



Designation: C 694 – 90a (Reapproved 2000)

Standard Test Method for Weight Loss (Mass Loss) of Sheet Steel During Immersion in Sulfuric Acid Solution¹

This standard is issued under the fixed designation C 694; 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, formed steel articles are pretreated to ensure 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 precon-

ditioned 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).

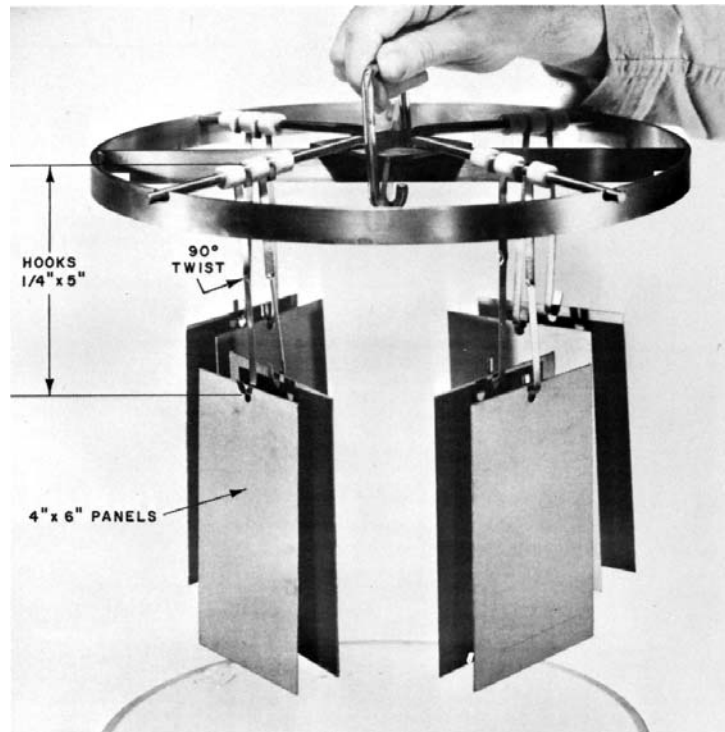
5.4 *Water Bath*, heated, of sufficient size to immerse the glass container (5.3) to within about 1 in. (25 mm) of its top.

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

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

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

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Metric Equivalents

in. (mm)	1/4 (6)	4 (102)	5 (127)	6 (127)
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NOTE 1—All materials are of Type 316 stainless steel.

FIG. 1 Specimens Suspended from Stainless-Steel Jig

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6. Reagents and Materials

- 6.1 *Distilled Water.*
- 6.2 *Isopropyl Alcohol ((CH₃)₂CHOH).*
- 6.3 *Methyl Alcohol (CH₃OH).*
- 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 1/4 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 (H₂SO₄)*, American Chemical Society (ACS) reagent grade.
- 6.6 *Trisodium Phosphate*—(Na₃PO₄·12H₂O), 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.
 - 9.1.1 Control the cleaner temperature at 190 to 195°F (88 to 91°C).