Designation: C 1203 – 91 (Reapproved 2002)

Standard Test Method for Quantitative Determination of Alkali Resistance of a Ceramic-Glass Enamel[1]

This standard is issued under the fixed designation C 1203; 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 This test method covers the determination of the resistance of a fired ceramic-glass enamel to a 10 % alkali solution held near its boiling point for 2 h.

1.2 This test method is not applicable to large tempered glass sheets or specimens larger than 9 by 9 cm.

1.3 *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.* For specific hazard statements, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

C 162 Terminology of Glass and Glass Products[2]

C 927 Test Method for Lead and Cadmium Extracted from the Lip and Rim Area of Glass Tumblers Externally Decorated with Ceramic Glass Enamels[2]

3. Terminology

3.1 *Definitions*—For additional definitions of terms refer to Terminology C 162.

3.1.1 *ceramic glass enamels (also glass enamels or ceramic enamels)*—predominantly colored, silicate-glass fluxes used to decorate glassware. **C 927**

3.1.2 *maturity—of a fired ceramic glass enamel*, a ceramic glass enamel has been fired to maturity when porosity of the ceramic glass enamel has been substantially eliminated, and the expected surface gloss of the fired ceramic glass enamel has been achieved.

3.1.2.1 *Discussion*—Refer to *GTA Engineering Standards Manual*, Section 4, D.3.3.1 and D.3.4.1, for testing criteria for maturity.[3]

4. Summary of Test Method

4.1 This test method measures the weight loss of a glass specimen decorated with a fired ceramic-glass enamel, when completely immersed in a 10 % alkali solution near its boiling point. A stainless steel beaker containing test samples and a 10 % alkali solution is heated at 95°C in a temperature controlled water bath for 2 h. Chemical dissolution of the ceramic-glass-enamel coating as well as any loss of weight due to the dissolution of the substrate is measured by determining weight loss of the specimen after exposure to the hot alkali solution. A substrate without ceramic-glass enamel is tested to determine the expected weight loss due to dissolution of the nondecorated side of the substrate.

5. Significance and Use

5.1 This test method is intended to be used when a quantitative measurement of the alkali durability of a ceramic-glass enamel is needed. The test is applicable to glass coated with fired enamels where exposure to strong alkalis, or alkalis at elevated temperatures might be encountered in service.

5.2 This test method is intended to be an accelerated, destructive test. Results can be used as an indicator of the relative durability of a particular enamel.

6. Interferences

6.1 The extent to which an enamel has been fired to maturity, as well as the residual stresses remaining after annealing of test specimen, can influence results.

6.2 Tests performed on tempered or heat-strengthened production ware should be done by annealing the chosen sheet before cutting the specimen.

7. Apparatus

7.1 *Balance or Scale*, accurate to 0.1 g, to weigh reagents.

7.2 *Balance*, accurate to 0.1 mg, to weigh specimens.

7.3 *Waterbath*, temperature controlled, capable of holding 95 ± 3°C.

7.4 *Glass Marking Scribe*.

7.5 *Beaker*, stainless steel, 1200-mL, with no pouring lip, as shown in Fig. 1.

7.6 *Cover*, stainless steel, for beaker.

7.7 *Sample Holder*, as shown in Fig. 1.

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[1] This test method is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.10 on Glass Decoration.

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[2] *Annual Book of ASTM Standards*, Vol 15.02.

[3] *GTA Engineering Standards Manual*, Glass Tempering Association, Topeka, KS, 1992.

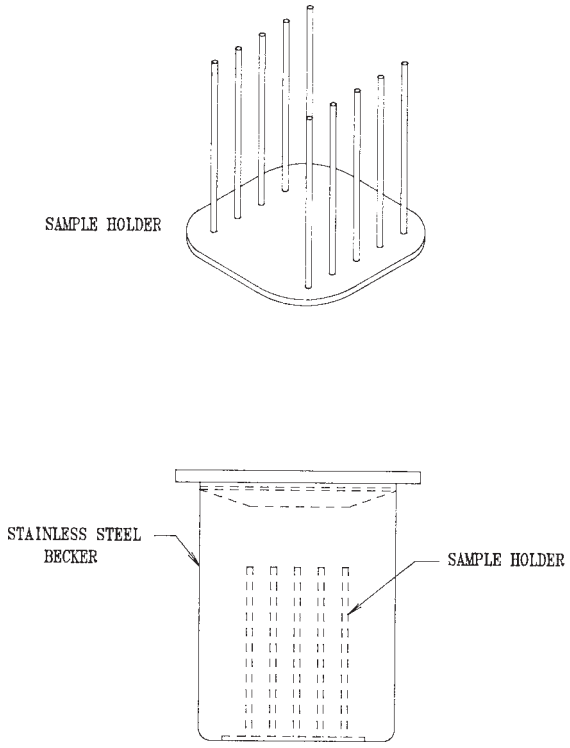

FIG. 1 Stainless Steel Beaker and Sample Holder

7.8 *Tongs*, heavy duty.
7.9 *Rubber Gloves*, chemically resistant.
7.10 *Apron or Lab Coat*, chemically resistant.
7.11 *Face Shield*.
7.12 *Fume Hood*, for ventilation.
7.13 *Cloth*, soft cotton.
7.14 *Caliper*, micrometer, accurate to 0.1 mm.
7.15 *Oven*, drying, capable of heating samples at 65 ± 5°C.

8. Reagents and Materials

8.1 *Alkali Solution*—Dissolve 70 g of sodium hydroxide (NaOH) in 630 mL of distilled water.

9. Hazards

9.1 Refer to the manufacturer's Material Safety Data Sheets for information regarding these materials.

9.2 A proper fume hood should be used when handling hot caustic solutions. Chemically resistant gloves, eye protection, and clothing should be worn, and tongs should be used. If accidental contact with caustic is encountered, or if ingestion occurs, seek medical attention immediately.

10. Test Specimen

10.1 The specimen to be tested should be glass decorated with ceramic glass enamel, and annealed to relieve stresses that could cause chipping or cracking during the test. Test a nondecorated specimen representative of the decorated substrate along with the decorated specimens as a control.

10.2 Cut a specimen with a diamond saw or glass cutter to a size that will easily fit into a 1200-mL, stainless steel beaker. Microscope slides which are 5 by 7.5 cm can be used. Similarly sized glass or sections of fired ware can also be used. The test specimen should be small enough for accurate weighing to the nearest 0.1 mg.

10.3 Bevel any sharp edges to avoid weight loss by chipping.

10.4 Mark the sample with a glass marking scribe for identification.

11. Procedure

11.1 Measure and record the length, width, and depth of each specimen to the nearest 0.1 mm. Record the initial weight of each specimen to the nearest 0.1 mg.

11.2 Place the specimens prepared as described in 10.1-10.4, in a sample holder such as shown in Fig. 1 so that the specimens are upright and separated from each other.

11.3 Prepare the alkali solution, as described in 8.1, in a 1200-mL stainless steel beaker with no pouring lip. This volume of solution is adequate for testing a maximum of 250 cm^2 of total specimen-decorated-surface area. A stainless steel cover should be used to cover the beaker.

11.4 Allow the alkali solution to reach normal room temperature or 25 ± 5°C. Using heavy-duty tongs, place the specimens and holder in this alkali solution. The test specimens must be completely immersed in the solution.

11.5 After placing the specimens into the solution, cover the beaker and place it in a temperature controlled water bath for 2 h at 95 ± 3°C. Hold the water bath at this temperature by using a solution of one part commercially available antifreeze to two parts water.

11.6 Monitor the temperature of the water bath during the test to assure that it does not exceed 98°C or drop below 92°C. Do not allow the alkali solution to boil.

11.7 After the test beaker has been heated for 2 h, remove it from the water bath.

11.8 Add tap water to the solution for 2 to 5 min to cool the specimens and flush the beaker.

11.9 Remove the specimens from the test beaker and wash them using tap water while vigorously scrubbing with a wet, soft, cotton cloth.

11.10 Rinse the specimens thoroughly with tap water, making sure that any lint is removed. Rinse next with distilled water and then dry in a drying oven at 65 ± 5°C for at least 15 min, or until dry.

11.11 Remove the specimens from the drying oven, and allow to cool to room temperature. Weigh the specimens to the nearest 0.1 mg.

12. Calculation

12.1 Calculate the total surface area, accounting for all six surfaces, and the decorated surface area of each specimen in square centimetres, rounded to two decimal places.

12.2 Calculate the weight loss for each decorated specimen, W_s, to the nearest 0.1 mg as follows:

$$W_s = W_i - W_f \quad (1)$$

where:
W_i = initial weight, mg, and
W_f = final weight, mg.

12.3 Calculate the areal weight loss, L_o, in grams per square centimetre for the nondecorated specimen as follows:

$$L_o = \frac{W_n}{A_o} \quad (2)$$

where:

W_n = weight loss of the nondecorated control sample, mg and

A_o = total surface area of the nondecorated control sample, cm^2.

12.4 Calculate the areal weight loss, L, in grams per square centimetre of the decorated area of each specimen, rounded to four decimal places as follows:

$$L = \frac{[W_s - L_o(A_t - A_d)]}{A_d} \quad (3)$$

where:

A_t = total surface area of the decorated specimen, cm^2, and

A_d = decorated surface area, cm^2.

13. Report

13.1 Report the following information:

13.1.1 The weight loss of the decorated area of the specimen, L, in grams per square centimetre (see 11.4).

14. Precision and Bias

14.1 No interlaboratory data are available at this time to determine experimental precision and bias. Committee C14 is planning to conduct round-robin testing to determine precision and bias.

15. Keywords

15.1 alkali; architectural; ceramic-glass enamel; glass enamel