Designation: C694 – 90a (Reapproved 2016)

Standard Test Method for Weight Loss (Mass Loss) of Sheet Steel During Immersion in Sulfuric Acid Solution[1]

This standard is issued under the fixed designation C694; 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.

INTRODUCTION

In the manufacture of porcelain-enameled ware, some formed steel articles are pretreated with acid and a nickel deposit to ensure one coat enamel adherence. The pretreatment comprises, in part, of etching the steel surface with sulfuric acid solution and in depositing nickel on the steel surface from a nickelous sulfate solution. Conditions are maintained to provide a minimum amount of metal removal (weight loss) (mass loss) in the acid solution and a minimum amount of nickel deposition. These minimums are particularly critical in direct-on enameling in which the ground-coat enamel with its adherence promoting oxides is omitted.

1. Scope

1.1 This test method covers the evaluation of the weight-loss (mass loss) characteristics of sheet steel in sulfuric acid solution.

1.2 This test method provides means of rating the effectiveness of in-plant pretreatment acid solutions in preparing steel surfaces for porcelain enameling.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses are 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. Terminology

2.1 *Definitions:*

2.1.1 *quarter lines*—imaginary lines parallel to the direction of rolling, positioned at a distance from the sheet mill edge equal to one quarter of the sheet width.

3. Summary of Test Method

3.1 Representative sheet-steel specimens are selected, measured, cleaned, and weighed prior to immersion for measured periods in a bath of dilute sulfuric acid that has been preconditioned by controlled solution of panels of the same sheet steel. The specimens are rinsed, dried, and reweighed after the timed exposure.

3.2 Values of weight loss (mass loss) per unit area are calculated for the four acid immersion periods and, if desired, the rate of weight loss (mass loss) per unit area per unit time is calculated.

4. Significance and Use

4.1 The results of this test method can be used to evaluate the pickle weight-loss (mass loss) characteristics of a given lot of sheet steel in dilute sulfuric acid solution, and may enable the enamel processor to select a pickling time that will provide satisfactory porcelain enamel bond.

4.2 The results of this test method can be used to evaluate the effectiveness of the enamel processor's pretreatment system in preparing the steel for porcelain enameling, and may aid the processor in obtaining satisfactory porcelain enamel bond.

5. Apparatus

5.1 *Analytical Balance,* accurate to 0.01 g.

5.2 *Linear Measuring Device.*

5.3 *Borosilicate Glass Container,* having an inside diameter of about 11½ in. (290 mm) and an outside depth of about 11 in. (280 mm).

5.4 *Water Bath,* heated, of sufficient size to immerse the glass container (5.3) to within about 1 in. (25 mm) of its top.

5.5 *Glass Plate or Acid-Resistant Porcelain-Enameled Steel Sheet,* sufficient to cover the container described in 5.3.

[1] This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatingsand is the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.

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5.6 *Stainless Steel Jig,* for support of test specimens (see Fig. 1, Fig. 2, and Fig. 3).

6. Reagents and Materials

6.1 *Distilled Water.*

6.2 *Isopropyl Alcohol* ($(CH_3)_2CHOH$).

6.3 *Methyl Alcohol* (CH_3OH).

6.4 *Steel Sheet,* sufficient in size to provide the panels and strips described in 6.4.1 and 6.4.2, and to provide the test specimens described in 7.1 and 7.1.1.

6.4.1 Shear two to four 4 by 6-in. (102 by 152-mm) panels from the steel sheet of 6.4. Use these panels in the preconditioning in accordance with 9.3.3 and 9.3.4.

6.4.2 Shear ten to twelve ¼ by 5-in. (6 by 127-mm) strips from the steel sheet of 6.4. Fashion these strips into hooks for hanging test specimens from the stainless steel jig.

6.5 *Sulfuric Acid* (H_2SO_4), American Chemical Society (ACS) reagent grade.

6.6 *Trisodium Phosphate*—($Na_3PO_4 \cdot 12H_2O$), granular, technical grade.

7. Sampling

7.1 Shear eight test specimens, each 4 by 6 in. (102 by 152 mm), from within the quarter lines of the sheet or coil.

7.1.1 Choose specimens from rust-free areas that do not contain the mill identification stamp.

7.1.2 Identify the specimens by steel die stamping.

8. Test Specimens

8.1 File edges of the eight test specimens lightly to remove shearing burrs.

8.1.1 Punch or drill a hole near one end, at the center of the specimen width.

8.1.2 Determine the width, *W,* and length, *L,* of the test specimens to the nearest 0.01 in. (nearest 1 mm).

8.1.3 Thoroughly clean the specimens with methyl alcohol. (Thereafter, handle the specimens by the edges with clean white gloves.)

8.1.4 Dry in still air.

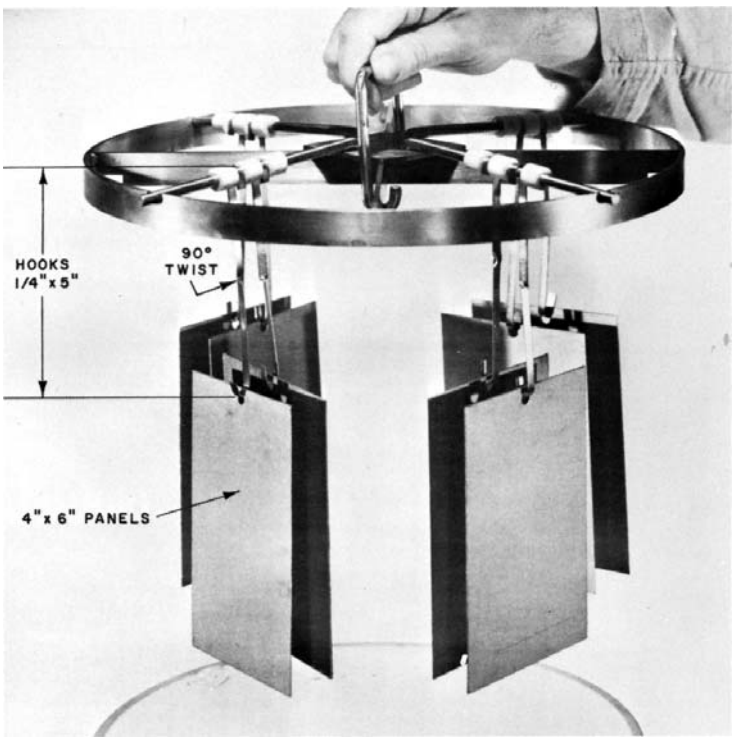
8.1.5 Store the specimens in a desiccator until ready for weighing.

8.2 Determine the initial weight (mass), W_1, of each test specimen to the nearest 0.01 g.

8.2.1 Store the specimens in a desiccator until ready to run the test.

9. Preparation of Solutions

9.1 *Cleaner*—Prepare at least 19 L of 5 ± 0.5 weight (mass) percent solution using 53 g of trisodium phosphate per litre of tap water.

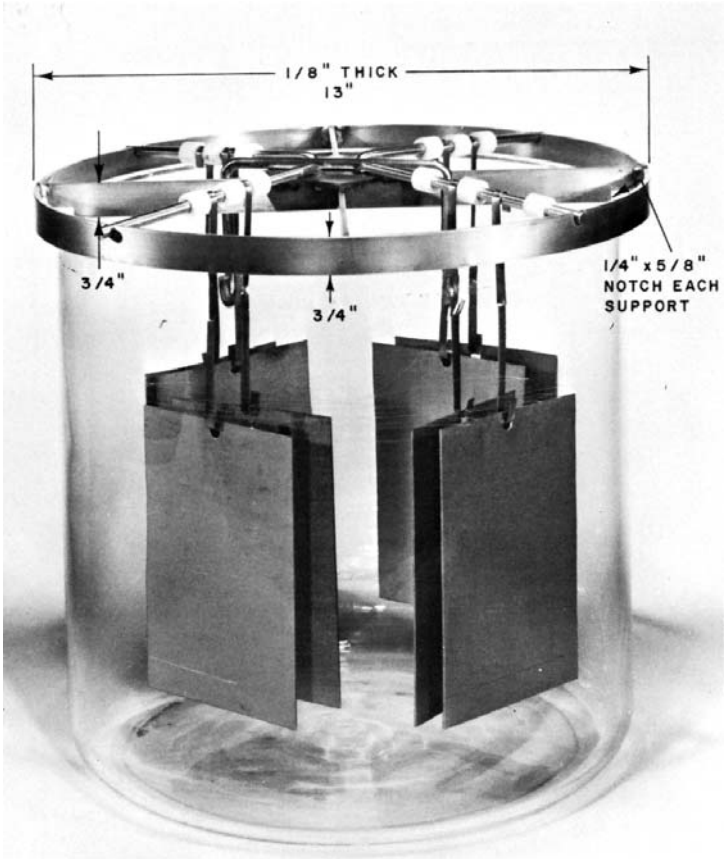


Metric Equivalents

in.	¼	4	5	6
(mm)	(6)	(102)	(127)	(127)

NOTE 1—All materials are of Type 316 stainless steel.

FIG. 1 Specimens Suspended from Stainless-Steel Jig



Metric Equivalents

in.	⅛	¼	⅝	¼	13
(mm)	(3.2)	(6.4)	(16)	(19)	(330)

FIG. 2 Panels Immersed in Acid Solution with Jig Seated on Rim of Battery Jar

9.1.1 Control the cleaner temperature at 190 to 195°F (88 to 91°C).

9.2 *Cleaner Rinse*—At least 19 L of tap water.

9.2.1 Control the rinse temperature at 170 ± 5°F (77 ± 3°C).

9.3 *Sulfuric Acid Solution*—Add 765 mL of ACS reagent grade H_2SO_4 to 16 900 mL of distilled water in the glass container described in 5.3.

9.3.1 Immerse the acid container to within about 1 in. (25.4 mm) of its top in the heated water bath described in 5.4. Do *not* heat the acid with an immersion heater.

9.3.2 Control the acid temperature at 155 ± 1°F (68 ± 1°C).

9.3.3 Process the two to four panels in 6.4.1 through the cleaner (9.1) and cleaner rinse (9.2) so that the panels are thoroughly cleaned, as evidenced by freedom from water breaks.

9.3.4 Immerse the two to four panels in the acid solution to dissolve a total of 15 to 30 g of iron. Do *not* add iron powder or chemicals such as ferrous sulfate ($FeSO_4 \cdot 7H_2O$) to the acid to obtain the required dissolved iron.

9.3.5 To avoid evaporation, place the glass plate or acid-resistant porcelain-enameled steel sheet on the acid container when the acid is not in use.

9.4 *Acid Rinse*—At least 19 L of tap water.

9.4.1 Adjust the rinse to a pH of 2.5 to 3.0 with H_2SO_4.

9.4.2 Maintain the acid rinse at room temperature.

9.5 *Final Rinse*—At least 19 L of tap water.

9.5.1 Maintain the final rinse at room temperature.

9.6 *Isopropyl Alcohol Rinse*—Sufficient quantity for complete immersion of two panels.

9.6.1 Maintain the alcohol rinse at room temperature.

10. Procedure

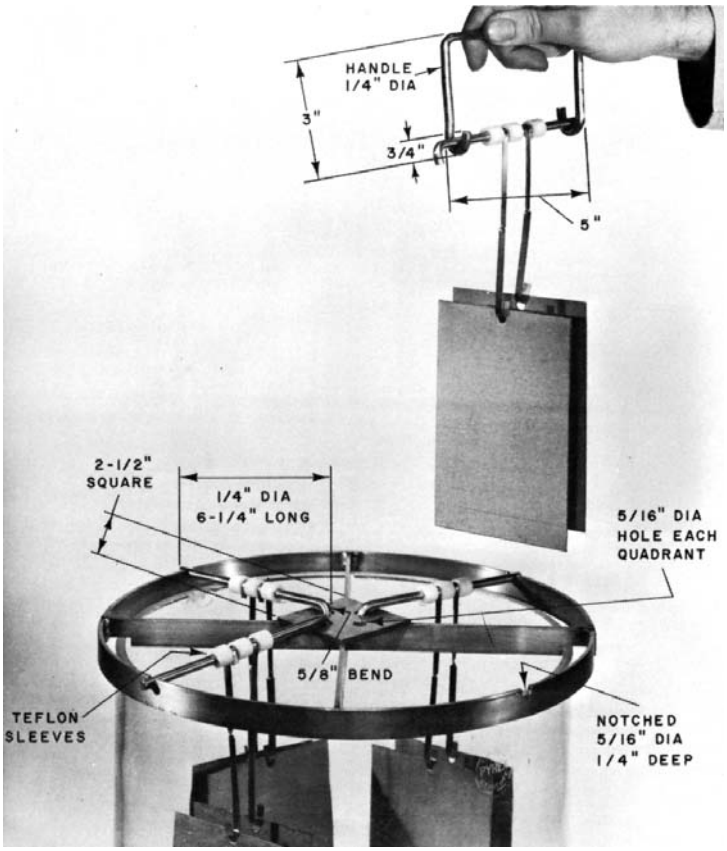
10.1 Suspend the eight specimens on the stainless steel jig (Fig. 1) using hooks made of metal strips (6.4.2).

10.2 Immerse the specimens in the cleaner (9.1) for 10 min.

10.2.1 Discard the cleaner in accordance with 10.3.2.

10.3 Transfer the eight specimens to the cleaner rinse (9.2), and immerse the specimens for five 15-s periods (momentarily remove the specimens from the rinse after each immersion period).

10.3.1 If water breaks are observed on the specimens after the last immersion period, repeat 10.2 and 10.3.



Metric Equivalents

in.	¼	5/16	⅝	¾	2½	3	5	6½
(mm)	(6.4)	(8)	(16)	(19)	(63.5)	(76)	(127)	(165)

FIG. 3 Panels Being Removed from Acid Solution After 5-min Immersion

10.3.2 If water breaks are observed after repeating 10.2 and 10.3 three times, prepare a new cleaner solution and repeat 10.2 – 10.3.1.

10.3.3 Discard the cleaner rinse after processing each group of eight specimens.

10.4 Transfer the eight specimens to the H_2SO_4 solution taking care not to agitate the solution or the specimens (Fig. 2).

10.4.1 Discard the acid solution after processing each group of eight specimens.

10.5 Transfer a set of two specimens from the acid solution (Fig. 3) to the acid rinse at successive 5 min ± 5-s intervals.

10.5.1 Agitate each set of two specimens for 1 min.

10.5.2 Discard the rinse after processing each group of eight specimens.

10.6 Transfer each set of specimens to the final rinse.

10.6.1 Agitate the specimens for 30 s.

10.6.2 Discard the final rinse after processing each group of eight specimens.

10.7 Transfer the specimens to the isopropyl alcohol.

10.7.1 Agitate the specimens for 30 s.

10.8 Dry the specimens in still air.

10.9 Place the specimens in the desiccator until they are ready for weighing.

10.10 Determine the final weight (mass), W_f, of each specimen to the nearest 0.01 g.

11. Calculation

11.1 Calculate the weight loss (mass loss) in grams per square feet of surface, X, using Eq 1:

$$X = 72(W_i - W_f)/(L \times W) \quad (1)$$

where:

W_i = initial panel weight (mass), g,
W_f = final panel weight (mass), g,
L = length of panel, in., and
W = width of panel, in.

11.2 Calculate the weight loss (mass loss) in grams per square decimetre of surface, Y, using Eq 2:

$$Y = 5(W_i - W_f)(10^3)/(L \times W) \quad (2)$$

where:

W_i = initial panel weight (mass), g,
W_f = final panel weight (mass), g,

L = length of panel, mm, and
W = width of panel, mm.

12. Report

12.1 The report shall include the following:

12.1.1 Calculated weight loss (mass loss) per unit area and immersion time for each of the eight panels,

12.1.2 Average weight loss (mass loss) per unit area for each set of two panels at each immersion time, and

12.1.3 Amount of iron dissolved in the acid solution (9.3.4).

13. Precision and Bias

13.1 *Precision*—The 95 % reproducibility limits showing the difference between two test results is 3.99 ± 0.255 g/ft^2 (55.42 ± 3.54 g/m^2) for repeatability and 3.99 ± 0.581 g/ft ^{2} (55.42 ± 8.07 g/m^2) for reproducibility, where 3.99 g/ft^2 (55.42 g/m^2) is the weight (mass) loss.

13.2 *Bias*—The major known sources of bias in this test method are: (*1*) the amount of iron dissolved in the sulfuric acid solution prior to the testing of the samples and its source, and (*2*) the requirement that the surface of the sheet steel samples be clean and show no water break before they are immersed in the sulfuric acid. Both of these sources of bias have been pointed out in this test method and the procedure is designed to minimize them.