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Standard Test Method for Acid Resistance of Porcelain Enamels (Citric Acid Spot Test)¹

This standard is issued under the fixed designation C 282; 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.

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

1. Scope

- 1.1 This test method covers a procedure for evaluating porcelain enamels in their resistance to citric acid exposure at room temperature. No attempt is made to categorize porcelain enamels as to their acid-resistance or non acid-resistance properties, since the requirements in the several branches of the industry differ.
- 1.2 The test method is applicable for ware of various shapes providing they contain a substantially flat area approximately 50 mm in diameter.
- 1.3 The test method is not applicable to finishes on chemical and hospital ware, which may come in contact with strong mineral acids, nor to cooking utensils, which may come in prolonged contact with hot acid solutions.
- 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. Summary of Test Method

2.1 The test method consists of a 15 min exposure of the test surface to a small pool of 10 % citric acid, and an evaluation of the effect in terms of the change in appearance and the "relative cleanability" of the surface resulting from the treatment.

3. Significance and Use

- 3.1 This test method is intended specifically for testing the porcelain enamel finish on stoves, refrigerators, table tops, sinks and other sanitary ware, laundry appliances, architectural units, etc., where the surface may come in contact with food acids at room temperature.
- 3.2 Citric acid has been chosen as the test medium because it is one of the most common of the food acids and will generally provide a measurable result in its action on porcelain enamel.

4. Apparatus

- 4.1 Dropper Bottle, or medicine dropper,
- 4.2 Watch Glass, 25 mm in diameter with fire polished edge,
- 4.3 Towel, soft cotton, and
- 4.4 Drafting Pencil, conventional graphite, degree 3B.

5. Reagents and Materials

- 5.1 Citric Acid Solution—Dissolve 10 g of anhydrous citric acid crystals ($H_3C_6H_5O_7$) in 100 mL of water. Solution shall be prepared not more than 48 h prior to use.
- 5.2 Cleaner Solution—Dissolve 10 g of trisodium phosphate (Na₃PO₄) in 1 L of tap water.

6. Sampling

6.1 The test specimens may be articles of commerce, pieces cut from articles of commerce, or laboratory specimens prepared especially for this test.

Note 1—Processing variables in the application and drying and firing operations materially affect the degree of attack by the acid on the surface of porcelain enamels. Sample specimens used for classification of acid resistance must be processed under identical conditions to the commercial ware they represent.

7. Procedure

7.1 Thoroughly wash area to be tested using a soft cotton towel moistened with a warm, 1% solution of trisodium phosphate. Rinse in warm, running tap water, and dry with a soft towel by blotting. Store the specimen at a temperature of 26 ± 1 °C (79 ± 2 °F) for a time sufficient to bring it within this range prior to and during the test.

NOTE 2—If, when rinsing, the water gathers in drops on the surface, repeat washing treatment until water spreads evenly.

7.2 On articles of commerce, select areas that are horizontal or nearly horizontal in service. Place the specimen in a position such that a flat area at least 38.1 mm in diameter is horizontal. With the specimen and the citric acid solution at $26 \pm 1^{\circ} \text{C}$ (79 $\pm 2^{\circ} \text{F}$), place several drops of the solution on the test area to form a pool, and immediately cover with a clean watch glass in the inverted position. Use a quantity of solution that is just sufficient to fill the inverted watch glass except for a small air bubble (three to six drops are usually required, depending upon the dropper and the curvature of the watch glass). After 15 min

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This test method is based on the Test for Acid Resistance of Porcelain Enamels (Citric Acid Spot Test) *Bulletin T-21*, of the Porcelain Enamel Institute.



of treatment, remove the watch glass and immediately rinse the spot of solution from the surface. Dry the specimen with a dry, clean, soft cotton towel by blotting (not rubbing).

NOTE 3—The test surface must be thoroughly dry before grading. The presence of a slight film of water on the surface may change the rating of specimens near the borderline between classes.

8. Grading

8.1 Grade the test specimen within 2 h after exposure to the test solution using the procedure outlined in the diagram, Fig. 1. Rate each specimen with the appropriate classification grade, AA, A, B, C or D, as explained in Table 1. The criteria referred to in Fig. 1 and Table 1 are described as follows:

8.1.1 Visual Examination—View the specimen at various angles with respect to the light source and eye, in order to detect whether the "treated area" differs in any respect from the "protected area." Use partially diffused daylight, supplemented if necessary by artificial light, the total intensity being approximately that available within a few feet of an outside window, but do not examine in direct sunlight. During observation, hold the specimen no nearer the light source (such as a window) than the minimum diameter of the source.

Note 4—The term "treated area" refers to that portion of the specimen which has been subjected to acid solution confined by the watch glass during treatment. The term "protected (untreated) area" refers to the area adjacent to the treated area.

Note 5—Some colored porcelain enamels exhibit a change in color as a result of the acid treatment. Since the test is designed primarily to evaluate durability, color changes as revealed by this test should be ignored except where otherwise specified.

8.1.2 Dry-Rubbing Test—Using the flat point of a degree 3B, conventional graphite drafting pencil held in a normal writing position and applied with firm pressure, draw two or more approximately parallel lines extending across the treated area. Starting with gentle pressure and then applying gradually increasing pressure, rub repeatedly across the lines with a dry, clean, soft cotton towel as illustrated in Fig. 2. If marks are completely removed from the treated area, the specimen shall be rated as Class AA. Otherwise, continue as directed.

Note 6—The pencil point should be prepared by holding a sharpened pencil in a normal writing position and rubbing on abrasive paper (1/0 Emery polishing or 400-grit silicon carbide or aluminum oxide abrasive

TABLE 1 Requirements for Respective Classes of Acid Resistance

Classification	Requirements
Class AA	No visible stain, and Pass dry-rubbing test
Class A	Pass blurring-highlight test, and Pass wet-rubbing test
Class B	Pass blurring-highlight test, and Fail wet-rubbing test
Class C	Fail blurring-highlight test, and Pass disappearing-highlight test
Class D	Fail disappearing-highlight test

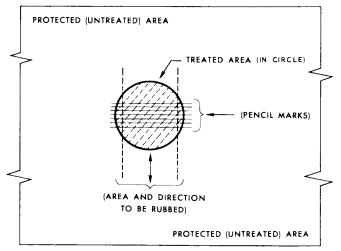


FIG. 2 Method of Applying and Rubbing Pencil Marks on the Treated Area

paper) until the minimum diameter of the flat, circular cross section is half that of the full graphite core.

8.1.3 Blurring-Highlight Test—In a well-lighted location, hold the specimen so that the image of a small light source, such as a frosted lamp bulb, is observed as a highlight in the protected area, the line of vision being within 45° perpendicular to the surface (a desk lamp with an incandescent bulb is recommended for this purpose). Focus the eyes on the image of the light source, then slowly shift the specimen just sufficiently to bring this image into the treated area observing it as it passes across the boundary line between the two areas. Ignore any

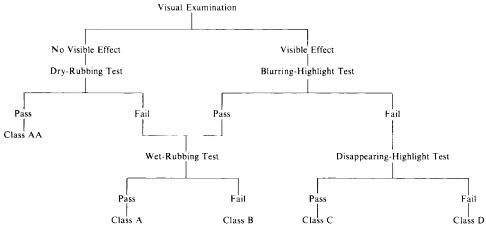


FIG. 1 Flow Sheet of Test Procedure for Classification of Treated Specimens



color difference in the enamel due to staining. If a definite blurring of the image is observed as it passes from the protected to the treated area, the specimen shall be rated as Class C or Class D. Proceed to 8.1.5 to determine final classification. If a blurring image is not observed, the specimen shall be rated as Class A or Class B. Proceed to 8.1.4 to determine final classification.

- 8.1.4 Wet-Rubbing Test—Using the procedure specified in 8.1.2 mark the treated area and rub the marks with a clean, soft cotton towel which has been dipped in water and twisted to remove any excess. For this test, make new marks in a location other than that used for the dry-rubbing test. Do not use soap, abrasive, or similar cleaning material. If the marks are completely removed from treated area, the specimen shall be rated as Class A. Otherwise it shall be rated as Class B.
- 8.1.5 Disappearing-Highlight Test—This test is similar in all respects to the blurring-highlight test specified in 8.1.3, except that the criterion in this case is the complete disappearance of the highlight in the treated area. If the highlight is visible in the treated area, the specimen shall be rated as Class

C. If the highlight disappears in the treated area, the specimen shall be rated as Class D.

9. Report

9.1 Report the assigned grade for each specimen. If the citric acid treatment is performed at temperatures outside the stated tolerances, this variation should be reported.

10. Reproducibility

- 10.1 Since variations may occur during enameling which affect the acid resistance, several specimens should be treated to determine the class of commercial ware.
- 10.2 Ratings of individual specimens shall be considered as due to actual differences in the acid resistance of the specimens, not to differences in the test procedure.

11. Precision and Bias

11.1 The precision and bias of this test method is being established.

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