



Standard Test Methods for Fusion Flow of Porcelain Enamel Frits (Flow-Button Methods)¹

This standard is issued under the fixed designation C374; 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.

^{ε1} NOTE—Sections 3 and 8 corrected editorially in May 2015.

INTRODUCTION

This test provides a procedure to compare the fluidity of a glass frit or a porcelain enamel powder to a standard reference material at a fixed temperature above the sample's glass temperature.

1. Scope

1.1 These test methods cover evaluation of the relative fusion flow characteristics of samples of a given porcelain enamel frit by comparison with an established standard for that frit.

1.2 Two test methods are included, differing only in certain details of the samples and in the apparatus and procedure for preparation of test specimens. Both test methods give equally reproducible results and provide a satisfactory basis for comparison of fusion flow of the sample with that of the established standard.

1.2.1 *Test Method A* employs granular particles of frit to which a bonding agent has been added. Button specimens are formed under high pressure in a hydraulic press.

1.2.2 *Test Method B* employs crushed, sized particles of frit to which a bonding agent has been added. Button specimens are formed in a steel mold by hand.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

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. Referenced Document

2.1 *ASTM Standards*:²

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

TEST METHOD A

3. Apparatus

3.1 *Mortar*, of hard steel, resistant to abrasion by the porcelain enamel frit, and conforming to the dimensions shown in **Fig. 1**.

NOTE 1—Suitable mortars are available commercially under the designation “tool steel crushing mortar.”

3.2 *Sieves*—No. 12 (1.70-mm) and No. 200 (75-μm) sieves conforming to Specification **E11**.

3.3 *Hydraulic Press*, capable of developing 3500-lbf (15 600 N) force (**Fig. 2**).

3.4 *Steel Mold Assembly*, consisting of a die and plunger, and having an inside diameter of ½ in. (12.7 mm) over its length of 2 ⅞ in. (54 mm) as illustrated in **Fig. 3**.

3.5 *Fusion Flow Rack*, preferably constructed of heat-resisting alloy and conforming to the detailed requirements shown in **Fig. 4**.

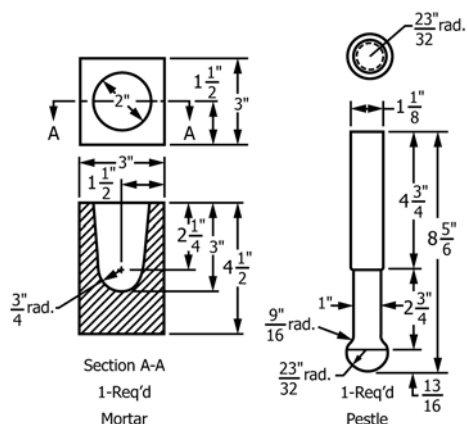
4. Sample

4.1 A representative sample of the frit to be tested shall be obtained, mixed thoroughly, and reduced by quartering to about 25 g. This sample shall be crushed in a hard steel mortar to pass a No. 12 (1.70 mm) sieve and be retained on a No. 200 (75 μm) sieve.

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

Current edition approved May 1, 2015. Published May 2015. Originally approved in 1955. Last previous edition approved in 2014 as C374 – 14. DOI: 10.1520/C0374-14R15E01.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



1 in. = 25.4 mm

FIG. 1 Mortar

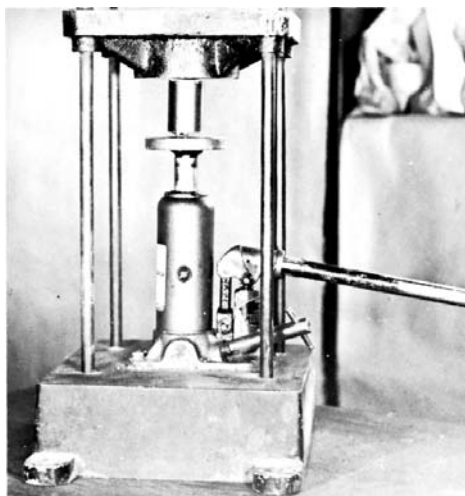


FIG. 2 Hydraulic Press

4.2 For wet-ground enamels the sample shall be evaporated to dryness in an evaporation dish. After cooling, the dried enamel shall be loosened and again pulverized using a pestle and mortar or similar apparatus.

4.3 Dry ground samples of frit, ready-to-use powder, or enamel powder for dry electrostatic application may also be tested.

5. Test Specimen

5.1 Weigh 3.5 ± 0.05 -g samples of the standard frit and of the material to be tested and thoroughly mix each of these samples with four or five drops of a 1 % gum arabic solution. Mold fusion button test specimens of the standard and of the sample as directed in 5.2. For dry electrostatic powder samples, use four to five drops of acetone instead of the aqueous gum arabic solution.

5.2 Place the sample in the steel mold. Place the mold assembly containing the sample in a hydraulic press and bring the press up to 3000 to 3500 lbf (13.3 to 15.6 kN) total load, and immediately release (Fig. 2). Force the formed button out

of the mold with the plunger, taking care not to damage the button in any way that might change the dimensions.

5.3 Dry the test specimens in a suitable oven at 212°F (100°C) for 1 h.

NOTE 2—Special care must be exercised in drying buttons formed by this method, before firing, in order to prevent bloating and possible eruption of the button when first placed in the furnace.

6. Procedure

6.1 *Mounting*—Place the dried fusion buttons on a fired groundcoat plate. The location of the test specimens with respect to the standard may be modified as desired. Although the gage of metal used for the ground-coated plate is not of critical importance, most satisfactory results will be obtained when the metal is not heavier than 18 gage nor lighter than 22 gage (1.02 to 0.044 mm). Use the same gage of metal consistently from test to test in order to reduce the number of variables to a minimum. The groundcoat enamel used to coat the groundcoat plate shall have a median firing temperature no more than 50°F (28°C) below the median firing temperature for the most refractory frit being tested. Place the groundcoat plate with fusion buttons in place in a horizontal position on the fusion flow rack, which shall previously have been heated to furnace temperature, and place the entire test assembly in the furnace immediately.

6.2 *Firing*—Fire in a furnace in which the atmosphere is oxidizing and entirely free of any products of combustion. Heat distribution within the furnace shall be such that no portion of the fusion buttons will vary more than $\pm 5^\circ\text{F}$ ($\pm 3^\circ\text{C}$) from any other button or portion thereof. The temperature shall approximate the median firing temperature for the standard that is used as a basis for comparison. Predetermine, by one or more pre-tests, the holding time, i. e. the time required from placing the fusion buttons in the furnace in the horizontal position (Fig. 5(a)) until such time as fusion of the buttons is evident from the rounded appearance of the tops of the buttons. For all tests of the same materials, the fusion buttons shall remain in the horizontal position for the predetermined holding time. At the end of the holding time, release the groundcoat plate to the vertical position (Fig. 5(b)) and allow the fusion buttons to flow a minimum of 50 mm, following which remove the test assembly from the furnace and allow the buttons to cool. When the flow is completed, the sides of the fusion buttons should be approximately parallel; that is, there should be no excessive spreading of the fusion buttons in the horizontal position, indicating that the buttons were allowed to remain in the horizontal position for too long a time.

6.3 *Measuring Flow*—Measure the greatest length of the fusion flow to the nearest 1 mm for each fusion button, including the standard.

7. Report

7.1 Report the length of the fusion flow for each test specimen in comparison with the standard run at the same time. For example, if a given specimen flows 50 mm against a flow for the standard of 55 mm, the results shall be reported as “50/55 mm.” If the width of the test specimen is observed to

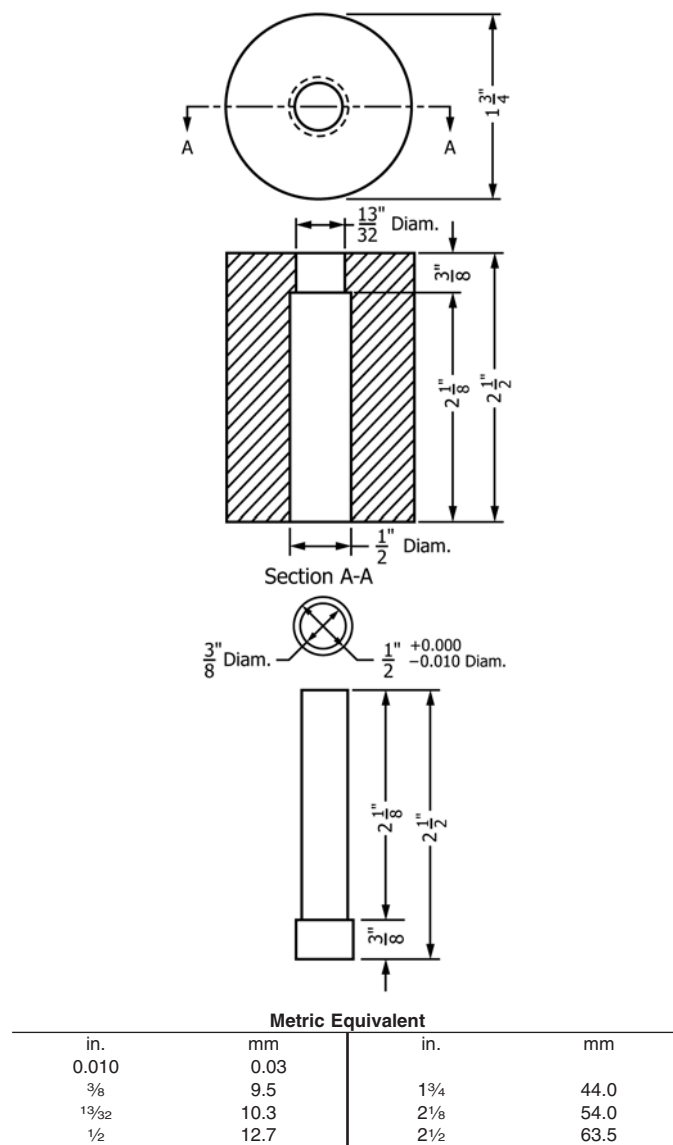


FIG. 3 Mold for Test Specimens—Test Method A

vary more than 10 % from the width of the standard, length-times-width values shall be calculated for the comparison.

TEST METHOD B

8. Apparatus

8.1 *Mortar*—See 3.1.

8.2 *Sieves*—No. 60 (250 μ m) and No. 200 (75 μ m) sieves conforming to Specification E11.

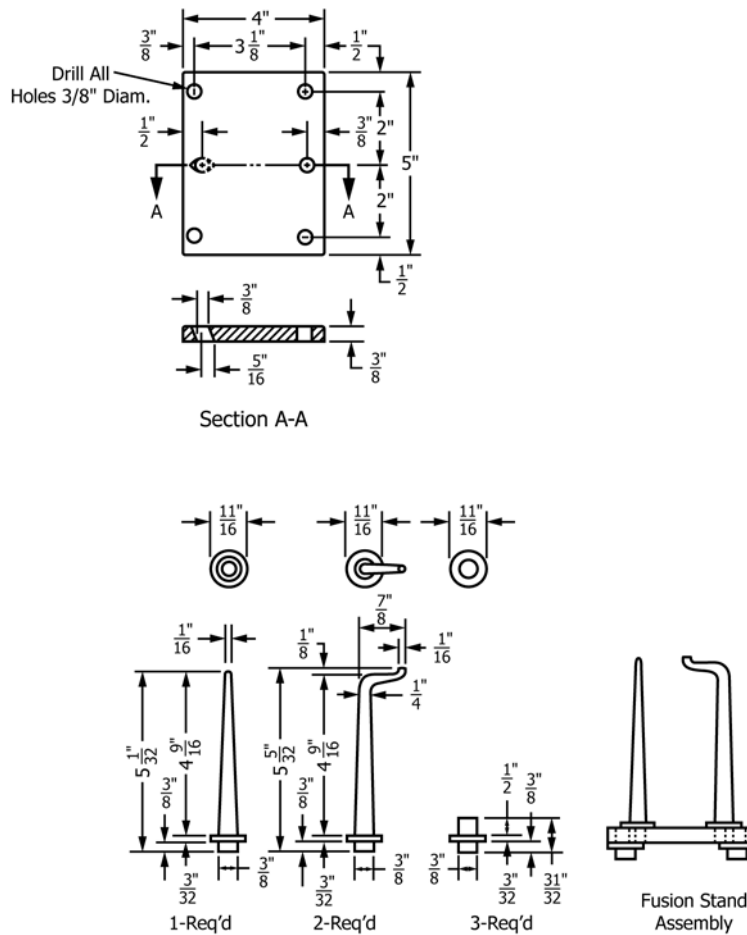
8.3 *Steel Mold Assembly*, consisting of a die, plunger, and backup disk as shown in Fig. 6. The inside diameter of the mold shall be 1/2 in. (12.7 mm) over its length of 1 1/4 in. (31.8 mm).

8.4 *Fusion Flow Rack*—See 3.5.

9. Sample

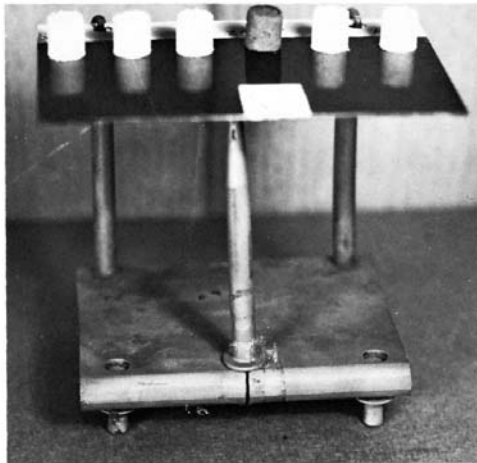
9.1 A representative sample of the frit to be tested shall be obtained, mixed thoroughly, and reduced by quartering to about 25 g. This sample shall be crushed by impact in a hard steel mortar to pass a No. 60 (250 μ m) sieve and be retained on a No. 200 (75 μ m) sieve. In order to avoid the excessive reduction of fines, they shall be removed frequently during the process of reduction by throwing the sample on the sieve and continuing the crushing of the coarser particles until all the sample passes through the sieve.

9.2 For wet-ground enamels the sample shall be evaporated to dryness in an evaporation dish. After cooling, the dried

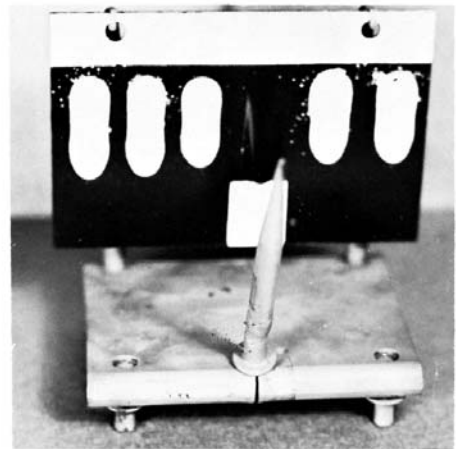


1 in. = 25.4 mm

FIG. 4 Fusion Flow Rack



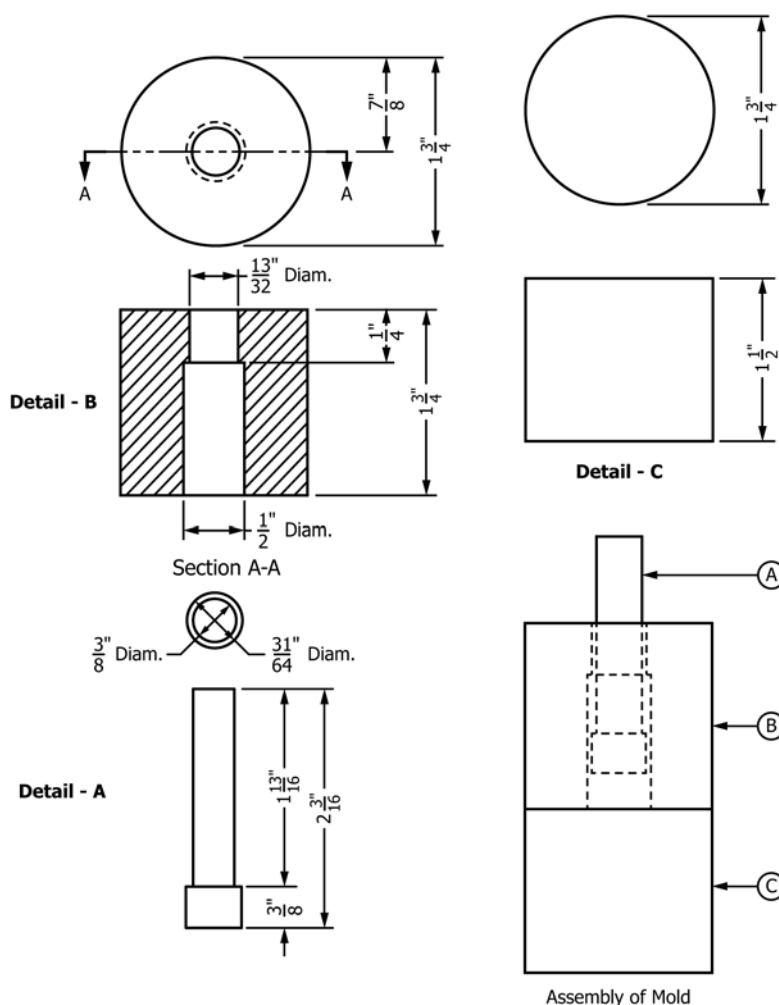
(a) Fusion Buttons in Horizontal Position



(b) Fusion Buttons in Vertical Position

FIG. 5 Fusion Test Assembly

enamel shall be loosened and again pulverized using a pestle and mortar or similar apparatus.



1 in. = 25.4 mm

FIG. 6 Details of Mold for Test Specimens—Test Method B

9.3 Dry ground samples of frit, ready-to-use powder, or enamel powder for dry electrostatic application may also be tested.

10. Preparation of Test Specimens

10.1 Weigh 3.5 ± 0.05 g samples of the standard and of the material to be tested and thoroughly mix each of these samples with 4 or 5 drops of a 1 % gum arabic solution. Mold fusion button test specimens of the standard and of the sample as directed in 10.2.

10.2 Place the mixture in the steel mold with the plunger in place and tamp the mixture to form a solid button (Note 3). Force the formed button out of the mold, taking care not to damage the button in any way that might change the dimensions.

NOTE 3—Since the height of the button will generally be controlled by the density of the material, for materials of approximately the same density such as leadless frits or lead-bearing frits, it will be found convenient to score the side of the plunger at a point representing the average height of the button for materials of the same density and tamp the

powder to this mark each time a button is formed.

10.3 Dry the test specimens (Note 2).

11. Procedure

11.1 Mount and fire the fusion buttons and measure the flow as directed in Section 6.

12. Report

12.1 Report the results of the test as directed in Section 7.

13. Precision and Bias

13.1 No statement is made for precision or bias of these test methods because the results are used to compare the relative fusion flow characteristics of a given porcelain enamel frit, with those of an established standard to determine anticipated performance.

14. Keywords

14.1 comparative test; fusion flow; glass coating; molten flow; porcelain enamel frits

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; <http://www.copyright.com/>