



Standard Test Method for Sulfide Resistance of Ceramic Decorations on Glass¹

This standard is issued under the fixed designation C 777; 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 qualitative determination of the sulfide resistance of ceramic decorations on glass to assure the necessary durability of the decoration.

1.2 This test method provides an indication of performance when and if the decorations are exposed to sulfide attack.

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.*

1.4 This test method generates hydrogen sulfide gas which is highly poisonous.

2. Referenced Documents

2.1 *ASTM Standards:*

C 224 Practice for Sampling Glass Containers²

3. Summary of Test Method

3.1 One half of the specimens are immersed in a volume 4 % acetic acid solution that contains 1 mL of saturated sodium sulfide solution per each 100 mL of the acetic acid solution in a suitable covered container at room temperature. The degree of attack is determined by visual observation and physical testing of the specimen exposed to a hydrogen sulfide atmosphere.

4. Apparatus

4.1 *Suitable Covered Container.*

4.2 *Stirrer.*

4.3 *Hot Plate.*

4.4 *Pipet.*

4.5 *Fume Hood*, or other means of adequately exhausting H_2S produced during the test.

5. Reagents

5.1 *Sodium Sulfide* ($Na_2S \cdot 9H_2O$), reagent grade.

5.2 *Acetic Acid* (CH_3COOH , min 99.8 %), reagent grade.

6. Test Specimen

6.1 The decorated ware should be representative of the lot, or run, and should be taken in accordance with the principles stated in Practice C 224.

6.2 Standard reference ware of known resistance must be run with each test.

7. Preparation of Test Solution

7.1 Make a 4 % volume acetic acid solution by adding to each 96 mL of distilled water 4 mL of acetic acid.

7.2 Dissolve an excess amount of sodium sulfide in warm (35 to 40°C) distilled water. Make sure there is undissolved sodium sulfide left over. Cool to room temperature.

7.3 Stir into each 100 mL of the 4 % acetic acid solution 1 mL of saturated, clear, sodium sulfide solution for 2 or 3 min. A milk of sulfur will precipitate.

8. Procedure

8.1 Place the test specimens and the reference standard of known resistance into the test solution prepared according to 7.3 at room temperature so that only half of the decoration is immersed. Stir and cover.

8.2 Remove specimens from solution after 15 min and allow to air dry.

8.3 Note the degree of attack after 15 min by visual observation and grade.

8.4 Retain a duplicate piece of ware of each decoration under test without exposure as an aid in judging the degree of attack on the specimens under test.

9. Interpretation of Results

9.1 *Visual Inspection:*

9.1.1 Grade the decoration on the test specimen and reference standard in accordance with one of the following visual test grades:

9.1.2 *Visual Evaluation:*

Grade V1—No attack apparent.

Grade V2—Appearance of iridescence or visible stain on the exposed surface when viewed at a 45° angle but not apparent at angles less than 30°.

Grade V3—A definite stain which does not blur reflected images and is visible at angles less than 30°.

Grade V4—Definite stain with a gross color change or strongly iridescent surface visible at angles less than 30° and

¹ This test method is under the jurisdiction of ASTM Committee C-14 on Glass and Glass Products, and was written by Subcommittee C14.10 on Glass Decoration in cooperation with the Society of Glass Decorator's Committee A-20.

Current edition approved Nov. 15, 1993. Published January 1994. Originally published as C 777 – 74 T. Last previous edition C 777 – 93.

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

which may blur reflected images.

Grade V5—Surface dull or matte and grossly discolored.

9.2 Physical Testing of Stain:

9.2.1 If a stain exists, grade the decoration on the test specimen and reference standard in accordance with one of the following physical test grades:

9.2.2 Physical Testing of Stain:

Grade P1—Stain is easily removed by gently rubbing the hydrogen sulfide atmosphere exposed decoration with a dry paper towel.

Grade P2—Stain is not completely removed by gently rubbing the hydrogen sulfide atmosphere exposed decoration with a dry paper towel, but can be removed by vigorous rubbing.

Grade P3—Stain cannot be completely removed by vigorous rubbing with a dry paper towel.

10. Report

10.1 Report the following information:

10.1.1 Identification of the ware, the decorating material used, and the firing cycle.

10.1.2 Grading of the specimen and the reference standard.

10.1.3 Date of test and name of operator conducting test.

11. Precision and Bias

11.1 Precision of this test method will be investigated in a round robin planned by Subcommittee C14.10.

11.2 Because of the subjective nature of the grading, no justifiable statement can be made about the bias of this procedure.

12. Keywords

12.1 ceramic decorations; glass; sulfide resistance

The American Society for Testing and Materials 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 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, 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).