



# Standard Test Methods for Residual Carbon, Apparent Residual Carbon, and Apparent Carbon Yield in Coked Carbon-Containing Brick and Shapes <sup>1</sup>

This standard is issued under the fixed designation C 831; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover the determination of residual carbon content in carbon-bearing brick and shapes after a prescribed coking treatment. They provide two procedures. The first procedure is based on the combustion of carbon and its measurement as carbon dioxide. However, when using the first procedure for articles that contain silicon carbide or other carbides, no distinction will be made between carbon present in the form of a carbide and carbon present as elemental carbon. The second procedure provides a method for calculating apparent residual carbon (on the basis of weight loss after igniting the coked specimens), apparent carbonaceous material content, and apparent carbon yield. If the second procedure is used for brick or shapes that contain metallic additives or carbides, it must be recognized that there will be a weight gain associated with the oxidation of the metals, or carbides, or both. Such a weight gain can change the results substantially and this must be kept in mind when interpreting the data.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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:* <sup>2</sup>

C 571 Methods for Chemical Analysis of Carbon and Carbon-Ceramic Refractories

D 2906 Practice for Statements on Precision and Bias for Textiles

E 11 Specification for Wire-Cloth Sieves for Testing Purposes

## 3. Significance and Use

3.1 These test methods are designed for use with carbon-containing products. The residual carbon content of a coked carbon containing brick or shape is an indication of how much carbon may be available, in service, to resist slag attack on, or oxidation loss of, that body. Apparent carbon yield gives an estimate of the relative efficiency of the total carbonaceous matter to be retained as residual carbon.

3.2 Residual carbon has a direct bearing on several properties of a pitch or resin containing refractory such as ignited porosity, density, strength, and thermal conductivity.

3.3 These test methods are suitable for product development, manufacturing control and specification acceptance.

3.4 These test methods are very sensitive to specimen size, coking rates, etc., therefore, strict compliance with these test methods is critical.

3.5 Appreciable amounts of reducible components, such as  $\text{Fe}_2\text{O}_3$ , will have a noticeable effect on the results. Thus, values obtained by these test methods will be different when brick removed from service is tested. This must be kept in mind when attempting to use these test methods in an absolute sense.

3.6 Oxidizable components such as metals and carbides can have a noticeable effect on the results. This must be kept in mind when using the second procedure, which is based on measuring weight loss after igniting the coked specimens.

3.7 Testing of brick or shapes that contain magnesium metal presents special problems since this metal is highly volatile and substantial amounts of the magnesium can be lost from the sample during the coking procedure. This must be kept in mind when interpreting the results of testing of brick that contain magnesium. In addition, magnesium can react readily with atmospheric humidity. This must be kept in mind when storing brick that contain magnesium.

## 4. Apparatus

4.1 *For Coking:*

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee C08 on Refractories and are the direct responsibility of Subcommittee C08.04 on Chemical Behaviors.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

4.1.1 *Gas or Electric Furnace* with heating chamber capable of receiving the coking box shown in Fig. 1.

NOTE 1—Samples should not be subjected to thermal gradients greater than 40°F (22°C) during heatup. In electric furnaces with silicon carbide heating elements, the length of the box should be parallel to these elements.

4.1.2 *Inner and Outer Box*, stainless steel (or equivalent alloy), as shown in Figs. 1-3.

4.2 *For CO<sub>2</sub> Absorption*:

4.2.1 *Laboratory Pulverizer*<sup>3</sup> designed to provide a sealed, dustproof grinding chamber, and having a capacity of at least 50 g of sample.

4.2.2 *Combustion-Tube Furnace* capable of operating at 183°F (1000°C)

4.2.3 *CO<sub>2</sub>-Absorption Train* as described in Fig. 4 and in Method C 571

NOTE 2—Commercial automatic and semi-automatic carbon determinators may replace the apparatus described in 4.2.2 and 4.2.3.

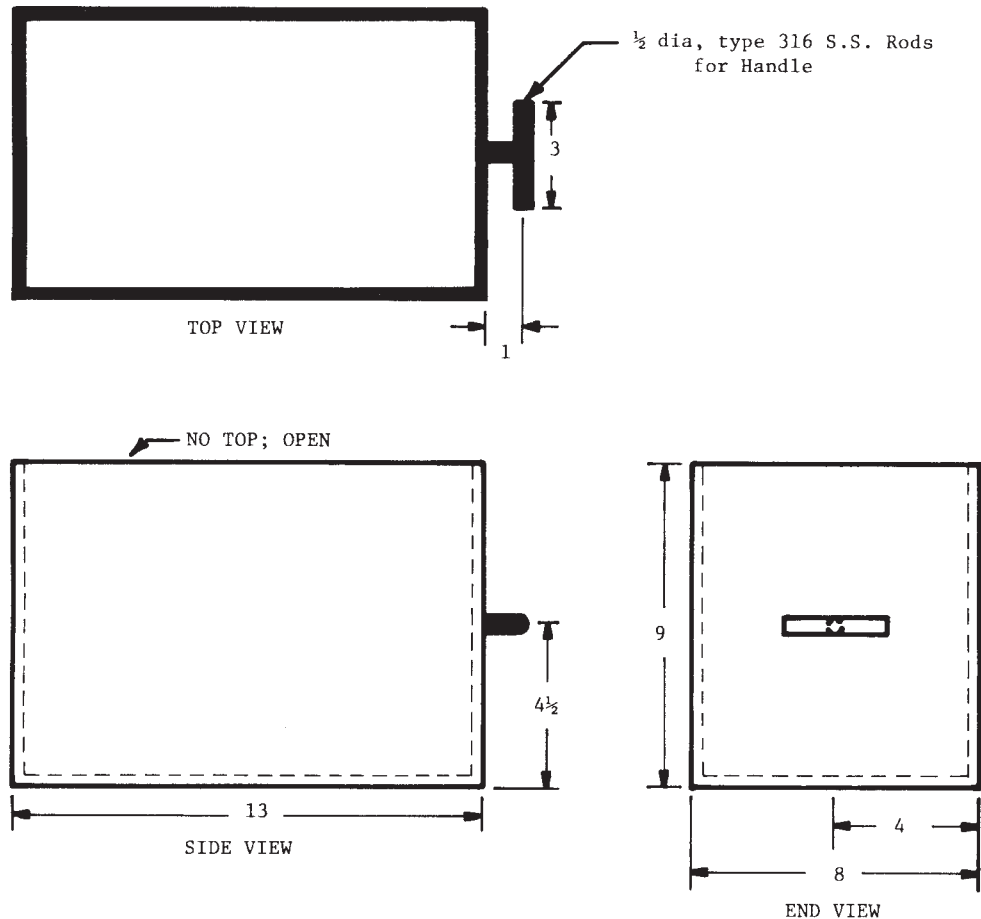
4.3 The precision obtained with these instruments shall meet the requirements specified in Section 10.

**5. Preparation of Test Specimens**

5.1 This method assumes that the number of specimens tested will be a statistically valid sample of the entire lot of brick or shapes being evaluated. The exact number is usually arrived at by mutual agreement between parties concerned.

5.2 Although sample brick from either the 4½-in. (114-mm) or the 6-in. (152-mm) series may be tested, it is preferable to use the larger size for the test. Cut slices 1 ± 1/32 in. (25 ± 0.8 mm) in thickness perpendicular to the length at the mid-section of each sample brick or shape. As shown in Fig. 5, the nominal

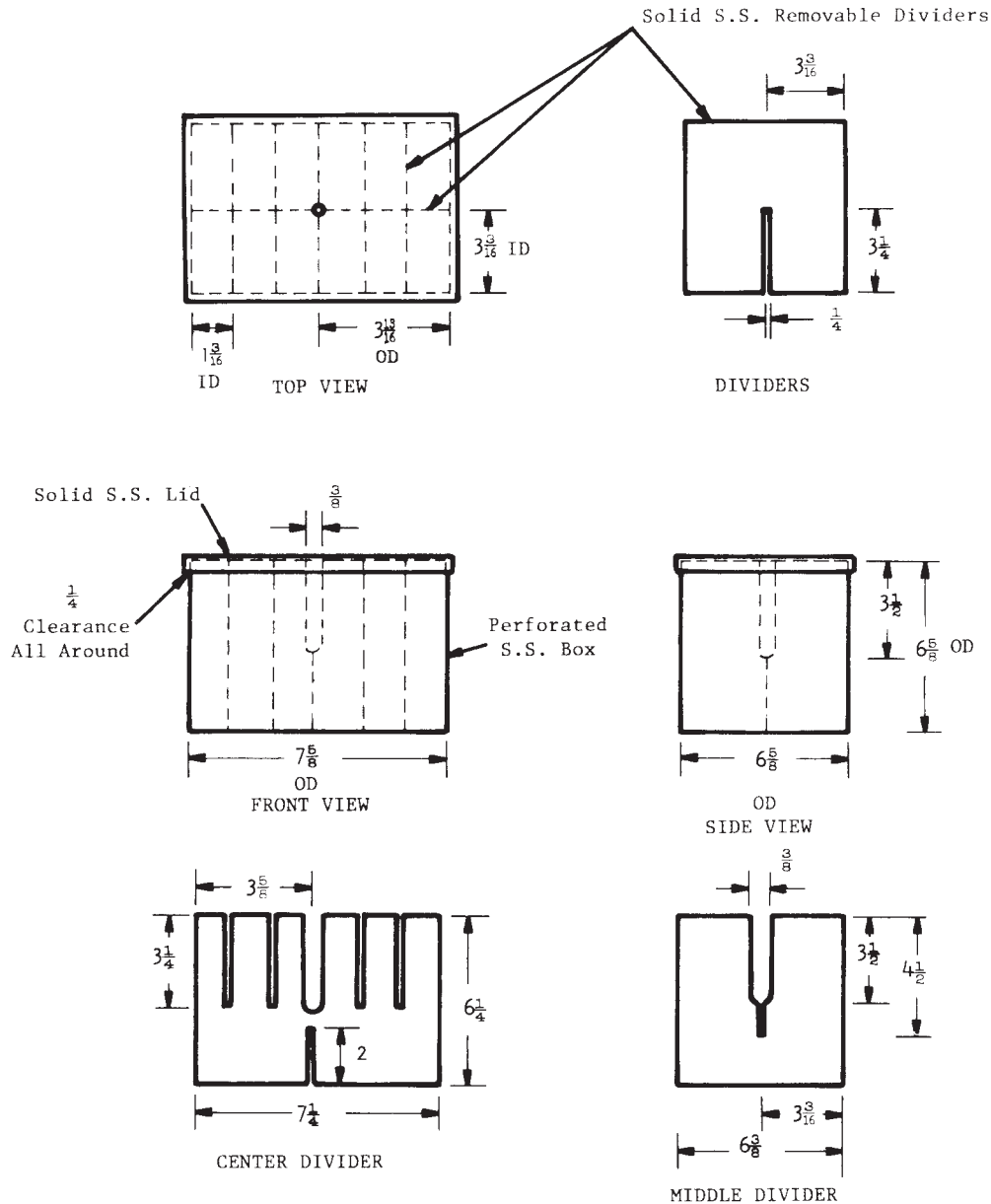
<sup>3</sup> Typical grinders are: Blueler Mill, Applied Research Laboratories, Sunland, CA; Laboratory Disc Mill, Angstrom, Inc., Bellville, MI; and Shatter Box, Spex Industries, Inc., Metuchen, NJ.



NOTE 1—Material specified is type 316 stainless steel (S.S.) or other suitable alloy (¼ in. (6 mm) recommended).  
 NOTE 2—All dimensions minimum outside diameter.

		Metric Equivalents						
in.	1/2	1	3	4	4½	8	9	13
mm	13	25	76	102	114	203	229	330

FIG. 1 Outer Coking Box (Dimensions are in Inches)



NOTE 1—Type 316 stainless steel (S.S.) or other suitable alloy, 14 gage (1.984 mm). Perforated S.S.: 14 gage.  $\frac{3}{16}$ -in. (5-mm) diameter perforations,  $\frac{1}{2}$ -in. (13-mm) centers, 11 % open.

NOTE 2—Dimensions are in inches.

Metric Equivalents

in.	$\frac{1}{4}$	$\frac{3}{8}$	$1\frac{1}{16}$	2	$3\frac{3}{16}$	$3\frac{1}{4}$	$3\frac{1}{2}$	$3\frac{5}{8}$	$3\frac{13}{16}$	$4\frac{1}{2}$	$6\frac{1}{4}$	$6\frac{3}{8}$	$6\frac{5}{8}$	$7\frac{1}{4}$	$7\frac{5}{8}$
mm	6	10	30	51	81	83	89	92	97	114	159	162	168	184	194

FIG. 2 Inner Coking Box

size of each slice shall be 1 by 3 by 6 in. (25 by 76 by 152 mm). The two 1 by 3-in. faces and the two 1 by 6-in. faces must be original surfaces.

5.3 Test specimens may be cut wet or dry except for products capable of hydration, such as dolomite brick, which must be cut dry and stored in a dry container prior to coking.

5.4 Specimens that are cut wet must be dried immediately with a paper or cloth towel and flash dried. For pitch-

impregnated samples, flash drying should be done at a sufficiently low temperature to avoid “weeping” of pitch from the pores of the brick. Drying can usually be done on a forced-air dryer at 220°F (105°C) by limiting exposure to 5 to 10 min. Repeat if necessary. These drying procedures are especially important for metal-containing brick because hydration of the metals can occur. Specimens containing a coating of pitch on

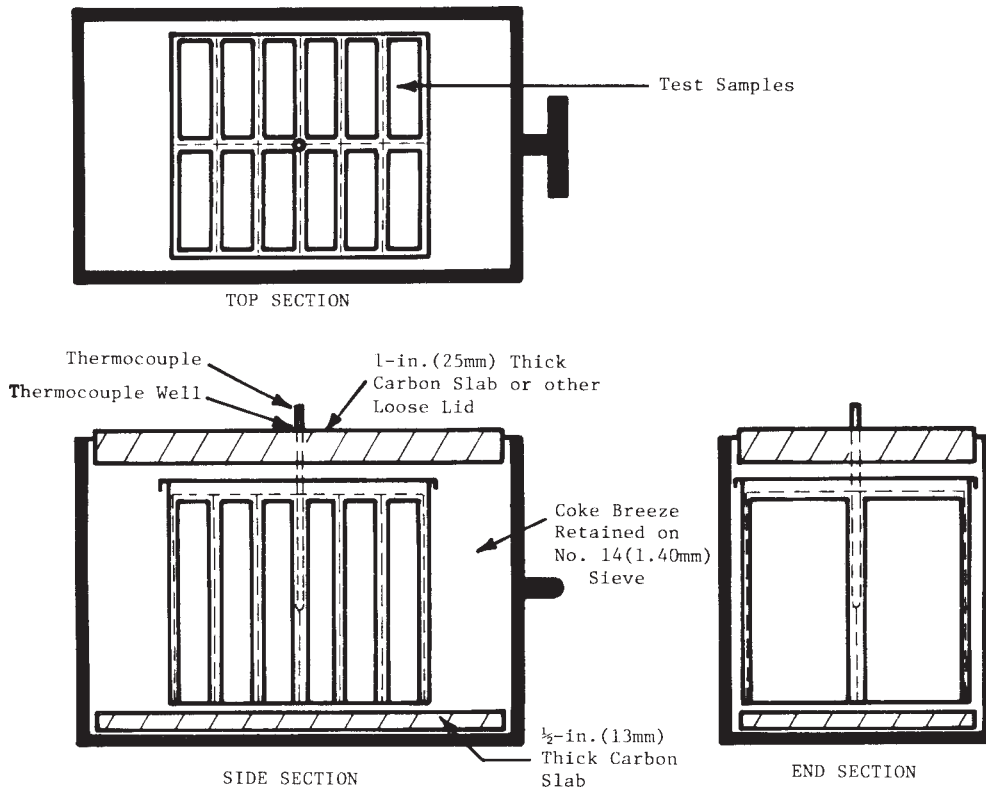
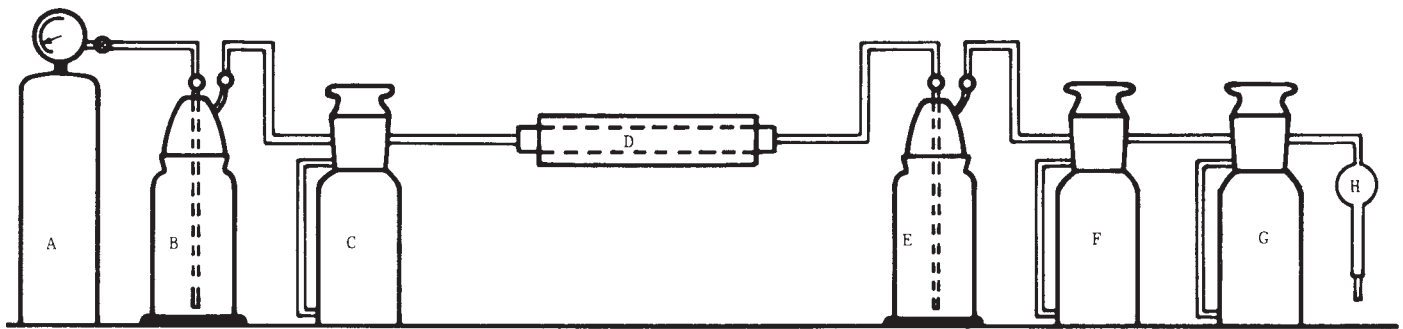


FIG. 3 Coking Box Arrangement



Key

- <sup>A</sup> Oxygen supply with gages and needle-valve control.
- <sup>B</sup> Drechsel bottle containing KOH solution (757 g/litre) for bubble count and removal of CO<sub>2</sub>.
- <sup>C</sup> Nesbitt absorption bulb containing anhydrous magnesium perchlorate (Mg(ClO<sub>4</sub>)<sub>2</sub>).
- <sup>D</sup> Gastight combustion tube of porcelain, sillimanite, fused quartz, or zircon.
- <sup>E</sup> Drechsel bottle with KMnO<sub>4</sub> solution (50 g/litre) to remove SO<sub>2</sub>.
- <sup>F</sup> Nesbitt absorption bulb containing anhydrous Mg(ClO<sub>4</sub>)<sub>2</sub>.
- <sup>G</sup> Nesbitt absorption bulb containing indicating soda-asbestos to absorb CO<sub>2</sub> liberated by the sample in combustion tube.
- <sup>H</sup> Drying tube filled one half with indicating soda-asbestos and one half with anhydrous Mg(ClO<sub>4</sub>)<sub>2</sub> (to protect CO<sub>2</sub>-absorption bulb from the atmosphere).

NOTE—Plug the absorption bulbs containing dry reagents with glass wool at both ends. The KOH solution should be replaced daily with fresh reagent.

FIG. 4 CO<sub>2</sub>-Absorption Train

uncut surfaces, as is typical of an impregnation process, must be scraped clean prior to drying.

5.5 Weigh all specimens after drying to constant weight ( $\pm 0.2$ g), recording weight to the nearest 0.1 g. This weight is "as-received weight, A," (This step may be omitted if residual carbon is to be determined by CO<sub>2</sub> absorption, as indicated in 1.1.)

## 6. Procedure for Coking

6.1 Place the test specimens randomly into the inner box, Fig. 2

NOTE 3—Burned pitch-impregnated magnesite brick should *not* be coked with tempered, tar-bonded, or dolomite brick because of carbon pickup by the impregnated samples and disruption of the bottom of

Sample Cut From Center of Brick

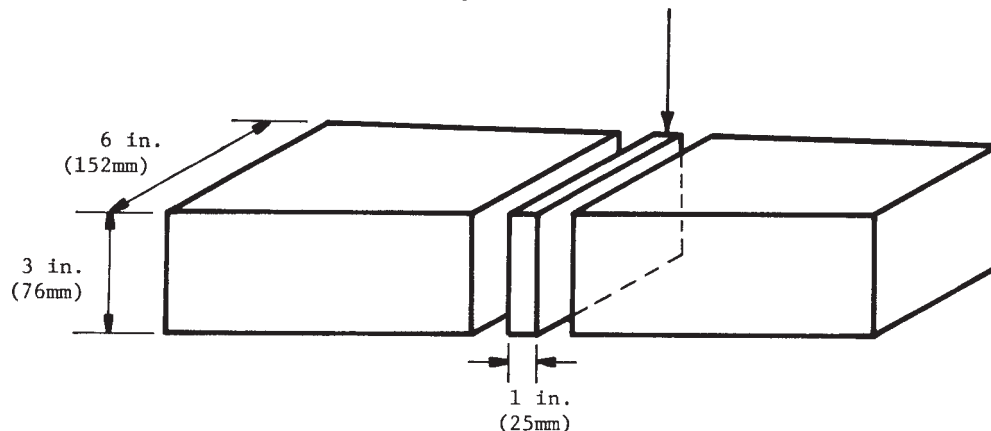


FIG. 5 Location of Test Specimen

tempered samples. Pitch-bonded, pitch-bonded tempered magnesite brick and dolomite brick may be coked in the same box or coking run.

NOTE 4—The number of samples coked per run should be constant within a laboratory. Dummy uncoked samples consistent with Note 3 may be used to fill any empty positions in the inner box.

6.2 Place the inner box into the center of the outer box (Fig. 3), on the bottom of which has first been placed a 1/2-in. (13-mm) slab of carbon, covered with a thin layer of dust-free metallurgical-grade coke breeze (No. 14 (1.40-mm) sieve size) (Note 5). To ensure that the coke breeze is free of moisture which could oxidize carbon during cooking, dry the coke at 400°F (205°C) for 24 h, and keep in a closed container at room temperature until needed.

NOTE 5—Detailed requirements for sieves are given in Specification E 11

6.3 Place the thermocouple well into the center of the inner box and put the lid on the inner box. The thermocouple well must be long enough to extend above the cover of the outer box.

6.4 Cover the inner box with metallurgical-grade coke breeze retained on a No. 14 sieve and place a loose-fitting lid over the coke breeze (see Fig. 3). Pack the coke breeze between the edges of the lid and box.

6.5 Place the coking-box assembly (Fig. 3) into the furnace, and insert a calibrated thermocouple into the thermocouple well.

6.6 Heat the furnace so that the thermocouple within the box registers 250°F (120°C) after the first hour, then heat the furnace so that the box is heated at a rate of  $400 \pm 20^\circ\text{F}$  ( $220 \pm 11^\circ\text{C}$ )/h to  $1800 \pm 20^\circ\text{F}$  ( $980 \pm 11^\circ\text{C}$ ).

6.7 Hold the temperature for  $3 \pm 1/2$  h, starting from the time 1780°F (970°C) is reached in the inner box.

6.8 After completing the hold period, shut off the furnace and allow the coking box to cool naturally within the furnace.

6.9 Remove the samples from the coking box after the box has cooled sufficiently to handle. After removing specimens from the inner box, clean by brushing carefully with a nylon or natural bristle brush to remove clinging particles. The proceed to either of the two alternatives for analyzing the specimens.

NOTE 6—After each run, clean the muffle and the bottom carbon plate of any adhering coke breeze.

6.10 Samples that contain dolomite or aluminum metal should be stored in a sealed container containing dessicant in the time interval between coking and measurement of carbon content. This is to prevent hydration of dolomite or aluminum carbide. The aluminum carbide is formed by reaction between aluminum and carbon in the shape during the coking operation. Aluminum carbide can react with a water source such as atmospheric humidity to form methane. Care should be taken since methane can be an explosion hazard.

## CO<sub>2</sub> ABSORPTION (FIRST ALTERNATIVE PROCEDURE)

### 7. Preparation of Sample

7.1 A sample consists of a single slice or multiple specimens of brick prepared as described in Sections 5 and 6.

7.2 Crush the sample in a laboratory jaw crusher, or other impact-type crusher, to pass a No. 4 (4.75-mm) sieve (Note 5). Thoroughly mix the crushed sample and reduce to approximately 50 g by quartering or riffing.

7.3 Place the sample in the laboratory pulverizer and grind to 100 % passing a No. 100 (150 μm) sieve. This takes approximately 90 to 100 s. Promptly transfer the ground sample to a suitable airtight container.

NOTE 7—Extreme care must be taken during the entire sample preparation to avoid loss of carbon by segregation or dusting. About 60 % of the variance in this procedure is in this step.

### 8. Procedure

8.1 With the furnace at operating temperature, pass oxygen through the absorption train until the CO<sub>2</sub>-absorption bulb attains constant weight (usually 15 to 30 min). Adjust the oxygen pressure and flow rate to provide 120 to 150 bubbles per minute through the bubbling tower. Close the stopcock, remove the absorption bulb from the train, cool to room temperature, and weigh to the nearest 0.1 mg.

8.2 Into a previously ignited combustion boat, weigh a 0.1 to 1.0 g sample to the nearest 0.1 mg. Return the weighed CO<sub>2</sub> absorption bulb to the train and open the stopcock. Then place the combustion boat with sample in the combustion tube and immediately reseal the train. Adjust the flow of oxygen as before (8.1), heat the furnace to 1740 to 1830°F (950 to

1000°C), and maintain until the CO<sub>2</sub> adsorption bulb attains constant weight (usually 45 to 60 min).

8.3 Remove the absorption bulb from the train, close the stopcock, cool to room temperature, and reweigh. The increase in weight is the CO<sub>2</sub> won from the sample by combustion of the carbon.

## 9. Calculation and Report

9.1 Calculate the percentage of residual carbon in the sample as follows:

$$\text{Residual carbon, \%} = \frac{\text{wt of CO}_2 \times 0.2729 \times 100}{\text{wt of sample}} \quad (1)$$

9.2 Run the determinations in duplicate. Results shall not vary by more than  $\pm 0.05$  % stated in terms of the whole sample as 100 %. If satisfactory checks are not obtained, repeat the analysis in duplicate. Report at least two individual analyses per slice.

## 10. Precision and Bias<sup>4</sup>

10.1 An interlaboratory study was conducted in 1970 using a nested experimental design wherein a composite of several sizes of magnesite grain and lampblack was mixed in accurately weighed proportions, divided into four samples, and sent to four laboratories for testing. Each laboratory split its sample into four specimens, ground them for analysis and made two replicate determinations on each. The components of variance (Note 8) of the results given in terms of standard deviations were found to be as follows:

	Carbon Content, %
Grand mean	4.572
Between laboratories ( $\sigma_L$ )	$\pm 0.0778$
Between samples ( $\sigma_S$ )	$\pm 0.0987$
Between replicates ( $\sigma_R$ )	$\pm 0.0161$

NOTE 8—A procedure for calculating precision is fully described in Practice D 2906. There is no known means for determining the bias of these test methods.

10.2 On the basis of the components of variance in 10.1, we would expect two averages of an equal number of specimens tested by this test method to be considered different at the 95 % probability level if their difference exceeds the values below (for  $t = 1.96$ ) (assume that two replicates are always used per test method:

Number of Samples in Each Average	Between Samples Within One Laboratory	Between Two Laboratories
1	0.274	0.350
6	0.116	0.245
12	0.085	0.232

10.3 These precision data may not be applicable for samples with substantially higher carbon contents or for samples that contain metals.

## IGNITION LOSS (SECOND ALTERNATIVE PROCEDURE)

### 11. Procedure

11.1 Weigh all specimens to the nearest 0.1 g and record as “coked weight, B.”

11.2 Place specimens on a layer of magnesia grain in a kiln or furnace.

11.3 Heat specimens in an air atmosphere (preferably circulating) at 500 to 700°F (280 to 380°C)/h to a temperature between 1800 and 2200°F (980 to 1205°C). For alumina-silica refractories, ignition temperature should be limited to 1800°F.

11.4 Hold the selected temperature for a minimum of 8 h (depending on the temperature in 11.3), or until a constant weight ( $\pm 0.2$  g) is obtained (Note 9).

NOTE 9—Samples containing 20 % or more carbon or samples containing oxidation inhibitors may require longer hold times of up to 40 h at a temperature of 2000°F (1095°C).

11.5 At the end of the soak, shut off the furnace and cool the specimens naturally within the furnace.

11.6 Weigh ignited specimens to the nearest 0.1 g and record as “ignited weight, C.”

## 12. Calculation and Report

12.1 The following equations apply:

$$\text{Apparent residual carbon (RC), \%} = \frac{B - C}{B} \times 100 \quad (2)$$

$$\text{Loss on ignition (LOI), (\% \text{ apparent pitch})} = \frac{A - C}{A} \times 100 \quad (3)$$

$$\text{Apparent carbon yield (CY), \%} = \frac{B - C}{A - C} \times 100 \quad (4)$$

where:

A = as-received weight (5.5),  
B = coked weight (11.1), and  
C = ignited weight (11.6).

12.2 Report the average, standard deviation, and number of specimens tested, retaining two significant figures.

## 13. Precision and Bias

13.1 *Interlaboratory Test Program*—A round-robin comparison among five laboratories was completed in early 1973. Each laboratory received two adjacent specimens from each of twelve pitch-impregnated, 95 % MgO class, 3 by 6-in. (76 by 152-mm) series brick of one brand. A second round-robin comparison was run in 1994 among three laboratories.<sup>4</sup> Each laboratory received five specimens each of a 20 % carbon MgO-carbon brick with a metal addition and without a metal addition.

### 13.2 Precision:

13.2.1 *Repeatability*—The maximum permissible difference due to test error between two test results obtained by one operator on the same material using the same test equipment is given by the repeatability interval and the relative repeatability interval (coefficient of variation). The 95 % repeatability intervals are given in Table 1. Two test results which do not differ by more than the repeatability interval will be considered to be from the same population and, conversely, two test results which do differ by more than the repeatability interval will be considered to be from different populations.

13.2.2 *Reproducibility*—The maximum permissible difference due to test error between two test results obtained by two operators in different laboratories on the same material using

<sup>4</sup> Supporting data for these test methods are on file at ASTM International Headquarters. Request RR:C-8-1012.



TABLE 1 Precision Statistics

Precision Data			95 %	95 %	Relative	Coefficient of Variation		Relative	Relative	
Product	Grand Average, $\bar{x}$	Standard Error, $S_e$	Deviation Between, $S_R$	Repeatability Interval, $r$	Reproducibility Interval, $R$	Precision Average, $\bar{x}$	Within Lab, $V_r$	Between Lab, $V_R$	Repeatability, % $r$	Reproducibility, % $R$
Apparent Residual Carbon, %										
95 % MgO TI	2.41	0.06	0.07	0.17	0.19	2.42	2.49	2.82	6.97	7.89
20 % MgO	17.79	0.56	0.56	1.56	1.56	17.79	3.12	2.69	8.75	7.54
20 % MgO-C w/metal	17.47	0.23	0.23	0.64	0.64	17.47	1.31	1.24	3.65	3.48
Loss On Ignition, %										
95 % MgO TI	5.30	0.09	0.07	0.24	0.20	5.30	1.60	1.32	4.49	3.70
20 % MgO	18.75	0.60	0.81	1.69	2.28	18.75	3.21	4.34	9.00	12.20
20 % MgO-C w/metal	18.32	0.35	0.43	0.98	1.21	18.32	1.91	2.36	5.33	6.61
Apparent Carbon Yield, %										
95 % MgO TI	44.30	1.30	1.50	3.64	4.20	44.30	2.94	3.39	8.22	9.43
20 % MgO	91.76	0.17	0.41	0.48	1.13	91.76	0.19	0.44	0.53	1.24
20 % MgO-C w/metal	93.36	0.29	1.39	0.81	3.88	93.36	0.31	1.49	0.87	4.16

the same test equipment is given by the reproducibility interval and the relative reproducibility interval (coefficient of variation). The 95 % reproducibility intervals are given in Table 1. Two test results which do not differ by more than the reproducibility interval will be considered to be from the same population and, conversely, two test results which do differ by more than the reproducibility interval will be considered to be from different populations.

13.3 *Bias*—No justifiable statement on bias is possible since the true property values cannot be established by an accepted reference material.

#### 14. Keywords

14.1 carbon yield; coking; loss of ignition; refractories; residual carbon

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