



## Standard Test Method for Thermal Conductivity of Unfired Monolithic Refractories<sup>1</sup>

This standard is issued under the fixed designation C 417; 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 This test method supplements Test Method C 201, and shall be used in conjunction with that test method for determining the thermal conductivity of unfired monolithic refractories.

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

1.3 *This standard does not purport to address all of the safety problems, 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 180 Method of Panel Spalling Testing Fireclay Plastic Refractories<sup>2</sup>

C 182 Test Method for Thermal Conductivity of Insulating Firebrick<sup>3</sup>

C 201 Test Method for Thermal Conductivity of Refractories<sup>3</sup>

C 862 Practice for Preparing Refractory Concrete Specimens by Casting<sup>3</sup>

E 220 Method for Calibration of Thermocouples by Comparison Techniques<sup>4</sup>

### 3. Significance and Use

3.1 The thermal conductivity of monolithic refractories is a property required for selecting their thermal transmission characteristics. Users select monolithic refractories to provide specified conditions of heat loss and cold face temperature, without exceeding the temperature limitation of the monolithic refractory. This test method establishes placement of thermocouples and positioning of test specimens in the calorimeter.

3.2 This procedure must be used with Test Method C 201 and requires a large thermal gradient and steady state condi-

tions. The results are based upon a mean temperature.

3.3 The data from this test method are suitable for specification acceptance, estimating heat loss and surface temperature, and the design of multi-layer refractory construction.

3.4 The use of these data requires consideration of the actual application environment and conditions.

### 4. Apparatus

4.1 The apparatus shall be in accordance with Test Method C 201, modified as in 4.2 of this test method, with the addition of thermocouples and refractory fiber paper, as described in Sections 6 and 7.

4.2 The furnace shall be modified by drilling a nominal  $\frac{3}{8}$ -in. (10-mm) diameter hole (Fig. 1) through the insulating firebrick in the furnace wall at each end of the center line of the 18-in. (456-mm) dimension of the furnace cavity. These holes shall be positioned so that the length of the hole will be parallel to the calorimeter surface and the bottom of the hole will coincide with the surface of the calorimeter. Copper tubing shall be placed within each hole so that a compressed-air source can be attached to one side and flexible leads to a flowmeter can be attached to the other.

4.3 A compressed-air supply and flowmeter for air.

### 5. Test Specimens

5.1 *Castable Refractories*—The test specimens may consist of either a panel 18 by 13½ by 2½ in. (456 by 342 by 64 mm), or an assembly of three straights 9 by 4½ by 2½ in. (228 by 114 by 64 mm) and six soaps 9 by 2¼ by 2½ in. (228 by 57 by 64 mm). These specimens shall be prepared as in one of the following methods and in general accordance with the manufacturer's recommendation for water content and Practice C 862.

5.1.1 *Panel Specimens*—This test specimen shall be a monolithic panel 18 by 13½ by 2½ in. (456 by 342 by 64 mm) in size, and shall be prepared in general accordance with Practice C 862, as outlined in 5.1. The panel shall be cast in a steel mold with two steel rods (Note 1) taped in place at the center line of the 18-in. length of the mold cavity. These steel rods form the slot required so that the panel will fit over the tubing used for the entrance and exhaust of air and moisture from the furnace (see Fig. 1).

NOTE 1—Two ½-in. (13-mm) diameter steel rods 2 in. (51 mm) long should have approximately ½ in. (0.8 mm) removed longitudinally to provide a flat base.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C-8 on Refractories and is the direct responsibility of Subcommittee C08.02 on Thermal Stress Resistance.

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<sup>2</sup> Discontinued, see 1984 *Annual Book of ASTM Standards*, Vol 15.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 15.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.03.

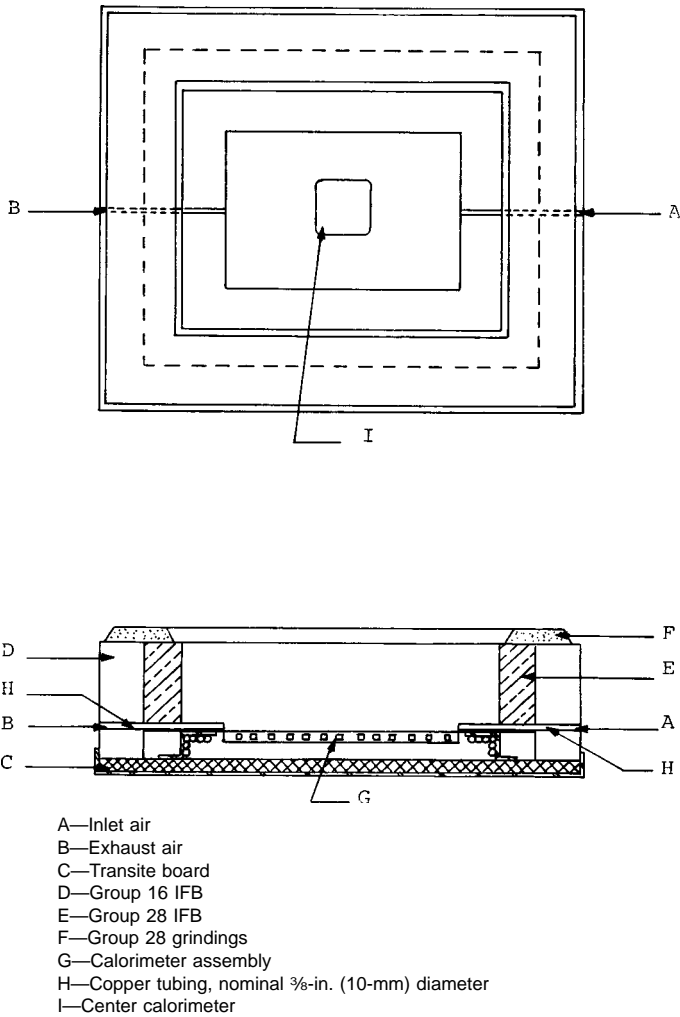


FIG. 1 Furnace Modification

5.1.2 *Straight Specimens*—This test specimen shall be three 9 by 4½ by 2½-in. (228 by 114 by 64-mm) straight brick and six 9 by 2¼ by 2½-in. (228 by 57 by 64-mm) soap brick and shall be prepared in accordance with Practice C 862, as outlined in 5.1 and 5.1.1, and by cutting as required. The 9 by 4½-in. face of the three straight brick and the 9 by 2¼-in. face of the soap brick shall be flat and parallel, and the thickness shall not vary more than ±0.01 in. (±0.3 mm). No grinding of the finish face is required if care is taken when removing the excess mix with the strikeoff bar and slicking the exposed surface with a minimum amount of troweling. Steel rods (described in Note 1) shall be used in two cavities to provide the required slots for air entry and exit.

5.2 *Plastic Refractories*—The test specimens shall be of the size and number described in 4.1 of Test Method C 201, and shall be prepared in accordance with Method C 180 and 3.2 of Test Method C 201. The soap specimens shall be prepared by cutting dry 9-in. (228-mm) straight specimens with a suitable abrasive cut-off saw. The soap brick adjacent to the 9-in. face of the guard brick shall be slotted with a suitable abrasive cut-off saw at the center line of the 9-in. length to fit over the tubing used for the entrance, and exhaust of the air and moisture.

5.3 *Specimen Curing and Drying*—After the specified curing, the specimens shall be placed in a dryer at 250°F (120°C) for a minimum of 24 h, or until constant mass has been achieved.

6. Installation of Thermocouples in Test Specimen

6.1 *Thermocouples*—Embed calibrated<sup>5</sup> thermocouples in the test specimen at two points for measurement of temperature. Use platinum-10 % rhodium/platinum, Awg Gage 28 (0.320-mm) wire in making the thermocouples.

6.2 Installation of Thermocouples:

6.2.1 For castable specimens prepared in accordance with 5.1.1, use the following thermocouple installation procedure. Place the hot junction of the thermocouples in the center of each 18 by 13½-in. (456 by 342-mm) face and just below the surface of the test specimen. Cut grooves to receive the wire in each 18 by 13½-in. face to a depth of 1/32 in. (0.8 mm) by means of an abrasive wheel 0.02 in. (0.5 mm) in thickness. The layout for the grooves allows all of the cold-junction ends of the wires to extend from one end of the specimen. Cut a groove in the center of each 18 by 13½-in. face along the 18-in. dimension and ending 1½ in. (38 mm) from the center point of the specimen. Extend the path of each groove at an angle of 90° to one end of the specimen by cutting grooves parallel to the 13½-in. edges and 1½ in. from the center point of the specimen. Before cementing the thermocouple wires in place, take measurements to obtain, within 0.01 in. (0.3 mm), the eventual distance between the center lines of the thermocouple junctions. Do this by measuring the 2½-in. (64-mm) dimension of the specimens at the location for the hot junctions and deducting the distance between the center line of each junction in its embedded position and the surface of the specimen.

6.2.2 For castable specimens prepared in accordance with 5.1.2 and plastic refractory specimens prepared in accordance with 5.2, use the following thermocouple installation procedure. Place the hot junction of the thermocouples in the center of each 9 by 4½-in. (228 by 114-mm) face, and just below the surface of the test specimen. Cut grooves to receive the wire in each 9 by 4½-in. face of the brick to a depth of 1/32 in. (0.8 mm) by means of an abrasive wheel 0.02 in. (0.5 mm) in thickness. The layout for the grooves allows all of the cold-junction ends of the wires to extend from one end of the brick. Cut a groove in the center of each 9 by 4½-in. face along the 4½-in. dimension, and ending 1 in. (25 mm) from the edge of the specimen. Before cementing the thermocouple wires in place take measurements to obtain within ±0.01 in. (±0.3 mm) the eventual distance between the center lines of the thermocouple junctions. Do this by measuring the 2½-in. (64-mm) dimension of the brick at the location for the hot junctions and deducting the distance between the center line of each junction in its embedded position and the surface of the brick.

7. Set-Up of Specimen and Silicon Carbide Slab

7.1 Specimen Set-up:

7.1.1 For castable specimens prepared in accordance with 5.1.1, use the following set-up procedure. Place two strips of

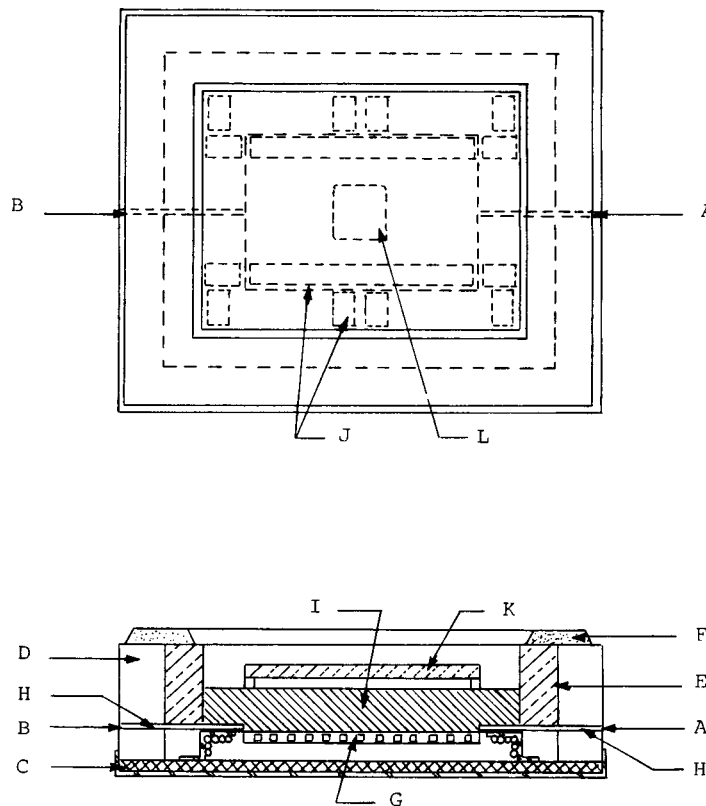
<sup>5</sup> Method E 220 specifies thermocouple calibration procedures for thermocouples.

refractory fiber paper 18 by ½ by 0.02 in. (456 by 13 by 0.5 mm) along the 18-in. dimension on the outer guard. These strips, used to prevent contact between the test material and the calorimeter assembly, also provide a passage for the flow of air. Push in a copper tube installed at each end of the furnace and position where their open ends are flush with the inside edge of the outer guard assembly. Pack the openings in the furnace walls where the tubes enter with ceramic fiber. Place the test specimen centrally over the center of the calorimeter and outer guard assembly on its 18 by 13½-in. (456 by 342-mm) face. Fill the small space between the furnace walls and the test specimen with granular insulating firebrick or ceramic fiber (Fig. 2).

7.1.2 For castable specimens prepared in accordance with 5.1.2 and plastic refractory samples prepared in accordance with 5.2, use the following set-up procedure. Place two strips of refractory fiber paper 13½ by ½ by 0.02 in. (342 by 13 by 0.5 mm) along the 13½-in. dimension of the inner guard at the outside edges. Place twelve strips of refractory fiber paper 2 by ½ by 0.02 in. (51 by 13 by 0.5 mm) on the outer guard at intervals where the soap-brick ends are placed. (See Fig. 1 of

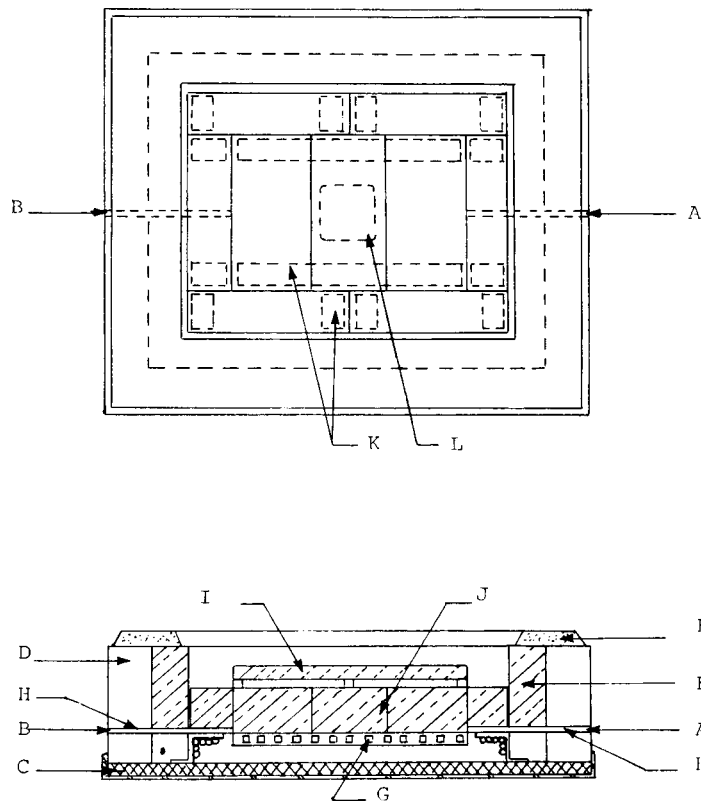
Test Method C 182.) These strips serve as spacers to prevent contact between the test material and the calorimeter assembly, and provide for passage of air. Push in the copper tubes installed at each end of the furnace and position where their open ends are flush with the inside edge of the outer guard assembly. Pack the openings in the furnace walls where the tubes enter with ceramic fiber. Place the test specimen centrally over the center of the calorimeter section on its 9 by 4½-in. (228 by 114-mm) face, place the guard brick at the sides of the test specimen so as to cover completely the calorimeter and inner guard area, and place the soap brick around the edge of the three bricks, so as to cover completely the calorimeter assembly. Fill the small space between the furnace walls and the test brick assembly with a granulated insulating firebrick or ceramic fiber (Fig. 3).

7.2 *Silicon Carbide Slab*—Place the silicon carbide slab centrally over the test specimen, spacing it 1 in. (25 mm) above the specimen or specimen assembly by placing under each corner of the slab, rectangular pieces of a high-aluminarefractory cut to measure ¾ in. (10 mm) square, and 1 in. long.



- A—Inlet air
- B—Exhaust air
- C—Transite board
- D—Group 16 IFB
- E—Group 28 IFB
- F—Group 28 grindings
- G—Calorimeter assembly
- H—Copper tubing, nominal ¾-in. (10-mm) diameter
- I—Monolithic panel, 18 by 13½ by 2½ in. (456 by 342 by 64 mm)
- J—Refractory fiber paper
- K—Silicon carbide plate, 13⅝ by 9 by ¾ in. (346 by 228 by 19 mm)
- L—Center calorimeter

FIG. 2 Monolithic Panel Specimen



- A—Inlet air
- B—Exhaust air
- C—Transite board
- D—Group 16 IFB
- E—Group 28 IFB
- F—Group 28 grindings
- G—Calorimeter assembly
- H—Copper tubing, nominal  $\frac{3}{8}$ -in. (10-mm) diameter
- I—Silicon carbide plate,  $13\frac{3}{8}$  by 9 by  $\frac{3}{4}$  in. (346 by 228 by 19 mm)
- J—Test brick, 9 by  $4\frac{1}{2}$  by  $2\frac{1}{2}$  in. (228 by 114 by 64 mm)
- K—Refractory fiber paper
- L—Center calorimeter

FIG. 3 Straight Specimen

## 8. Procedure

8.1 Place the heating chamber in position, start the water flowing through the calorimeter assembly, and apply the current to the heating unit. Maintain the rate of water flow through the calorimeter between 120 and 200 g/min and determine the flow by weighing the quantity of water collected during a measured time period. The mass of water collected shall be not less than 200 g and shall be weighed to an accuracy of 0.5 g. The rate of flow shall be constant within 1 % during the test period.

8.2 Allow the furnace to reach a temperature of 500°F (260°C) as recorded by the control thermocouple, and soak for at least 2 h. Introduce compressed air to the copper tubing and adjust the flow rate to provide 0.5 ft<sup>3</sup>/h (14.2 dm<sup>3</sup>/h). See Fig. 1 and Fig. 2 for furnace modification detail and specimen modification detail. This is determined with a flowmeter connected to the exhaust vent. After the flow is adjusted, disconnect the flexible hose and allow the exhaust to escape to

free air. After exhausting moisture for 10 h, shut off the air supply and plug the outlet only with ceramic fiber. Maintain this temperature until a condition of steady heat flow has been reached. This will require 12 to 16 h. A steady heat flow shall be that condition when the measured flow of heat into the calorimeter varies less than 2 % over a 2-h period, during which time the temperature difference between the calorimeter and the inner guard has not been more than 0.05°F (0.03°C), the hot face of the test specimen has not varied more than 5°F (3°C), and the temperature of the water entering the calorimeter has not varied at a rate of more than 1°F (0.5°C)/h.

8.3 After the steady state of heat flow has been reached, measure the temperatures in the test specimen, the rate of water flow through the calorimeter, and the temperature rise of the water flowing through the calorimeter. Take at least four sets of readings at approximately 30-min intervals during the 2-h holding period, and average these for the final values for that

particular heating chamber temperature. Calculate the thermal conductivity.

NOTE 2—From these data a preliminary thermal conductivity calculation may be made, using the estimated distances between thermocouple junctions in the test specimens.

8.4 The heating schedules shall be as follows:

8.4.1 For products not exceeding 2300°F (1260°C) use limit: Repeat 8.1, 8.2, and 8.3 at a temperature midway between the initial point and the maximum hot-face temperature. The maximum hot-face temperature shall be 100°F (55°C) below the recommended use limit of the product. Determine the thermal conductivity of the product in order of increasing temperature. After completing the conductivity determination at maximum temperature, make at least two additional determinations as the furnace temperature is lowered. Omit the introduction of compressed air when decreasing the furnace temperature.

8.4.2 For products exceeding 2300°F (1260°C) use limit: Repeat 8.1, 8.2, and 8.3 at the initial temperature and at two intermediate temperatures spaced equally between the initial temperature and maximum hot-face temperatures. The maximum hot-face temperature shall be 100°F (55°C) below the recommended use limit of the product, but not to exceed the recommended use limit of the furnace. Determine the thermal conductivity of the product in order of increasing temperature. After completing the conductivity determination at maximum

temperature, make at least three additional determinations as the furnace temperature is lowered. Omit the introduction of compressed air when decreasing the furnace temperature.

8.5 At the conclusion of the test, examine the specimens for changes that may have taken place as a result of heat treatment. If significant cracking or a linear shrinkage in the hot face of more than 1 % has taken place, test new specimens using a hot-face temperature 100°F (55°C) lower than the maximum temperature of the initial test. Then cut the specimen in half through the 4½ by 2½-in. (114 by 64-mm) dimension close to the thermocouple junctions. Measure the distance between the center line of the hot junctions to the nearest 0.01 in. (0.3 mm). If the test specimen, upon being cut in half, shows voids or cracks, or both, state this fact in the report, as the results will not be representative of the material.

## 9. Record of Test Data, Calculations, and Report

9.1 The record of test data, the calculations, and report shall be made in accordance with Test Method C 201.

## 10. Precision and Bias

10.1 Refer to Test Method C 201 for a statement of precision and bias.

## 11. Keywords

11.1 calorimeter; castable refractories; monolithic refractories; plastic refractories; refractories; thermal conductivity

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