



Standard Test Methods for Sieve Analysis of Wet-Milled and Dry-Milled Porcelain Enamel¹

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^{ε1} NOTE—Sections 4 and 10 corrected editorially in May 2015.

INTRODUCTION

These test methods provide a rapid means of determining the fineness of glass frit in wet- or dry-milled porcelain enamel coating materials by sieve analysis. Fineness is a key predictor of fusibility, tearing, gloss, opacity, suspension in the slip, and ease of spraying because of the direct relationship to surface area.

1. Scope

1.1 These test methods cover the determination of the fineness of frit in wet- or dry-milled porcelain enamels and other ceramic coatings for metals by means of the No. 200 (75-μm) or No. 325 (45-μm) sieve.

1.2 The two methods appear as follows:

	Sections
Method A—Referee Method	4 to 9
Method B—Routine Method	10 to 14

1.3 Method A is intended for use where a referee method of higher accuracy is required, while Method B is intended to meet the needs of normal enamel plant production control operations where a rapid, simplified method of sieve testing is required. The accuracy of the simplified method has proved to be entirely adequate for this use. The simplified test, however, is not recommended where high accuracy is required.

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.*

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.

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2. Referenced Documents

2.1 *ASTM Standards*:²

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Significance and Use

3.1 The fineness of the frit has a direct bearing on many of its properties, such as fusibility, tearing, gloss, opacity, suspension in the slip, and ease of spraying.

METHOD A—REFEREE METHOD

4. Apparatus

4.1 *Balance*—The balance or scale shall be of at least 500-g capacity, and accurate to 0.1 g.

4.2 *Sieves*—The sieves shall conform to Specification E11. They shall include the No. 40 (425-μm) sieve and also the No. 200 (75-μm) or the No. 325 (45-μm) sieve, or both. A No. 325 sieve shall be used when the fineness is such that, from a sample containing 100 g of dry solids, less than 2 g is retained on a No. 200 sieve. An 8-in. (203-mm) full-height sieve is recommended. This height is preferred because there is less tendency to flood or splash, and also because it fits commercial automatic tapping and shaking machines. All sieves used for

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.

testing shall be standardized initially and after every 50 tests against a reference sieve tested by the National Bureau of Standards and bearing its precision seal. The correction for the sieve used in this test shall be determined by sieving tests made in conformity with the procedure of this test method. Identical samples shall be sieved through the reference sieve and the test sieve. Test materials shall be chosen so that 5 to 10 % of the material will be retained on the reference sieve. The difference between the percentage residue on the reference sieve and that on the test sieve is the amount of correction which shall be algebraically added to, or subtracted from, the correction for the reference sieve to obtain the final correction (**Note 1**). The No. 40 sieve need not be calibrated.

NOTE 1—For example, when comparing the reference sieve with a test sieve, should 8.5 g be retained on the reference sieve and 7.5 g on the test sieve, the total correction for the test sieve would then be $8.5 - 7.5$, or $+ 1.0$.

4.3 Dryer—A suitable means for drying the sieves and slip sample, without exceeding a temperature of 250°F (122°C), shall be provided. No dryer is needed for sieve tests of dry-milled enamel.

4.4 Mechanically Operated Sieve Shaker—The mechanical shaking device shall be such as to produce a lateral and vertical motion of the sieve, accompanied by a jarring action so as to keep the sample moving continuously over the surface of the sieve. If a machine shaker is to be used, the thoroughness of sieving shall be tested by comparing and calibrating with the hand method of sieving, as described in Section 6.

5. Sample

5.1 Wet-Milled Enamel—Select a sample of slip representative of the material to be tested. Protect samples from evaporation. Determine the water content of the slip by drying a representative 100-g sample of slip to constant weight at a temperature not exceeding 250°F (122°C). Pass the sample through a No. 40 sieve before weighing, and discard the material retained on the sieve. Sample the slip after stirring by weighing out to the nearest 0.1 g, a quantity calculated to contain 100 g of dry solids.

5.2 Dry-Milled Enamel—Select a sample representative of the material to be tested and containing 100 g of dry solids, weighed to the nearest 0.1 g.

6. Procedure for Wet-Milled Enamel

6.1 Wet Sieving—Transfer the weighed sample to the No. 200 or No. 325 sieve. Wash the sample through the sieve with a gentle flow of water from a rubber hose until the water passing through the sieve appears clear and free of cloudiness. This usually requires 2 min. Exercise care to prevent any loss of sample because of splashing or overflowing. Dry the sieve with its residue until the residue easily moves about as a dry powder when the sieve is shaken. Complete the sieving and weighing operation in accordance with 6.2 or 6.3. Make tests in duplicate.

6.2 Hand Sieving—Hold the sieve, with pan and cover attached, in one hand at an angle of about 20° from the horizontal. Move the sieve up and down in the plane of

inclination at a rate of about 150 times per minute, and strike against the palm of the other hand at the top of each stroke. After every 25 strokes, turn the sieve about one sixth of a revolution in the same direction. Continue the operation until not more than 0.05 g passes through the sieve in 1 min of continuous sieving. Weigh the portion of the sample retained on the sieve to the nearest 0.1 g.

6.3 Machine Sieving—If a mechanically operated sieve shaker is used, vary the time during which the sieve (with pan and cover attached) and the sample are shaken, and note the length of time necessary to operate the sieve shaker in order to get the same result as that obtained with hand sieving. Calibrate the machine-sieving operation in terms of hand sieving.

7. Procedure for Dry-Milled Enamel

7.1 Transfer the sample quantitatively to the No. 200 or 325 sieve. Complete the sieving and weighing operation in accordance with 5.2 or 5.3. Make tests in duplicate.

8. Calculation and Report

8.1 Using the average of duplicate runs, report the fineness of the frit in percentage by mass (to the nearest 0.1 %) of the dry solids content of the sample retained on the No. 200 or 325 sieve. The weight in grams is equivalent to weight percentage.

9. Precision and Bias

9.1 Precision—It is generally accepted within the porcelain enamel industry that duplicate tests run by the same operator in the same laboratory should show a precision of ± 0.5 or less. Failure to duplicate determinations within this limit indicates the necessity for repetition of the test. The precision of duplicate tests is negatively influenced by variations in operator technique, sieve binding and wear, and, when it is used, the condition of mechanical sieving equipment. It is also influenced by changing conditions in the test sample which occur over time and which result in particle agglomeration due to chemical reactions.

9.2 Bias—No justifiable statement on bias can be made since the true value of sieve analysis cannot be established by an acceptable standard sample.

METHOD B—ROUTINE METHOD

10. Apparatus

10.1 Balance—The balance or scale shall be at least 200-g capacity, and accurate to 0.1 g.

10.2 Sieves—The sieves shall conform to Specification E11 and shall include the No. 40 (425- μ m) and the No. 200 (75- μ m) sieves. An 8-in. (203-mm) full-height sieve is recommended. This height is preferred because there is less tendency to flood or splash, and also because it fits commercial automatic tapping and shaking machines. A sieve properly cared for will have 500 tests. It should, however, be compared from time to time with a master standard or reference sieve. It is recommended that an extra sieve be purchased and preserved as a master standard against which all sieves in use or subsequently purchased can be standardized.

10.3 *Container*—A container suitable for weighing a 100-g sample.

10.4 *Dryer*—See 4.3

10.5 *Mechanically Operated Sieve Shaker*—See 4.4.

11. Sample

11.1 Select a sample of slip from the mill before unloading and pass it through a No. 40 sieve before weighing. Discard the material retained on the sieve. The sample shall consist of 100 g of slip for wet-milled porcelain enamel or 100 g of ground frit for dry-milled porcelain enamel.

12. Procedure

12.1 *Wet-Milled Porcelain Enamel*—Transfer the 100-g sample of slip to the No. 200 sieve. Wash the sample through the sieve with a stream of gently running water, care being exercised to prevent splashing or overflowing of the sieve. Continue washing until the water passing through the sieve appears clear and free from cloudiness. This usually requires at least 2 min. Dry the sieve with its residue until the residue easily moves about as a dry powder when the sieve is shaken (Note 2). Shake the sieve in an automatic shaking and tapping machine, or by hand, until no further material passes through the meshes. This usually requires from 5 to 7 min. Transfer the residue remaining on the sieve to the balance and weigh to the nearest 0.1 g.

NOTE 2—It is recommended that, at this point, the residue be transferred to another sieve for shaking. The use of a second sieve minimizes the error caused by the clogging when both washing and shaking are accomplished with the same sieve.

12.2 *Dry-Milled Porcelain Enamel*—Place the 100-g sample of frit powder in a No. 200 sieve and shake in an automatic shaking and tapping machine, or by hand, until no further material passes through the meshes (Note 3). This usually requires from 5 to 7 min. Transfer the residue remaining on the sieve to the balance and weigh to the nearest 0.1 g.

NOTE 3—The end point is usually taken as the time at which not more than 0.1 g of material passes through the sieve with 1 min shaking. A little experience will indicate to the operator when the shaking operation is complete.

13. Calculation and Report

13.1 Report the weight in grams of the residue on the screen as equivalent to weight percentage of the original slip, or frit powder, specimen.

14. Precision and Bias

14.1 *Precision*—It is generally accepted within the porcelain enamel industry that duplicate tests run by the same operator in the same laboratory should show a precision of ± 0.5 or less. Failure to duplicate determinations within this limit indicates the necessity for repetition of the test. The precision of duplicate tests is negatively influenced by variations in operator technique, sieve binding and wear, and, when it is used, the condition of mechanical sieving equipment. It is also influenced by changing conditions in the test sample which occur over time and which result in particle agglomeration due to chemical reactions.

14.2 *Bias*—No justifiable statement on bias can be made since the true value of sieve analysis cannot be established by an acceptable standard sample.

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