to Test Methods C 92 for dry sieve analysis. Weigh the amount of material retained on the No. 50 sieve. Record the individual specimen weight, *H*.

6. Calculation and Report

6.1 Calculate the percentage hydration as follows:

$$\text{hydration, \%} = [(G - H)/\text{G}] \times 100$$

where:

G = weight of dried specimen after hydration, g, and
H = weight of hydrated specimen retained on a No. 50 (300-µm) sieve, g.

6.2 Report the amount of hydration of each specimen as the percentage of material passing the No. 50 (300-µm) sieve after the hydration test.

7. Precision and Bias

7.1 *Interlaboratory Study*:

7.1.1 An interlaboratory test program between six laboratories was conducted. Each laboratory tested four magnesia materials. Each of the four magnesia materials was tested four times. Typical chemical analyses and bulk specific gravity data on the four magnesia materials appear in Table 1.[4]

7.1.2 No ruggedness test was run on this test method prior to conducting the interlaboratory study.

7.2 *Precision*:

7.2.1 *Repeatability*—The maximum permissible difference due to test error between two test results obtained by one operator on the same material is given by the repeatability interval and the relative repeatability interval (coefficient of variation). The 95 % repeatability intervals are given in Table 2. Two test results that do not differ by more than the repeatability interval will be considered to be from the same population, and, conversely, two test results that do differ by more than the repeatability interval will be considered to be from different populations.

7.2.2 *Reproducibility*—The maximum permissible difference due to test error between two test results obtained by two operators in different laboratories on the same type of material using the same type of test equipment is given by the reproducibility interval and relative reproducibility interval (coefficient of variation). The 95 % reproducibility intervals are given in Table 2. Two test results that do not differ by more than the reproducibility interval will be considered to be from the same population, and, conversely, two test results that do differ by more than the reproducibility interval will be considered to be from different populations.

7.3 *Bias*—This test method does not lend itself to a statement of bias.

8. Keywords

8.1 autoclave; grain; hydration resistance; magnesite; periclase; refractories

[4] These data were not measured on the actual samples tested in the hydration test, but are representative of the materials used in the interlaboratory study.