

Standard Practice for Firing Refractory Concrete Specimens¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice covers the firing of specimens made from refractory concretes (castable refractories) in accordance with Practice C 862 for cast specimens. The procedure is also recommended for heating rates to be used for high-temperature test methods such as Methods C 16, C 583, etc., when these methods are used to test refractory concretes.

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:

- C 16 Method of Load Testing Refractory Brick at High Temperatures²
- C 20 Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water²
- C 113 Test Method for Reheat Change of Refractory Brick²
- C 133 Test Methods for Cold Crushing Strength and Modulus of Rupture of Refractories²
- C 210 Test Method for Reheat Change of Insulating Firebrick²
- C 288 Test Method for Disintegration of Refractories in an Atmosphere of Carbon Monoxide²
- C 401 Classification of Alumina and Alumina-Silicate Castable Refractories²
- C 546 Method of Load Testing Refractory Brick at High Temperatures, Long-Time³

- C 583 Test Method for Modulus of Rupture of Refractory Materials at Elevated Temperatures²
- C 704 Test Method for Abrasion Resistance of Refractory Materials at Room Temperature²
- C 832 Test Method for Measuring the Thermal Expansion and Creep of Refractories Under Load²
- C 862 Practice for Preparing Refractory Concrete Specimens by Casting²
- E 220 Method for Calibration of Thermocouples by Comparison Techniques⁴

3. Significance and Use

3.1 This practice is used to standardize the firing conditions of refractory concrete specimens prepared in accordance with Practice C 862. The standards are set down to minimize laboratory-to-laboratory variation and do not attempt to duplicate any particular field applications.

4. Apparatus

4.1 *Kiln*, equipped with instruments capable of controlling the heating rate of the kiln at 100 to 700° F (55 to 380° C)/h (see 6.5) and holding the soak temperature to $\pm 10^{\circ}$ F (5.5°C) of the nominal soak temperature. For temperatures up to 2500°F (1370°C) an electrically heated kiln is preferred, but gas- or oil-fired kilns can be used for all temperatures, provided the heating rates specified can be maintained, the flame of the burners does not impinge directly on any specimen, and the furnace atmosphere contains a minimum of 0.5 % oxygen with 0 % combustibles.

4.2 *Balances*—For 9 by $4\frac{1}{2}$ by 2 or 3-in. (228 by 114 by 51 or 76-mm) samples, a balance with a capacity of 15 lb (6.8 kg) and a sensitivity of 0.01 lb (4.5 g) is recommended; for smaller specimens (for example, 6 by 1 by 1-in. (152 by 25 by 25-mm) bars), a 2-kg balance with a sensitivity of 0.1 g is recommended.

4.3 *Caliper or Steel Rule*, to measure the dimensions of the specimens. For large specimens, a 12-in. (305-mm) steel rule with 0.01-in. (0.3-mm) divisions is recommended. For smaller

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approved in 1977. Last previous edition approved in 1987 as C 865 – 87. ² Annual Book of ASTM Standards, Vol 15.01.

³ Discontinued. See 1985 Annual Book of ASTM Standards, Vol 15.01.

⁴ Discontinued. See 1994 Annual Book of ASTM Standards, Vol 14.03.

specimens, a 250-mm caliper with 0.1-mm divisions is recommended. Other measuring devices of the same or better precision may also be used.

5. Preparation of Samples

5.1 Samples are prepared by casting in accordance with Methods C 862. If the sample size of the cast specimen is the same as that specified for the test, it can be used directly. However, cutting samples of the required size from larger cast blocks will often be necessary (Note 1). In this case, it is recommended that the samples be cut with a diamond saw. After cutting, the samples should be dried at 230°F (110°C) for a minimum of 18 h. All cut samples should have sharp edges and corners and should not show pull-out of grains on the cut surfaces. For some low-strength castables, drying prior to cutting may be needed to increase their strength and resistance to pull-outs.

NOTE 1—Specimens cut from the interior of large cement bonded castables shapes may be stronger than specimens cut from small cast shapes because the interior of large cast shapes are exposed to high pressure steam during dryout which causes more complete hydration of the cement.

5.2 Some types of castables, especially those containing aggregates of a relatively low hardness (such as lightweight castables), may be cut on a dry saw. This procedure is acceptable provided that specimens with sharp corners and edges, which show no signs of grain pull-out at the cut surfaces, are obtained.

6. Procedure

6.1 Label all specimens with ceramic ink or ceramic crayons.

6.2 Measure all dimensions of the specimens using a steel rule or caliper. Make the measurement at the center of each side and to the nearest 0.01 in. (0.3 mm) or 0.1 mm, depending on the instrument used and the sample size. When using an indentation mark in accordance with 7.3 in Practice C 862, remeasure the distance between the reference marks to determine the total linear change from the as-cast state (Note 2).

6.3 Measure the weight of the specimens to the nearest 0.01 lb (4.5 g) for samples weighing approximately 4 lb (1.8 kg) or more, or to the nearest 0.0002 lb (0.1 g) for samples weighing less than 4 lb.

NOTE 2—Linear change measurements based on the initial cast specimen cannot be determined when specimens are cut from larger sections.

6.4 Place the specimens in the kiln so that each specimen will rest on the surface formed by its longest and smallest dimension (that is, the 9 by $2\frac{1}{2}$ -in. (230 by 65-mm) surface for 9-in. straight, or the 6 by 1-in. (152 by 25-mm) surface for 6 by 1 by 1 in. samples). Support each specimen on a flat supporting brick, which does not soften or shrink at the intended soak temperature. Place between the test specimens and the supporting brick a layer of suitable refractory material that is nonreactive under the test conditions and passes a No. 16 (1.18-mm) sieve and is retained on a No. 40 (425-µm) sieve.

NOTE 3—Specimens tested for refractoriness under load or creep must be placed in a furnace as indicated in Methods C 16 and C 546, respectively.

6.5 The following maximum heating rates at temperatures below 1500°F (815°C) are recommended:

Cross Section of Specimen		Maximum Heating Rate	
in.	mm	°F/h	°C/h
4 1 / 2 by 3	114 by 76	100	55
4 1 / 2 by 2 1 / 2	114 by 65	100	55
2 by 2	228 by 64	300	170
1 by 1	25 by 25	700	380

At temperatures over 1500°F a heating rate up to 700°F (390°C)/h is permissible. Generally, a slower heating rate does not affect the properties of the specimens adversely. Thus, when smaller and larger specimens are fired together, the heating rate applicable to the larger specimens should be used.

NOTE 4—Different heating rates are specified for various samples to ensure that the difference in temperature between the center and the surface of the specimen does not exceed 45°F (25° C). Experimental evidence indicates that cracking may occur when a castable sample is heated too rapidly. The safe heat-up rate increases with decreasing sample size.

6.6 Hold the specimens at the specified temperature for 5 h. During the holding period a furnace atmosphere containing a minimum of 0.5 % oxygen with 0 % combustibles should be maintained. For special high-temperature tests a longer or shorter holding time may be required, that is, $1\frac{1}{2}$ h for the refractoriness-under-load test (Method C 16), or 12 h for the hot modulus of rupture test (Test Method C 583).

6.7 Measure the temperature within the kiln by means of a platinum-rhodium/platinum type thermocouple, calibrated in accordance with Method E 220, and a potentiometer, preferably of the recording type for temperatures up to 2700° F (1480°C). For temperatures in the 2700 to 3100° F (1480 to 1705°C) range, either a platinum-rhodium/platinum or a platinum 6 % rhodium-platinum 30 % rhodium (Type B) thermocouple can be used. For temperatures over 3100° F, a Type B thermocouple must be used. The hot junction of the thermocouple should be placed in close proximity to the specimens. An alternative method is the use of a calibrated optical pyrometer where applicable. Make temperature readings at intervals of 15 min or less when the temperature is not recorded and controlled automatically. The use of cone plaques is advised as a guide to temperature uniformity within the kiln.

6.8 After completion of the firing, cool the specimens in a closed kiln at a cooling rate not exceeding $500^{\circ}F$ ($280^{\circ}C$)/h.

6.9 Within 2 h after the fired specimens reach room temperature, remeasure their dimensions and weight in accordance with 6.2 and 6.3 if density change or dimensional change, or both, data are required. Adhering loose setting materials and small blisters must be removed prior to such measurement.

6.10 When room temperature strength measurements (Test Methods C 133) are to be made after firing, store the fired specimens in a desiccator, or alternatively in a drying oven held at 230°F (110°C), to prevent moisture pick up from the air prior to testing. Strength measurements should be made within 2 h after removal from the desiccator or drying oven.

NOTE 5—Refractory concrete specimens exposed to room air after firing have shown strength changes as exposure time in room air increases when tested in accordance with Test Methods C 133 or other strength test methods.



7. Use of Recommended Firing Practices

7.1 Refractory concrete specimens required for the

methods listed in 7.1.1 through 7.1.6 should be fired in accordance with this practice. If the specimens are tested at temperature, the firing procedures described in this practice should be used to heat the specimens to the required test temperature and to cool them after the test is completed (the latter is necessary only if subsequent measurements are made at room temperature). The most frequently used tests are:

7.1.1 *Methods C 16 and C 546*—In these methods, the heat-up schedule should follow the procedure described in this practice.

7.1.2 *Test Methods C 20*—Holding temperature(s) as required for the intended service temperature.

7.1.3 *Test Methods C 113 and C 210*—The procedures described in these practices include all measurements needed to calculate the linear and, optionally, the volume change of castables from the dried state only. The total linear change from the as-cast specimens are calculated by either using the distances between the reference marks measured as-cast and

after drying or firing or both, or by using the calipered longitudinal inside mold dimension as the initial specimen size or zero point. Holding temperatures appropriate for the ASTM classification of castables (Classification C 401) or for the intended service temperature may be selected.

7.1.4 Test Methods C 133 and C 583—Holding temperature(s) for determining cold crushing strength and modulus of rupture are as required for the intended purpose (service temperatures) or as specified by the consumer, usually 1500 to 2000° F (815 to 1095° C).

7.1.5 *Test Method C* 288—Carbon monoxide disintegration is determined at holding temperatures of 1000 and 2000°F (540 and 1095°C).

7.1.6 *Test Method C 704*—Holding temperature(s) for specimens to be tested for abrasion resistance at room temperature are as required for the intended purpose or as specified by the purchaser.

8. Keywords

8.1 castables; elevated temperatures; linear change; refractory concrete

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