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Standard Test Method for Measuring the Resistance of Ceramic Tile to Freeze-Thaw Cycling¹

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1. Scope

1.1 This test method describes the procedures and equipment required to test either glazed or unglazed ceramic tile for resistance to repeated cycles of freezing and thawing. Ceramic tile of any size or shape may be tested by this test method.

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:*²

C242 [Terminology of Ceramic Whitewares and Related Products](#)³

E220 [Test Method for Calibration of Thermocouples By Comparison Techniques](#)

3. Summary of Test Method

3.1 A designated test-load of tile specimens is saturated with water, and placed in a freezer with thermocouples (thermometer) inserted. Freezing is followed by a thawing cycle with the specimens immersed in water. The number of tile damaged after freezing and thawing for 5, 10, ~~and 15~~, 20, 25 . . . 150 cycles is determined by visual examination.

4. Significance and Use

4.1 The test for resistance to freezing and thawing functions as a guide to the selection of ceramic tile suitable for outdoor service in geographic areas subjected to freezing. It can serve as a test method to verify compliance with specifications for ceramic tile, and provides a control test for determining the uniformity of tile being manufactured for exterior installations.

5. Apparatus (See Fig. 1)

5.1 The freezing chamber for this test method may be of any type provided it has the capacity to cool the center of the test load to 0°F (-18°C)(-18°C) within a period of 6 to 8 h. By adjusting the mass of the test load, freezers with various freezing rates may be used in this test as long as the 6- to 8-h period to reach 0°F (-18°C)(-18°C) is maintained.

5.2 Two freezer thermometers of the type that use a thermocouple are required. They shall be calibrated to 0°F (-18°C) against a thermometer of known accuracy (see 2.1).

5.3 ~~Vacuum Chamber, capable of withstanding a vacuum of 760 mm Hg (102 MPa).~~ Pan, in which the specimens may be boiled.

5.4 ~~Vacuum Pump, that can go down to 20 mm Hg (2.6 MPa) pressure.~~ Potable Water.

5.5 Drill Press.

5.6 Carbide Tipped Bit, slightly larger in diameter than the thermocouple, or other means of drilling the tile.

5.7 Ultraviolet Light Source and Fluorescent Dye, (unglazed tile) or black ink (glazed tile).

¹ This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.06 on Ceramic Tile.

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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

³ *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website, Vol 15.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.03.

6. Test Samples

6.1 Select ten samples at random from the lot to be tested.

7. Procedure

7.1 Calibration of the Freezer Capacity:

7.1.1 Calibrate the two freezer thermometers.

7.1.2 Select a group of tiles having the same approximate absorption, size, and total mass as the samples to be used in the final test for use as a test load.

7.1.3 Drill a hole through the center of the tile that will be in the middle of the load as it is placed in the freezer in order to accommodate the thermocouple. The fit of the thermocouple bead in the tile should be as snug as possible. The wire should be trailed out either between the ribs or in a slot cut in the back of one of the tiles. It is important that the tile, when stacked, are not held apart by the thermocouple or wire.

7.1.4 Remove the thermocouple from the prepared tile and subject them and the remainder of the tile to be used as the test load to the following:

7.2 Test Load Preparation:

~~7.2.1 Place as many tile at a time as possible in a vacuum container. Evacuate to a pressure of 20 mm Hg for 20 min, then add potable water at room temperature to cover the tile completely while still under vacuum. Hold under vacuum for 40 min. Make sure all tile are submerged in water throughout the 40-min soak. After this treatment, release the vacuum and store the treated tile immersed in water until ready for~~

7.2.1 Record the dry weight of each specimen. Place the specimens in a pan of potable water and soak for 24 h. Use setter pins or some similar device to separate the specimens from the bottom and sides of the pan and from each other. Record the 24 soak weights, return the specimens to the water and boil for 5 h, taking care that the specimens are covered with water at all times. After the 5-h boil, allow the specimens to soak for at least an additional 24 h, record the 5-h boil weights, and continue to immerse the treated tiles in water until ready for 7.2.3 :

~~7.2.2 Mount the thermometers to the outside of freezing chamber which has a no-load capability of -10 to -20°F (-23 to -29°C). of -10 to -20°F (-23 to -29°C). Place one thermocouple in the freezer so that it is suspended in the atmosphere of the freezer, not touching the sides or bottom. The other thermocouple will be used in the next step.~~

7.2.3 Remove the tile drilled to hold the thermocouple from the water, and wipe it lightly with a damp cloth. Insert the thermocouple into the tile as discussed previously. Add enough tiles to the top and bottom of the stack so that the thermocouple is in the center of a stack of tile of shape and weight approximating the test load.

7.2.4 Check the inside temperature of the freezer and record (see Appendix X1).

7.2.5 Remove the remaining tiles for the load from the water and wipe them with a damp cloth.

7.2.6 Place the entire load of tile in the freezer, preferably in a specially made basket or container. If the tile are to be stacked in the freezer, make sure the stack is straight, particularly around the thermocouple, and that the wire is in its groove.

7.2.7 Take a reading, both of the internal tile temperature, and inside the freezer.

7.2.8 Continue to record both temperatures on an hourly basis until the temperature inside the tile reaches 0°F (-18°C). (-18°C).

7.2.9 Prepare a table similar to Appendix X1 that shows temperature in relation to time.

7.2.10 The total time will be affected by the size of the freezer, the size of the load, and the recovery rate of the freezer. The load is the only one of these three that can be varied easily, so in order to maintain the specified rate of freezing, adjust the load so that the total time required to reach 0°F (-18°C) (-18°C) on the thermocouple in the load falls between 6 and 8 h.

7.3 Frost Resistance Load Preparation:

7.3.1 Select a set of tile similar in size and mass to the test load.

7.3.2 Number each tile with an indelible marker.

7.3.3 Remove tile from water, wipe with damp cloth, and place in the freezing chamber, stacked in a manner similar to the test load described in 7.2.6.

7.4 Freezing and Thawing:

7.4.1 When the tile have reached 0°F (-18°C) (-18°C), remove them from the freezer and place in water that is at a temperature from 50 to 60°F (10 to 16°C) and is of a sufficient quantity that the temperature remains within this range (about 4 lb (kg/kg) of water per pound of tile). Place the tile into the water one piece at a time and leave submerged for a minimum of 30 min. Keep the tile separated at least ½ in. (13 mm) to permit water circulation.

7.4.2 Remove tile from the water after thawing, wipe, observe in oblique light for surface flaws, then reimmerse in water until ready for next freezing cycle. Repeat the freezing, thawing, observation procedure for a total of five cycles. After five cycles, wipe tile, weigh, and augment detection of surface flaws by rubbing surface with a washable black ink (glazed tile) or a fluorescent solution (unglazed tile). It is necessary to observe the unglazed tile illuminated with ultraviolet light. Record observations. Return tile to water after observation. When running both glazed and unglazed tile simultaneously, keep the two types in separate containers of water.

7.4.3 Upon completion of observations after the fifth cycle, remove any tile showing crazing, chipping (nonmechanical), spalling or cracking either of the body or glaze from the lot and subject the balance of the tile to an additional five cycles of freezing