

Designation: C 650 - 97

Standard Test Method for Resistance of Ceramic Tile to Chemical Substances¹

This standard is issued under the fixed designation C 650; 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 a procedure for determining whether, and to what degree, ceramic tiles are affected by prolonged exposure to chemical substances that are commonly used in the household or for cleaning purposes as well as other more severe conditions.
- 1.2 The units used for concentration in this standard are v/v which refers to the volume of reagent/1 L of solution and g/L which refers to the weight of reagent, in g, to be dissolved in 1 L of water.
- 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. Summary of Test Method

2.1 This test method calls for one or more tests, each consisting of exposing flat pieces of ceramic tile to the action of a specific chemical substance for a definite period of time at a prescribed temperature. After exposure, the surfaces of the tile are rinsed with water and inspected for effect.

3. Significance and Use

- 3.1 This test method is intended for testing ceramic tile that are to be used for food counters, lavatories, and similar residential, medical, and commercial installations, where they may come in contact with food, chemical, and waste substances and for tile in areas where they may be exposed to contact with strong cleaning agents.
- 3.2 The specification of 10 % hydrochloric acid solution and 10 % potassium hydroxide solution as normal testing fluids, and the length of exposure time and temperature are based on experimental laboratory work and interlaboratory tests, where it was indicated that resistance to these two substances probably assures resistance also to any other acidic or alkaline substance coming in contact with tile, except hydrofluoric acid.

4. Apparatus

- 4.1 *Test Tubes*, plain-end flint glass test tubes with a diameter of 20 mm and a length of 150 mm.
- 4.2 *Pipets*, glass with a volume of at least 50 mL with gradations in 1-mL increments.
- 4.3 *Oven*, capable of maintaining a constant temperature of 110 ± 5 °C.
 - 4.4 *Pencil*, HB hardness grade.
- 4.5 *Cloths*, a supply of lintless, absorbent cloths or paper towels.
- 4.6 Distilled or Deionized Water, for preparation of the solutions.
- 4.7 Light Source, standard, that supplies a 300 lux of illumination.

5. Reagents

- 5.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.² Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 5.2 The required dilutions for the preparation of each test solution is found in Table 1 of this test method.
 - 5.2.1 Common Household and Cleaning Chemicals:
 - 5.2.1.1 Acetic acid, 3 % (v/v).
 - 5.2.1.2 Acetic acid, 10 % (v/v).

^{3.3} The validity of using color difference as means of determining the degree to which tile surfaces are affected by acids and alkalies has been established by interlaboratory test and was found to be more reliable than the determination of loss of gloss, particularly for unglazed tile and mat or semimat glazed surfaces. Lack of coordination between instrumental value and visual effect was noted only in the case of minute surface changes, detectable only by oblique viewing.

¹ This test method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.06 on Ceramic Tile.

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² Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeial and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Preparation of Test Solutions

Test Solution	Reagent Source	Amount of Reagent Required for Test Solution	Distilled Water Required for Test Solution
	Common Household and Cleaning Chemicals		
Acetic acid, 3 % (v/v)	99.7 % glacial acetic acid	30 mL	1000 mL
Acetic acid, 10 % (v/v)	99.7 % glacial acetic acid	1000 mL	1000 mL
Ammonium chloride, 100 g/L	99.5 % ammonium chloride	100 g	1000 mL
Citric acid solution, 30 g/L	99.5 % citric acid	30 g	1000 mL
Citric acid solution, 100 g/L	99.5 % citric acid	100 g	1000 mL
_actic acid, 5 % (v/v)	85 % lactic acid	59 mL	991 mL
Phosphoric acid, 3 % (v/v)	85 % phosphoric acid	35 mL	995 mL
Phosphoric acid, 10 % (v/v)	85 % phosphoric acid	118 mL	982 mL
Sulfamic acid, 30 g/L	99 % sulfamic acid	30 g	1000 mL
Sulfamic acid, 100 g/L	99 % sulfamic acid	100 g	1000 mL
-	Swimming pool chemicals	_	
Sodium hypochlorite solution, 20 mg/L	technical grade sodium hypochlorite with 13 % active clorine	20 mg	1000 mL
	Acids and Bases		
Hydrochloric acid solution, 3 % (v/v)	38 % hydrochloric acid	79 mL	951 mL
Hydrochloric acid solution, 18 % (v/v)	38 % hydrochloric acid	474 mL	706 mL
Potassium hydroxide, 100 g/L	90 % potassium hydroxide	100 g	1000 mL
Potassium hydroxide, 30 g/L	90 % potassium hydroxide	30 g	1000 mL

- 5.2.1.3 Ammonium chloride, 100 g/L.
- 5.2.1.4 Citric acid solution, 30 g/L.
- 5.2.1.5 Citric acid solution, 100 g/L.
- 5.2.1.6 Lactic acid, 5 % (v/v).
- 5.2.1.7 Phosphoric acid, 3% (v/v).
- 5.2.1.8 Phosphoric acid, 10 % (v/v).
- 5.2.1.9 Sulfamic acid, 30 g/L.
- 5.2.1.10 Sulfamic acid, 100 g/L.
- 5.3 Swimming Pool Chemicals—Sodium hypochlorite solution, 20 mg/L, prepared from technical grade sodium hypochlorite with about 13 % of active chloride.
 - 5.4 Acids and Bases:
 - 5.4.1 Hydrochloric acid solution, 3 % (v/v).
 - 5.4.2 Hydrochloric acid solution, 18 % (v/v).
 - 5.4.3 Potassium hydroxide, 100 g/L.
 - 5.4.4 Potassium hydroxide, 30 g/L.
- 5.5 This test method may be used for exposing tile to any chemical substance deemed appropriate for the expected service conditions and may specify any reasonable combination of time and temperature for the exposure period. Exposure substances, time, and temperatures should be selected, either to simulate service conditions or accelerate deterioration that is possible under expected service conditions, or bear some other meaningful relation to expected service conditions.
- 5.6 Hydrofluoric acid should not be used as a test substance because most ceramics are silicates and are quite readily attacked by this acid.

6. Samples

- 6.1 Number of Test Specimens—One defect-free test specimen that is representative of the entire surface for each test solution is to be used. Test specimens shall be representative of the sample, and where tiles have different colors of decorative effects, take care to include all distinctive parts and more test specimens as necessary to incorporate all surface features.
- 6.2 *Size of Specimens*—The original tile for testing should be cut to 50 by 50-mm squares for testing with each cut piece labeled according to the testing solution to be applied.

- 6.3 The sample surfaces should be cleaned thoroughly with a suitable solvent, such as acetone, and completely dried before testing.
- 6.4 The test specimens can be glazed or unglazed tile. If the tile are glazed, then apply the testing solutions to the glazed surface.

7. Procedure for Test Solution Application

- 7.1 Application of the Test Solutions:
- 7.1.1 Dry the test specimens thoroughly at 110 ± 5 °C before testing, and then cool the tile to room temperature.
- 7.1.2 The labeled test specimens are laid out on a table in a well-ventilated area.
- Note 1—**Precaution:** Since this test method involves the use of concentrated acids and bases, take proper precautions for the handling of these chemicals, such as protective clothing and fume hoods.
 - 7.1.3 Transfer 20 mL of the testing solution to a test tube.
- 7.1.4 Place the surface of the tile to be tested face down on the open end of the test tube.
- 7.1.5 While firmly holding the test tube and tile assembly together, invert the assembly so that the back of the tile is facing the table and the closed end of the test tube is facing up.
- 7.1.6 Carefully place the assembly on the table and leave undisturbed for 24 h.
 - 7.2 Removal of the Test Solutions:
- 7.2.1 After 24 h of contact between the test specimens and the test solution has expired, again invert the assembly so that the test solution is contained in the test tube, and remove the test tube from the specimen and dispose of the testing solution properly.
- 7.2.2 Thoroughly rinse the specimens under running water for a period of 10 min to remove any residual testing solution. If needed, clean the surface with a soft bristle brush to remove test solutions
- 7.2.3 Dry the specimens thoroughly at 110 ± 5 °C, and cool to room temperature before evaluation.





8. Evaluation of Results

- 8.1 Visual Evaluation—Examine the surface at a standard distance of 25 cm and a standard illumination of approximately 300 lux. Rotate the sample to examine it for appearance color and texture from multiple angles. Examine for differences in appearance between the treated and untreated area. If the sample is attacked visibly by the test solution, then the results of that testing solution will be recorded as "affected." If the sample passes the visual test, then use the pencil test in the following section to verify the results.
- 8.2 Pencil Test—Draw several lines across the test specimen with the HB grade pencil, making sure to include treated and untreated areas. Attempt to remove the pencil lines with a damp cloth. If the pencil lines are removed from the treated surface, then the results for that test solution will be recorded as "not affected." If the pencil lines are not removed from the treated surface, the results for that test solution will be recorded as "affected." If the pencil mark is not removed from the untreated area, as well as the treated area, then the pencil test may not be applicable in this case.

9. Report

9.1 Report the following information:

- 9.1.1 Make out a report or test certificate with each of the testing solutions used (a sample procedure is in Table 2).
- 9.1.2 Identify the tile being tested by type, size, and state whether glazed or unglazed.
- 9.1.3 State the test conditions, that is, type of testing solutions used, temperature maintained during the test, and length of exposure.
- 9.1.4 Report the results of the visual inspection for each tile by the simple statements "affected" or "not affected," as the case may be.
- 9.1.5 Report the results of the pencil test, if necessary, for each tile by the simple statements "affected" or "not affected," as the case may be.
 - 9.1.6 State whether a repeat test was necessary.

10. Precision and Bias

10.1 *Qualitative Procedure*—This is a qualitative or passfail test; hence, precision and bias are not applicable.

11. Keywords

11.1 acid resistance; alkali resistance; ceramic tile; chemical resistance; glaze durability



TABLE 2 90 % Sample Test Certificate

Sample Name:				
Sample Size:				
Sample Color:				
Sample Surface Condition:				
Test Solution	Visual Test Pencil Test (Affected/Not (Affected/Not Affected) Affected)	Number of Samples		
Common Household and				
Cleaning Chemicals				
Acetic acid, 3 % (v/v)				
Acetic acid, 10 % (v/v)				
Ammonium chloride, 100 g/L				
Citric acid solution, 30 g/L				
Citric acid solution, 100 g/L				
Lactic acid, 5 % (v/v)				
Phosphoric acid, 3 % (v/v)				
Phosphoric acid, 10 % (v/v)				
Sulfamic acid, 3 % (v/v)				
Sulfamic acid, 10 % (v/v)				
Swimming Pool Chemicals				
Sodium hypochlorite solution,				
20 mg/L,				
Acids and Bases				
Hydrochloric acid solution, 3 %				
(v/v)				
Hydrochloric acid solution, 18 %				
(v/v)				
Potassium hydroxide, 100 g/L				
Potassium hydroxide, 30 g/L				

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