Designation: C 201 - 93 (Reapproved 2004)

Standard Test Method for Thermal Conductivity of Refractories¹

This standard is issued under the fixed designation C 201; 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 comparative thermal conductivity of refractories under standardized conditions of testing. This test method is designed for refractories having a conductivity factor of not more than 200 Btu·in./h·ft²·°F (2818 W/m·K), for a thickness of 1 in. (25 mm).
- 1.2 Detailed ASTM test methods to be used in conjunction with this procedure in testing specific types of refractory materials are as follows: Test Method C 182, Test Method C 202, Test Method C 417, and Test Method C 767.
- 1.3 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses are provided for information only.
- 1.4 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 134 Test Methods for Size, Dimensional Measurements, and Bulk Density of Refractory Brick and Insulating Firebrick
- C 155 Classification of Insulating Firebrick
- C 182 Test Method for Thermal Conductivity of Insulating Firebrick
- C 202 Test Method for Thermal Conductivity of Refractory Brick
- C 417 Test Method for Thermal Conductivity of Unfired Monolithic Refractories
- C 767 Test Method for Thermal Conductivity of Carbon Refractories
- E 220 Method for Calibration of Thermocouples by Comparison Techniques

3. Significance and Use

- 3.1 The thermal conductivity of refractories is a property required for selecting their thermal transmission characteristics. Users select refractories to provide specified conditions of heat loss and cold face temperature, without exceeding the temperature limitation of the refractory. This test method establishes the testing for thermal conductivity of refractories using the calorimeter.
- 3.2 This procedure requires a large thermal gradient and steady state conditions. The results are based upon a mean temperature.
- 3.3 The data from this test method are suitable for specification acceptance, and design of multi-layer refractory construction.
- 3.4 The use of these data requires consideration of the actual application environment and conditions.

4. Apparatus

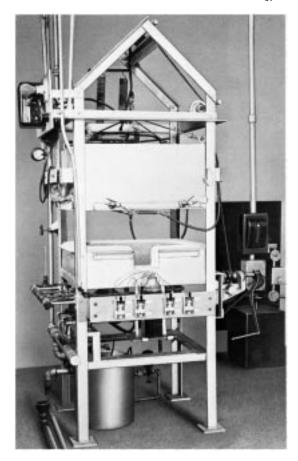
- 4.1 The apparatus shall conform in close detail with that shown in the approved drawings.³ The equipment is shown in Fig. 1 and Fig. 2, and the essential parts are as follows:
- 4.1.1 Heating Chamber—A heating chamber, shown in Fig. 3, shall be capable of being heated electrically over a temperature range from 400 to $2800^{\circ}F$ (205 to $1540^{\circ}C$) in a neutral or oxidizing atmosphere. The temperature of the heating unit shall be controlled by a mechanism capable of maintaining the temperature in the chamber constant to within $\pm 5^{\circ}F$ ($\pm 3^{\circ}C$). A silicon carbide slab $13\frac{1}{2}$ by 9 by 1 in. (342 by 228 by 25 mm), with the $13\frac{1}{2}$ by 9-in. (342 by 228 mm) faces plane and parallel, shall be placed above the sample for the purpose of providing uniform heat distribution. A layer of insulation equivalent at least to 1 in. (25 mm) of Group 20 insulating firebrick (see Classification C 155) shall be placed below the calorimeter and guard plates.
- 4.1.2 Calorimeter Assembly—A copper calorimeter assembly, of the design shown in Fig. 4, shall be used for measuring the quantity of heat flowing through the test specimen. The

¹ This test method is under the jurisdiction of ASTM Committee C08 on Refractories and is the direct responsibility of Subcommittee C08.02 on Thermal Stress Resistance.

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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 The complete set of approved drawings necessary for the construction of the apparatus and suggested operating instructions, each of which requires too much space to be included with this test method, were originally drafted by the Insulating Products Division of Babcock and Wilcox Co. ASTM has been advised that these drawings are no longer available. Subcommittee C08 .05 currently is taking this issue under advisement.



Note 1—The upper half of the heating chamber has been raised to permit introduction of the test samples.

FIG. 1 Photograph of Thermal Conductivity Apparatus

water circulation is such that adjacent passages contain incoming and outgoing streams of water. The calorimeter shall be 3 by 3 in. (76 by 76 mm) square and shall have one inlet and one outlet water connection. The inner guard surrounding the calorimeter shall be $13\frac{1}{2}$ by 9 in. (342 by 228 mm) and shall have two inlet and two outlet water connections. The outer guard shall extend 2 in. (51 mm) laterally from the inner guard and shall extend vertically to the member comprising the bottom of the heating chamber (see Fig. 3). The separation between the calorimeter and the inner guard shall be $\frac{1}{32}$ in. (0.8 mm).

4.1.3 Water-Circulating System—A water-circulating system shall be provided for supplying the calorimeter assembly with water at constant pressure and at a temperature that is not changing at a rate greater than $1^{\circ}F$ (0.5°C)/h. The inlet water pressure shall be at least the equivalent of 10 ft of hydrostatic pressure (29.9 kPa). The inlet water temperature shall at all times be within $+5^{\circ}F$ ($+3^{\circ}C$) or $-2^{\circ}F$ ($-1^{\circ}C$) of the room temperature. Fig. 5 shows the arrangement that shall be used for meeting these conditions. The regulating valves for controlling the rate of water flow through the calorimeter assembly shall be capable of maintaining a constant rate of flow within ± 1 % during the test period.

4.1.4 Instruments for Measuring Temperature of Specimen—Calibrated⁴ thermocouples shall be embedded in the test specimen for measuring the temperature. The electromo-tive force (emf) for the temperature readings shall be taken with a potentiometer having an instrument error of not more than ± 0.05 mV, and the cold junctions of the thermocouples shall be immersed in a mixture of ice and water.

4.1.5 Instrument for Measuring Temperature Rise in Calorimeter Water—A multiple differential thermocouple shall be used for measuring⁵ within an accuracy of not less than 1 % of the temperature rise of the water flowing through the calorimeter. The thermocouple shall be immersed at least 3½ in. (89 mm) in the inlet and outlet connections, and the junctions shall be not more than ¼ in. (6 mm) distant from the bottom of the calorimeter. A calibrated differential 10X copper-constantan thermocouple shall be used, and the millivolt readings shall be taken with a potentiometer having an instrument error of not more than ±0.01 mV in the range between 0 and 2 mV.

4.1.6 Instruments for Measuring Temperature Difference Between Calorimeter and Inner Guard—Calibrated differential 10X copper-constantan thermocouples shall be located in the calorimeter and inner guard for measuring⁵ the temperature differences between the calorimeter and inner guard. The temperature difference during a test shall be maintained at a value less than ± 0.05 °F (± 0.03 °C). The thermocouple junctions shall be placed in the four wells provided for that purpose, and millivolt readings shall be taken with a potentiometer having an instrument error of not more than ± 0.01 mV in the range between 0 and 2 mV.

5. Test Sample and Its Preparation

5.1 Test Sample—The test sample shall consist of three 9-in. (228-mm) straight brick and six 9 by 2½ by 2½-in. (228 by 64 by 57-mm) soap brick (Note 2) that are representative of the material being tested. These brick shall be selected for uniformity of structure and bulk density, and they shall be free of broken corners or edges. One brick shall be used as the test specimen, and one each of the other two brick shall be used as guard brick on either side of the specimen. The six soap brick shall be placed around the edges of the test specimen and guard brick to prevent side flow of heat. The test specimen and guard brick shall cover an area of approximately 18 by 13½ in. (456 by 342 mm).

Note 1—A total of nine 9-in. (228-mm) straight brick may be submitted for test, six of which would be cut to obtain the soap brick.

5.2 Preparation of Test Sample—The 9 by $4\frac{1}{2}$ -in. (228 by 114-mm) faces of the three straight brick and the 9 by $2\frac{1}{4}$ -in. (228 by 57-mm) faces of the soap brick shall be ground flat and parallel, and the thickness shall not vary more than ± 0.01 in. (± 0.3 mm). The thickness shall be not more than 3 (76 mm) nor less than 2 in. (51 mm). The sides that are to be placed in

 $^{^{\}rm 4}$ Method E 220 specifies calibration procedures for thermocouples.

⁵ The following procedures are recommended: Roeser, W. F., "Thermoelectric Thermometry," and Roeser, W. F., and Wensel, H. T., "Methods of Testing Thermocouples and Thermocouple Materials," *Temperature, Its Measurement and Control*, Reinhold Publishing Corp., New York, NY, 1941, pp. 180 and 284, respectively.

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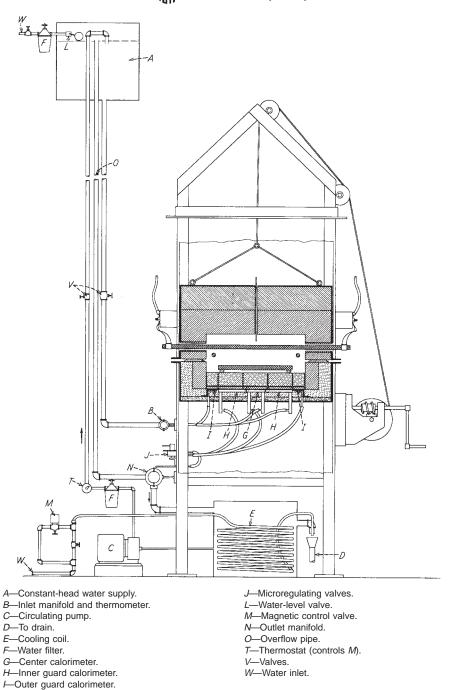


FIG. 2 Diagram Showing Essential Parts of Thermal Conductivity Apparatus

contact shall be ground flat and at right angles to the 9 by $4\frac{1}{2}$ -in. face of the brick and the 9 by $2\frac{1}{4}$ -in. face of the soap brick.

NOTE 2—Additional instructions are given in the methods of test for specific materials (see Section 7) concerning the preparation of the specimen, placing of guard brick, and the like.

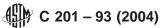
6. Bulk Density of Test Specimen

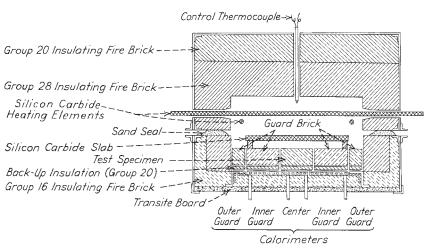
6.1 The test specimen shall be dried at 220 to 230°F (105 to 110°C) for 12 h, after which time its bulk density, in pounds

per cubic foot (or kilograms per cubic metre) shall be determined in accordance with Test Methods C 134, with the exception that the thickness measurement shall be made in accordance with those methods.

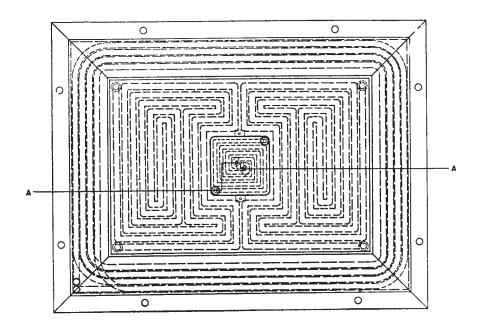
7. Procedure

7.1 Use the procedures for testing specific types of refractory materials as described in the following test methods: Test Method C 182, Test Method C 202, Test Method C 417, and Test Method C 767.





Note 1—When testing insulating firebrick, the back-up insulation is removed. FIG. 3 Diagrammatic Section Through Heating Chamber



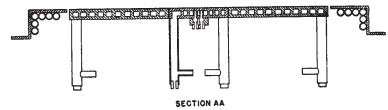
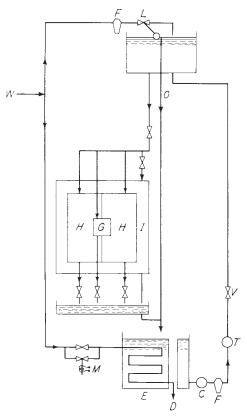


FIG. 4 Design of Calorimeter and Guard Rings

8. Record of Test Data

- 8.1 Record the following data, and record 8.1.3 to 8.1.7 for each 2-h test period (steady state of heat flow):
 - 8.1.1 Linear dimensions of test specimen,

- 8.1.2 Distance between thermocouple junctions located in the test specimen,
- 8.1.3 Three sets of temperature readings as measured by the thermocouples in the test specimen,



C—Circulating pump.

D—To drain.

E-Cooling coil.

F—Water filter.

G—Center calorimeter.

H-Inner guard calorimeter.

—Outer guard calorimeter.

L—Water-level valve.

M—Magnetic control valve.

O—Overflow pipe.

T—Thermostat (controls M).

V—Valves.

W-Water inlet.

FIG. 5 Water-Circulating System with Automatic Temperature
Control

- 8.1.4 Mean temperature between each pair of thermocouples in the test specimen as calculated from the temperatures recorded in 8.1.3,
- 8.1.5 Average rise in temperature of the water flowing through the calorimeter,
- 8.1.6 Average rate of water flow through the calorimeter,
- 8.1.7 Rate of heat flow through the test specimen per unit area.

9. Calculation

9.1 Calculate the thermal conductivity as follows:

$$[k = qL/[A(t_1 - t_2)]$$

where:

 $k = \text{thermal conductivity, Btu·in./h·ft}^{2.\circ}F \text{ (or W/m·K)},$

q = Btu/h flowing into the calorimeter (temperature rise, °F (K) of the water flowing through the calorimeter times the weight of flowing water, lb/h (or W)),

L = thickness (distance between hot junctions at which t_1 and t_2 are measured), in. (or m),

 t_1 = higher of two temperatures measured in the test specimen, °F (or K),

 t_2 = lower of two temperatures measured in the test specimen, $^{\circ}$ F (or K), and

 $A = \text{area of center calorimeter, ft }^2(\text{or m}^2).$

10. Report

10.1 The report shall include the following:

10.1.1 Brand name or other identifying information,

10.1.2 Bulk density of the dried test specimen (see Section 6).

10.1.3 General description of the test specimen before and after test with respect to possible structural changes caused by exposing the test specimen to the heating chamber temperatures.

10.1.4 The thermal conductivity data as calculated in accordance with Section 8 at the mean temperatures recorded during a 2-h holding period with a steady state of heat flow, and reported at the mean of the two temperatures used in the calculation.

10.1.5 A curve showing the actual thermal conductivity values obtained *versus* mean temperatures, and

10.1.6 When requested, the data recorded for Section 8 shall be included in the report.

11. Precision and Bias

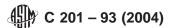
- 11.1 Interlaboratory Test Data:
- 11.1.1 Results of round-robin tests between four laboratories on three varieties of refractory material ranging in *k*-value from 2 to 165 were evaluated.
- 11.1.2 Polynomial regressions were established by computer, and the residual sum of squares and degree of freedom were summated for the within-laboratory variances. Between-laboratory variances were calculated from the regression curves of the four laboratories at four mean temperatures (500°F, 1000°F, 1500°F, and 2000°F).
- 11.1.3 The components of variance for the thermal conductivity, k, (Btu·in./h·ft².°F) expressed as coefficients of variations were:

Within laboratories,
$$V_{\rm w}$$
= 3.4 %
Between laboratories, $V_{\rm b}$ = 9.0 %

11.2 *Precision*—For the components of variation given in 11.1, two averages of test values will be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed as follows: (t = 1.96)

| No. of Samples in Each | Critical differences, % of g | grand average <i>k</i> (Btu⋅in./ |
|------------------------|------------------------------|----------------------------------|
| Average | h⋅ft²-°F) | |
| | within-lab | between-lab |
| (n) | precision | precision, % |
| 1 | 9.4 % | 26.6 |
| 2 | 6.6 % | 25.8 |
| 3 | 5.5 % | 25.5 |
| E | 4 2 0/ | 25.0 |

11.3 Supplemental Interlaboratory Data— One refractory material was tested by four laboratories in which the thermocouples were permanently affixed by one laboratory. Polynomial regression equations on these data revealed the following components of variance:



Within laboratories, $V_{\rm w}$ = 3.1 % Between laboratories, $V_{\rm b}$ = 2.3 %

12. Keywords

12.1 calorimeter; refractories; thermal conductivity

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