



## Standard Test Methods for Abrasion Resistance of Porcelain Enamels<sup>1</sup>

This standard is issued under the fixed designation C 448; 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.

### 1. Scope

1.1 These test methods cover determination of the resistance of porcelain enamels to surface abrasion and subsurface abrasion.

1.2 *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 Documents

#### 2.1 ASTM Standards:

C 346 Test Method for 45-deg Specular Gloss of Ceramic Materials<sup>2</sup>

### 3. Summary of Test Methods

3.1 The first of the tests described herein is intended for the determination of the resistance to surface abrasion of porcelain enamels for which the unabraded 45° specular gloss is more than 30 gloss units. It consists essentially of measuring the specular gloss of the specimens before and after a specified abrasive treatment of the surface, and taking the percentage of the original specular gloss that is retained after treatment as the surface abrasion index.

3.2 The second test is intended for the determination of the resistance to surface abrasion of porcelain enamels for which the unabraded 45° specular gloss is 30 gloss units or less. It consists of determining the weight loss by a specified abrasive treatment and multiplying this weight loss by an adjustment factor associated with each abrasive tester, lot of abrasive, and lot of calibrated plate glass standards used. The adjusted weight loss is taken as an index of resistance to surface abrasion.

3.3 The third test is intended for the determination of the resistance of porcelain enamels to subsurface abrasion. It consists of determining the slope of the linear portion of the abrasion time-weight loss curve and multiplying by an adjustment factor associated with each abrasion tester, lot of abrasive, and lot of calibrated plate glass standards used. The

adjusted slope is taken as an index of resistance to subsurface abrasion.

### 4. Significance and Use

4.1 When a porcelain enamel is first subjected to abrasion of the type involved in these tests, the rate of wear or attrition is relatively low. As the enamel is subjected to continued abrasion, the rate of wear increases until it reaches a steady value. Thereafter, the rate of wear remains almost constant until the enamel is penetrated and the underlying ground coat or metal exposed. The abrasion that occurs during the period of increasing rate-of-weight loss is defined as surface abrasion and results in reduced gloss and cleanability with high-gloss enamels and a modification of color, appearance, or surface texture, or combination thereof with low-gloss enamels. The abrasion that occurs during the period of steady rate-of-weight loss is defined as subsurface abrasion and results in the destruction of the continuity of the coating. These two types of abrasion are not necessarily proportional, and since it is desirable to be able to determine the resistance of porcelain enamel to both types of abrasion, it is necessary to deal with each one separately.

### 5. Apparatus

5.1 *Balance*, having a capacity of approximately 200 g and accurate to 0.0001 g.

5.2 *Alloy Balls*<sup>3</sup>—Rust-resisting alloy balls of the type used for bearings,  $\frac{5}{32}$  in. (4 mm) in diameter. About 4 lb (1.8 kg) are required. Second-grade balls are satisfactory.

5.3 *Apparatus for Measuring Specular Gloss*,<sup>4</sup> conforming to the requirements of Test Method C 346 and having a repeatable precision of not less than  $\pm 0.5$  gloss unit. Means shall be provided for positioning the specimen, with respect to the gloss head, so that, for a given orientation, the position of the specimen during the final gloss reading may be controlled to within  $\frac{1}{16}$  in. (1.6 mm) of that during the original gloss reading.

NOTE 1—Care should be taken to keep the interior of the glossmeter free of dust. If a glossmeter is used with the opening at the top, a piece of phenolic resin or similar material should be placed over the opening when

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee B-8 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 02.05.

<sup>3</sup> Balls meeting the requirements of this paragraph are available from the Mobay Corporation, 5601 Eastern Avenue, Baltimore, MD 21224.

<sup>4</sup> Suitable instruments are available from: Pacific Scientific Company, Gardner/Neotec Instrument Division, 2431 Linden Lane, Silver Spring, MD 20910; and Hunter Lab, 11495 Sunset Hills Road, Reston, VA 22090.

the instrument is not in use. The lenses should be cleaned at regular intervals with a soft camel's-hair brush.

The black gloss standard should be kept free of scratches, and should be protected by wrapping with a soft cloth when not in use.

**5.4 Apparatus for Oscillating Specimens<sup>5</sup>**—The apparatus used for oscillating the specimens during test shall impart to nine specimens simultaneously a horizontal circular motion such that every point on each specimen describes a circle  $\frac{7}{8}$  in. (22.2 mm) in diameter. The apparatus shall be provided with an automatic timing device capable of being preset to within 1 s of the desired time and shall operate at a frequency of  $300 \pm 3$  cpm (for machine manufactured prior to July 1981) or  $345 \pm 3$  cpm (for machines manufactured after July 1981). The apparatus shall be operated on a firm and level surface.

**5.5 Retaining Rings**—The retaining rings shall be constructed of metal lined with rubber. The inside height and diameter of the rubber-lined ring shall be  $1\frac{3}{16} \pm \frac{1}{16}$  in. ( $30.2 \pm 1.6$  mm) and  $3\frac{7}{16} \pm \frac{1}{16}$  in. ( $87.3 \pm 1.6$  mm), respectively. Provision shall be made for clamping the retaining ring to an enameled metal specimen to produce a watertight seal. Means shall be provided for introducing an abrasive charge after the specimen is secured. Nine retaining rings are required.

**5.6 Buret**, of suitable capacity to deliver 20 mL of water at 20°C (68°F).

## 6. Abrasives

**6.1** For the surface abrasion tests the  $-70 +100$ -mesh fraction of Pennsylvania-type glass sand,<sup>6</sup> preferably as ground from quartzite (quartz rock), shall be used. For the subsurface abrasion test No. 80 grit aluminum oxide abrasive medium<sup>6</sup> shall be used.

## 7. Reference Standards

**7.1** Specimens of standard calibrated polished plate glass<sup>6</sup> which shows a coefficient of variation no greater than 1.5 % when tested for surface abrasion in accordance with the procedure as specified in Section 10, and which meets requirements of Table A1.4 shall be used as reference standards.

## 8. Test Specimens

**8.1** Test specimens shall be  $4\frac{3}{8} \pm \frac{1}{4}$  in. ( $111.1 \pm 6.4$  mm) square, and may be prepared by enameling metal blanks of that size or by cutting a larger piece. Specimens for the weight loss tests should not exceed the capacity of the analytical balance selected for obtaining the weights of the specimens. When gloss measurements are to be made, the specimens tested should be as flat and free of orange peel or wavy surface as possible. Variations from flatness decrease the accuracy of gloss measurements.

**8.2** Six specimens shall be tested for each determination of resistance to surface abrasion or resistance to subsurface abrasion.

## 9. Specimen Preparation

**9.1** Before making any measurements, wash each specimen

with a soft sponge moistened with a warm 1 % solution of trisodium phosphate (distilled water not essential) and rinse in warm, running tap water. If, when rinsing, the water gathers in drops on the surface, repeat the washing treatment until the rinse water spreads evenly. While the specimen is still wet, rinse it with ethyl alcohol. A small stream of alcohol from an ordinary chemical wash bottle will suffice for rinsing. Pure ethyl alcohol is preferable, but if it is not available, ethyl alcohol that has been denatured with up to 5 % of a noncorrosive, highly volatile organic compound such as methyl alcohol may be used. Allow the specimens to air dry in a vertical position and then place in a desiccator. This will prevent damage and moisture absorption of the specimens which can adversely affect the weight values. Gloss and weight determinations should be obtained within a 15-min period after the specimens have been cleaned and placed in the desiccator.

**NOTE 2**—Other denaturants, approved by the U. S. Bureau of Internal Revenue, that are not objectionable for this use are ethyl ether and benzene, either alone or in combination with methyl alcohol. If ethyl alcohol is not available, isopropyl alcohol or acetone may be used, but pure ethyl alcohol is recommended if available. Avoid alcohol denatured with an ingredient of low volatility, which will remain as a surface film on the specimen when used to rinse it before or after treatment. Surface films may significantly affect gloss readings.

## 10. Resistance to Surface Abrasion of Porcelain Enamels Having 45° Specular Gloss of More than 30 Gloss Units

**10.1 Marking of Specimens and Determining Initial 45° Specular Gloss**—Mark each specimen so that its orientation may be controlled. A mark on the back at one edge will suffice. Place this edge against the specimen guide on the gloss head for the first reading. Then make three other readings, turning the specimen clockwise through 90° between readings.

**NOTE 3**—The measured gloss of light-transmitting specimens of such materials as transparent or translucent glass or plastic may be affected by light reflected from the back side of the specimen or transmitted through the specimen from the room. To minimize errors from this source, the following precautions should be taken:

(1) Roughen the back of transparent specimens to eliminate specular reflection from this surface.

(2) Cover back and edges of light-transmitting specimens with an opaque black cloth when measuring gloss to prevent normal room illumination from being transmitted through the specimen, or light from the instrument from being reflected back through the specimen from a light surface in contact with the back of the specimen. This is particularly important when using a glossmeter with the specimen holder on top.

(3) Put no labels near the center of light-transmitting specimens.

**10.2 Determining Correct Abrasion Time**—The correct abrasion time is the time required to reduce the 45° specular gloss of a standard plate glass specimen to  $53 \pm 1$  %.<sup>7</sup> Determine this time by abrading six standard plate glass specimens and calculating the average percentage 45° specular gloss retained. A good trial time is 6.117 min (184 counts) on machines manufactured prior to July 1981, or 4.367 min (150 counts) on machines manufactured after July 1981.

<sup>5</sup> A suitable apparatus is the P.E.I. Abrasion Tester, manufactured by the Keystone Electric Co., 2807 Annapolis Road, Baltimore, MD 21230.

<sup>6</sup> These standard materials are available from the Mobay Corp., 5601 Eastern Ave., Baltimore, MD 21224.

<sup>7</sup> This value may change slightly from time to time when it is necessary to replenish the supply of standards. In any case, the corrected value will be furnished with each lot of standards (see Annex A4).

**10.3 Securing Specimens to Table of Abrasion Tester and Introducing Abrasive Mixture**—Center each specimen in one of the nine available positions and secure by means of the retaining ring. Tighten the two wing nuts simultaneously and uniformly. The amount of tightening shall be just sufficient to provide a watertight seal between the retaining ring and the specimen. Introduce an abrasive charge of  $175 \pm 0.15$  g of  $\frac{5}{32}$ -in. (4-mm) rust-resisting alloy balls,  $3 \pm 0.01$  g of  $-70 + 100$  mesh Pennsylvania-type glass sand, and  $20 \pm 0.2$  mL of water, in that order, through the hole in the top of each retaining ring. After the abrasive charge is introduced, seal the hole in the top of the retaining ring with a cork or rubber stopper.

**10.4 Treatment of Specimens**—Set the automatic timing device for the previously determined time required to reduce the  $45^\circ$  specular gloss of a standard plate glass specimen to 53 %. Set the selector switch to the “automatic” position and close the toggle switch, starting the oscillator. The abrasion tester will then stop after the prescribed abrasion time.

**10.5 Cleaning Specimens after Treatment:**

**10.5.1** Clean the sample specimens and equipment as follows:

**10.5.1.1** Immediately after treatment, loosen the wing nuts that secure the specimens to the abrasion tester. Carefully remove the sample panel, retaining ring apparatus and abrasive medium as a sealed unit. (This can be accomplished by compressing the panel to the retaining ring apparatus with the hands and rotating the assembly from the confines of the securing posts of the abrasion tester. The ball bearings, the abrasive grit, and water can be discharged through a sieve that will allow the sand and water to pass through, but will retain the ball bearings. This method facilitates the cleaning and drying of the ball bearings so that they can be used in subsequent testing cycles.) If any of the ball bearings are dropped and cannot be accounted for during the cleaning of the individual retaining ring assemblies, another  $175 \pm 0.15$  g of ball bearings shall be used in subsequent testing cycles.

**10.5.1.2** Scrub the abraded portion very lightly with a clean sponge that has been saturated with warm water, rinse with warm, running water, and, while still wet, rinse with alcohol and place in a vertical position to dry.

**10.6 Determining Final  $45^\circ$  Specular Gloss**—Make the final gloss readings within 15 min after the specimens have been given the final rinsing, following the procedure outline in 10.1.

**10.7 Computation of Surface Abrasion Index**—For each of the four orientations of the specimens, divide the final specular gloss reading by the initial reading and multiply by 100. The average percentage residual specular gloss for the four positions shall be taken as the surface abrasion index of the specimen. The average index of six specimens after treatment shall be taken as the abrasion index of a given index of enameled metal.

**NOTE 4**—If it is desirable, nine samples may be treated simultaneously, this procedure being repeated until six specimens of each sample have been tested. If less than nine samples are to be tested, any arrangement desired may be used and the arrangement may be such that the number of operations required to test six specimens of each sample is a minimum.

**10.8 Use of Standard Plate Glass Specimens**—As a check

on the performance of the apparatus, test standard, calibrated, plate glass specimens at regular intervals. If the computed surface abrasion index obtained in a check test of six standard glass plates falls outside the limits 52.0 to 54.0 but within the limits 51.5 to 54.5, adjust the abrasion time in accordance with 10.2. If a value below 51.5 or above 54.5 is obtained, defective technique or equipment is indicated, and the source of difficulty should be found and remedied.

**NOTE 5**—This check test need not be made each time enameled metal specimens are tested. The time between check tests will be determined by the number of tests made by the laboratory.

## **11. Resistance to Surface Abrasion of Porcelain Enamels Having $45^\circ$ Specular Gloss of 30 Gloss Units or Less**

**11.1 Determining Initial Weight of Specimens**—Weigh each specimen to the nearest 0.1 mg within 15 min after it has been rinsed with alcohol.

**NOTE 6**—When weight determinations are to be made, the specimens should be handled with care to prevent chipping, which may introduce significant errors. This precaution is particularly important for glass plates, and in determining surface abrasion by weight loss.

**11.2 Securing Specimens to Table of Abrasion Tester and Introducing Abrasive Mixture**—Secure the test specimens to the table of the abrasion tester and introduce the abrasive mixture in accordance with 10.3.

**11.3 Treatment of Specimens**—Set the selector switch to the “automatic” position, set the automatic timing device for 10 min (300 counts) on machines manufactured prior to July 1981, or 10 min (342 counts) on machines manufactured after July 1981.

**11.4 Cleaning Specimens After Treatment**—After treatment, clean the specimens in accordance with 10.5. If the retaining ring has left a black mark on the specimen, this may be removed by scrubbing lightly with a soft sponge. Do not scrub the abraded area.

**11.5 Determining Final Weight of Specimens**—Determine the final weight of each specimen in accordance with 11.1.

**11.6 Computation of Surface Abrasion Index**—For each specimen, multiply the weight loss by an adjustment factor determined as specified in 12.7, but using Pennsylvania-type glass sand. The average adjusted weight loss is taken as the surface abrasion index of the enamel (Note 4).

**11.7 Use of Standard Plate Glass Specimens**—As a check on the performance of the apparatus, test standard, calibrated plate glass specimens at regular intervals in accordance with 10.8 (Note 5).

## **12. Resistance to Subsurface Abrasion**

**12.1 Determining Initial Weight of Specimens**—Determine the initial weight of each specimen in accordance with 11.1.

**12.2 Securing Specimens to Table of Abrasion Tester and Introducing Abrasive Mixture**—Secure the test specimens to the table of the abrasion tester and introduce the abrasive mixture in accordance with 10.3, except use  $3 \pm 0.01$  g of No. 80 grit aluminum oxide abrasive medium in place of the glass sand.

**12.3 Treatment of Specimens**—Set the selector switch to the “automatic” position, set the automatic timing device for 15 min (450 counts) on machines manufactured prior to July



1981, or 15 min (513 counts) on machines manufactured after July 1981.

**12.4 Cleaning Specimens After Treatment**— After treatment, clean the specimens in accordance with 10.5. If the retaining ring has left a black mark on the specimen, this may be removed by scrubbing lightly with a soft sponge. However, do not harshly rescrub the abraded area.

**12.5 Determining Final Weight of Specimens**—Determine the final weight of each specimen in accordance with 11.1.

**12.6 Repeat the steps listed in 12.2-12.5 two times** (a total of three 15-min abrasion periods). Designate the specimen weights after 15, 30, and 45 min of abrasion as  $W_{15}$ ,  $W_{30}$ , and  $W_{45}$ , respectively.

**12.7 Determination of Adjustment Factor**— Determine the adjustment factor for the abrasion tester as follows:

**12.7.1 Test 24 standard plate glass specimens** for eight consecutive 15-min periods as specified in 12.1-12.5, except that it is not necessary to determine the specimen weights after the first three 15-min periods, as these weights are not needed for the subsequent computations. Prior to each weighing, clean the standard plate glass specimens by thoroughly rinsing all surfaces with warm, running tap water; washing all surfaces with a soft sponge moistened with a warm, 1 % solution of trisodium phosphate (distilled water not essential); thoroughly rinsing all surfaces with warm, running tap water; while the specimen is still wet, rinsing front and back with ethyl alcohol. Allow the specimens to air dry in a vertical position and then place them in a desiccator.

**12.7.2 For each specimen**, subtract the weight at the end of eight 15-min periods,  $W_{120}$ , from the weight at the end of four 15-min periods,  $W_{60}$ , and divide by 60 to obtain the rate-of-weight loss.

**12.7.3 Calculate the average rate-of-weight loss** for the 24 specimens.

**12.7.4 Divide this average rate-of-weight loss** for the last four 15-min periods into 4.5671<sup>7</sup> (see Annex A4). An example is given in Table 1.

**12.8 Calculation of Subsurface Abrasion Index**—For each specimen, subtract the weight after 45 min of abrasion,  $W_{45}$ , from the weight after 15 min of abrasion,  $W_{15}$ , and divide the difference by 30. The quotient,  $X_t$ , is the slope of the linear portion of the abrasion time - weight loss curve and shall be taken as the true rate-of-weight loss of the specimen as determined by the laboratory with its particular abrasion tester. The true rate-of-weight loss for each specimen shall then be multiplied by the adjustment factor determined in accordance with 12.7, giving an adjusted rate-of-weight loss,  $\bar{X}_a$ . The average adjusted rate-of-weight loss shall be taken as the subsurface abrasion index of the enamel. An example is given in Table 2 (Note 4).

**12.9 As a check to verify that the correct procedure** has been followed, plot the average weight loss of the enamel as a function of abrasion time for 15, 30, and 45 min of abrasion. These three points should fall approximately on a straight line.

**12.10 Use of Standard Plate Glass Specimens**—As a check on the performance of the abrasion tester, test standard, calibrated, plate glass specimens at regular intervals (Note 5). Use six specimens for the check test. These specimens may be

**TABLE 1 Calculation of Adjustment Factor for Abrasion Tester from Weight-Loss Values for 24 Standard Plate Glass Specimens**

Specimen No.	$W_{60}$ , g	$W_{120}$ , g	$(W_{60} - W_{120})$ , mg	$(W_{60} - W_{120})/60$ , mg/min
1	181.981	181.692	289	4.817
2	179.995	179.701	294	4.900
3	183.633	183.346	287	4.783
4	182.734	182.451	283	4.717
5	176.193	175.911	282	4.700
6	184.109	183.817	292	4.867
7	182.212	181.927	285	4.750
8	176.128	175.837	291	4.850
9	178.492	178.204	288	4.800
10	184.363	184.079	284	4.733
11	173.998	173.712	286	4.767
12	180.299	180.006	293	4.883
13	182.742	182.451	291	4.850
14	180.259	179.977	282	4.700
15	177.668	177.381	287	4.783
16	176.877	176.582	295	4.917
17	181.681	181.397	284	4.733
18	173.362	173.069	293	4.883
19	184.004	183.714	290	4.833
20	181.214	180.919	295	4.917
21	179.292	179.011	281	4.683
22	174.673	174.384	289	4.817
23	176.476	176.191	285	4.750
24	178.610	178.328	282	4.700
Avg				4.797

Adjustment factor =  $5.000/4.797 = 1.042$ .

**TABLE 2 Calculation of Subsurface Abrasion Index of a Porcelain Enamel from Weight-Loss Values for Six Specimens**

Specimen No.	$W_{15}$ , g	$W_{45}$ , g	$X_t$ , mg/min = $(W_{15} - W_{45})/30$	$X_a$ , mg/min = $(1.042)X_t$
1	123.043	122.899	4.80	5.002
2	123.675	123.538	4.57	4.762
3	123.113	122.977	4.53	4.720
4	127.498	127.363	4.50	4.689
5	122.662	122.519	4.77	4.970
6	122.907	122.764	4.77	4.970
Avg				4.8522

Subsurface abrasion index = 4.85.

taken from the group of 24 specimens previously used for determining the adjustment factor as prescribed in 12.7. The same specimens may be used repeatedly for these check tests. Test the six glass plates for four consecutive 15-min periods as specified in 12.1-12.5. Compute the average rate-of-weight loss for this hour of abrasion and multiply by the adjustment factor for the abrasion tester. The adjusted rate-of-weight loss thus obtained should fall within  $\pm 0.15$  mg/min of the appropriate abrasion index value supplied with the standard glass specimens. If an adjusted rate-of-weight loss outside the specified  $\pm 0.15$  mg/min tolerances is obtained, this indicates that a significant change has occurred in the apparatus itself or in the test procedure. If the value obtained in a recheck of six different glass plates, in which all variables are closely controlled, confirms the value obtained in the check test, determine a new adjustment factor by abrading the remaining 12 plate glass specimens for four 15-min periods and dividing an average rate-of-weight loss for all 24 specimens into the 4.5671 (See Annex A4). Use this adjustment factor in subsequent computations. If new plate glass specimens are to be

used, test them first for four 15-min periods in accordance with 12.2-12.4. It is not necessary to make weight-loss determinations for these first four abrasion periods. After this initial hour of abrasion to remove the surface, the specimens may be used as described above.

### 13. Calculations for Single Determination

13.1 Six specimens comprise a sample. For surface abrasion of porcelain enamels having 45° specular gloss of more than 30 gloss units, calculate the grand average of the six average abrasion indices,  $X_g$ , each obtained by averaging the percentage residual gloss for the four positions of one specimen, thus obtaining the mean abrasion index for the sample,  $\bar{X}_g$ . For subsurface abrasion, average the six individual adjusted rates of weight loss,  $X_a$ , to obtain the mean abrasion index for the sample,  $\bar{X}_a$ .

13.2 Calculate the statistical error of the determination as follows (Note 7):

$$e = 1.05 s \quad (1)$$

where:

$e$  = statistical error of the mean value for the sample (95 % confidence), and

$s$  = standard deviation of the six average abrasion indices for individual specimens.

NOTE 7—The factor 1.05 applied only when the number of specimens is 6 and the percentage of confidence is 95. See STP 15D, Part 2, Table 2.<sup>8</sup>

13.3 The standard deviation may be calculated as follows:

$$s = \sqrt{\frac{n(\sum X^2) - (\sum X)^2}{n(n-1)}} \quad (2)$$

where:

$s$  = standard deviation,

$X$  = value of a single observation,

$\sum X$  = sum of the set of observations, and

$n$  = number of observations.

13.4 Sample calculations are given below, the recommended number of decimal places being given in each case (Note 8). In the example of surface abrasion, 60 is subtracted from each value,  $X_g$ , giving much smaller values,  $X'_g$ , which are correspondingly easier to square. Then, to compensate, 60 is added to the average  $\bar{X}'_g$  to obtain the average  $\bar{X}_g$  (Note 9). For Surface Abrasion

Specimen No.	$X_g$	$X'_g = X_g - 60$	$(X'_g)^2$
1	61.5	1.5	2.25
2	61.3	1.3	1.69
3	62.6	2.6	6.76
4	61.3	1.3	1.69
5	61.6	1.6	2.56
6	62.8	2.8	7.84
Sum		11.1	22.79
Mean		1.850	

Applying Eq 2,

$$s = \sqrt{\frac{6(22.79) - (11.1)^2}{6 \times 5}}$$

$$= \sqrt{\frac{136.74 - 123.21}{30}}$$

$$= \sqrt{\frac{13.53}{30}} = \sqrt{0.451}$$

$$s = 0.6716$$

$$e = 1.05 \times 0.6716 = 0.705 \text{ (Eq 1)}$$

$$\bar{X}_g = 1.85 + 60 = 61.85$$

The surface abrasion index is reported as  $61.9 \pm 0.7$ .

For Subsurface Abrasion

Specimen No.	$X_a$	$(X_a)^2$
1	5.002	25.0200
2	4.762	22.6766
3	4.720	22.2784
4	4.689	21.9867
Specimen No.	$X_a$	$(X_a)^2$
5	4.970	24.7009
6	4.970	24.7009
Sum	29.113	141.3635
Mean	4.8522	

Applying Eq 2,

$$s = \sqrt{\frac{6(141.3635) - (29.113)^2}{6(6-1)}}$$

$$= \sqrt{\frac{848.181 - 847.5668}{30}}$$

$$= \sqrt{0.020473} = 0.1431$$

$$= 1.05 \times 0.1431 = 0.1503 \text{ (Eq 1)}$$

$$\bar{X}_a = 4.85$$

The subsurface abrasion index of the group of specimens is reported as  $4.85 \pm 0.15$ .

NOTE 8—Take care to carry the calculations to two or three places beyond the decimal when so indicated in the sample calculations. Otherwise, significant errors are frequently introduced in computing standard deviations.

NOTE 9—The number to be subtracted should be an integer, just lower than the lowest value in the set.

### 14. Difference Between Two Determinations

14.1 The significance (or lack of significance) of a difference between two mean values shall be determined from the ratio  $d/e'$  where  $d$  represents the difference in means and  $e'$  the statistical error in the determination of  $d$ . From the graph in Fig. 1, the value of the ratio  $d/e'$  may be translated into terms of the percentage confidence that the difference in mean values indicates a systematic difference in the types of specimens being tested rather than mere chance fluctuations in sampling and testing, such as might occur even though both sets of specimens were taken from groups in which the grand averages were equal.

14.2 The statistical error,  $e'$  of the difference,  $d$ , between two means shall be determined from the following equation:

$$e' = \sqrt{e_1^2 + e_2^2} \quad (3)$$

where:

$e'$  = error of the difference in means,

$e_1$  = error of one mean value, and

$e_2$  = error of the other mean value.

<sup>8</sup> Manual on Presentation of Data and Control Chart Analysis, ASTM STP 15D, ASTM, 1976. (Issued as a separate publication.)

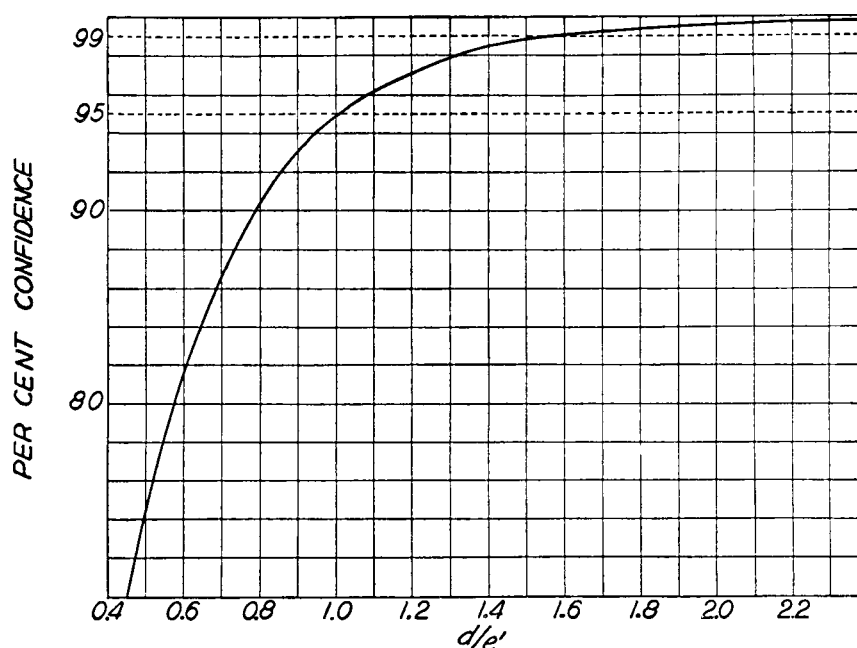


FIG. 1 Variation in Percentage Confidence that a Difference Between Two Determinations is Not Due to Chance Fluctuations with the Ratio  $d/e'$  for Samples of Six Specimens

14.3 Sample calculations are given below:  
For Surface Abrasion:

$$\bar{X}_{g1} = 53.30 \pm 0.92$$

$$\bar{X}_{g2} = 55.62 \pm 1.13$$

$$d = \bar{X}_{g2} - \bar{X}_{g1} = 55.62 - 53.30 = 2.32$$

Applying Eq 3,

$$e' = \sqrt{e_1^2 + e_2^2} = \sqrt{(0.92)^2 + (1.13)^2} \\ = \sqrt{2.123} = 1.46$$

$$d/e' = 2.32/1.46 = 1.59$$

For Subsurface Abrasion:

$$\bar{X}_{a1} = 4.593 \pm 0.143$$

$$\bar{X}_{a2} = 4.309 \pm 0.122$$

$$d = \bar{X}_{a1} - \bar{X}_{a2} = 4.593 - 4.309 = 0.284$$

Applying Eq 3,

$$e' = \sqrt{(0.143)^2 + (0.122)^2} = \sqrt{0.0353} = 0.188 \\ d/e' = 0.284/0.188 = 1.51$$

14.4 From Fig. 1, it is apparent that a ratio of 1.59 indicates slightly more than 99 % confidence and that a ratio of 1.51 indicates slightly less than 99 % confidence. A ratio indicating a percentage confidence equal to or exceeding 99 % is consid-

ered highly significant, 95 % or more but less than 99 % is considered significant, and 90 % or more but less than 95 % is considered indicative. The basing of conclusions on differences in which less than 90 % confidence can be placed is not recommended. If  $d/e'$  equals or exceeds 1, a confidence of 95 % or more is indicated, and the difference is considered significant.

## 15. Precision and Bias

15.1 The precision and bias of this test method will depend upon the uniformity of the samples being tested and the skill and ability of the operator in following the procedures outlined.

15.1.1 The precision of the surface abrasion test as indicated by the maximum allowable variation in determining the surface abrasion index of standard plate glass specimens having an average surface abrasion index of 53 % with a coefficient of variation of 1.5 % or less is  $\pm 1$  % (range 52.0 to 54.0).

15.1.2 The precision of the subsurface abrasion test as indicated by the maximum allowable variation in measuring the subsurface abrasion index of standard plate glass specimens having an adjusted subsurface index of 4.5671 (see Annex A4) with a coefficient of variation of 1 % or less is  $\pm 0.15$ .

15.2 Results of the test may be affected by the frequency of oscillation in cpm delivered by the particular machine used.

ANNEXES

(Mandatory Information)

**A1. CHARACTERISTICS OF ABRASION RESISTANCE TEST MATERIALS**

A1.1 This annex specifies the characteristics of abrasion-resistance test materials (alloy balls, Pennsylvania-type glass sand, No. 80 grit aluminum oxide abrasive medium, and standard soda-lime plate glass specimens) sufficiently to permit the purchase of these materials directly from the appropriate manufacturers if the indicated central source of these materials (see Note 3 and Note 6) is no longer the supply source. The characteristics of the abrasion resistance test materials are listed in Tables A1.1-A1.4.

**TABLE A1.1 Alloy Balls**

Material description	Alloy balls (see 5.2)
Procurement source	Bearings, Inc., 2818 Loch Raven Road, Baltimore, Md. 21218
Characteristics	5/32 in. (4 mm) diameter, rust-resisting, Type 440, 200 grade, 200 balls per pound (0.5 kg)
Amount required for one specimen	175 ± 0.15 g (see 10.3)

**TABLE A1.2 Pennsylvania-Type Glass Sand**

Material description	-70 + 100-mesh fraction of Pennsylvania-type glass sand (see 6.1)		
Procurement source	Pennsylvania Glass & Sand Corp., Berkley Springs, W. Va. 25411		
Characteristics	The abrasive characteristic of this glass sand depends upon the number of cutting surfaces available per sand particle. To obtain the greatest number of cutting surfaces possible it is necessary to specify this silica sand as coming from ground quartzite (quartz rock).		
Amount required for one specimen	3 ± 0.01 g (see 10.3)		
Typical screen analysis	Mesh Size	% Cumulative Retained	
	40	...	
	50	trace	
	70	7.0	
	100	86.0	
	150	99.9	
	Pan	100.—	

**TABLE A1.3 Aluminum Oxide Abrasive Medium**

Material description	No. 80 grit aluminum oxide abrasive medium (see 6.1)			
Trade name	Dynablast			
Procurement source	Norton Co., Abrasive Materials Div., Worcester, Mass. 01606			
Characteristics	Size- 80 grit See composition			
Amount required for one specimen	3 ± 0.01 g (see 12.2)			
Typical composition and screen analysis	Screen Analysis			
	Component	Amount, (%)	Mesh Size	% Cumulative Retained
	Al <sub>2</sub> O <sub>3</sub>	95.8	60	0.08
	TiO <sub>2</sub>	2.6	70	3.79
	SiO <sub>2</sub> <sup>A</sup>	1.0	80	26.91
	Fe <sub>2</sub> O <sub>3</sub>	0.2	100	67.74
	MgO	0.2	150	93.38
	ZrO <sub>2</sub>	0.1	Pan	100.00
	Na <sub>2</sub> O, K <sub>2</sub> O	0.1		

<sup>A</sup> The silica (SiO<sub>2</sub>) value shown is present as combined silica not as free silica. Composition varies slightly with change in grit size.

**TABLE A1.4 Standard Plate Glass Specimens**

Material description	Calibrated plate glass standards (see 10.8 and 12.10)		
Procurement source	Libbey-Owens-Ford, Company Technical Center, 1701 East Broadway, Toledo, OH 43605		
Characteristics	Size - 4-3/8 by 4-3/8 by 1/4 in. (111 by 111 by 6.4 mm) Float glass, center tension between 313 and 362 psi (2157 and 2494 kPa) When plate glass is produced by the float process the side of the sheet in contact with the molten tin will show a somewhat higher stress (higher abrasion resistance) than the surface which has not been in contact with the tin. The higher stressed surface should <i>not</i> be used in the calibration and standardization tests (10.2, 10.8, and 11.7) since it is not the calibrated side of the glass. The side of the glass which has been in contact with the molten tin can be readily identified by the fact that it will fluoresce under ultraviolet light. See Typical Composition.		
Amount required for one test	6 Calibrated glass plates (see 10.8 and 12.10)		
Typical composition, % normal soda-lime plate glass	SiO <sub>2</sub>	73	
	Na <sub>2</sub> O	15.2	
	CaO	6.7	
	MgO	4.6	
	R <sub>2</sub> O <sub>3</sub> <sup>A</sup>	1.56	
	B <sub>2</sub> O <sub>3</sub>	0.03	
	K <sub>2</sub> O	0.05	
	Li <sub>2</sub> O	<0.01	

<sup>A</sup> R<sub>2</sub> O<sub>3</sub> includes <sub>2</sub> O<sub>3</sub>, Fe<sub>2</sub> O<sub>3</sub>, TiO<sub>2</sub>, and ZrO<sub>2</sub>.



## A2. ABRASION TESTER

A2.1 An abrasion tester that meets the requirements of 5.4 and 5.5 of this test method is shown in Fig. A2.1. The figure

vertical shaft which transmits to it the horizontal circular motion required to cause every point on each specimen to



1. Rubber-lined retaining ring with accessory hole for introducing abrasive charge. 2. Test specimen. 3. Phenolic or aluminum top. 4. Aluminum housing. 5. ON-OFF switch. 6. Selector switch. 7. Circuit breaker button. 8. Automatic timer.

**FIG. A2.1 Abrasion Tester with Retaining Rings and Test Specimens in Place**

shows the abrasion tester with test specimens and retaining rings clamped in place. The pertinent parts of the instrument are numbered and described as follows:

A2.1.1 Rubber-coated retaining rings (specified in 5.5), showing opening in the top for introducing the abrasive charge. Also shown is the method used to clamp the ring and the specimen to the top plate of the abrasion tester, forming a waterproof seal between ring and sample plate.

A2.1.2 Test specimen.

A2.1.3 Top plate of the tester. The plate is supported by a

describe a circle 0.875 in. (22.2 mm) in diameter.

A2.1.4 Aluminum housing containing the synchronous drive motor, sealed gear train, and cams required to rotate the top plate.

A2.1.5 ON-OFF toggle switch.

A2.1.6 MANUAL-AUTOMATIC selector switch.

A2.1.7 Circuit breaker button.

A2.1.8 Automatic timer.

A2.1.9 *Procurement Source*—Keystone Electric Co., 2807 Annapolis Road, Baltimore, MD 21230.

## A3. GLOSSMETER CHARACTERISTICS AS APPLIED TO THIS TEST METHOD

A3.1 The instruments that claimed precision of  $\pm 0.5$  gloss units can be improved in the upper ranges by frequent checking against the black (high-gloss) standard. Checks after every four readings (with correction if needed) increased the precision of the instrument to  $\pm 0.1$  unit.

A3.2 In a similar manner, read an intermediate (25-unit) standard after every four readings of the abraded plates, followed by the high gloss check. High gloss checks alone do not improve low gloss precision.

NOTE A3.1—Intermediate standards are subject to change in gloss value if they are mishandled, scratched, or even cleaned in a careless

manner. Glossmeters should therefore always be standardized by means of the black gloss standard. The intermediate (or series of intermediate) standard(s) should be used infrequently to check the long-term stability of the glossmeter. They should be handled as little as possible.

A3.3 Due to minor variations in internal geometry, the precision between instruments and between laboratories ranges from  $\pm 1$  to  $\pm 2$  gloss units. This can give rise to an 8 % variation between laboratories.

A3.4 In view of the above, differences between laboratories should be arbitrated by using the same glossmeter and standards in both laboratories, or by an independent laboratory.

A3.5 Surface abrasion will readily differentiate between different material types, but is not precise enough for control and most mill formula variations.

#### **A4. ABRASION INDICES FOR THE CURRENT BATCH OF STANDARD PLATE GLASS SPECIMENS.**

A4.1 Subsurface abrasion index as determined on the machine used as a standard, manufactured prior to July 1981: 4.5671.

A4.2 Subsurface abrasion index as determined on the machine used as a standard, manufactured after July 1981: 5.9000.

A4.3 Surface abrasion index (weight loss method) as

deter-mined on the machine used as the standard, manufactured prior to July 1981: 3.8948.

A4.4 Surface abrasion index (weight loss method) as determined on the machine used as the standard, manufactured after July 1981: 3.3666.

A4.5 Subsurface abrasion index (gloss loss method):53 %.

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