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Standard Test Method for Silver Tarnishing by Paper¹

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1. Scope

1.1 This test method covers the qualitative identification of the presence or absence of materials in paper that will tarnish or stain silver which comes in contact with the paper. This test method is chiefly intended for testing tissue papers that are in direct contact with silver. The test results indicate the type of tarnish or stain and the relative distribution of the materials causing such tarnishing or staining.

Note 1—The main material in paper causing silver to tarnish or stain is reducible sulfur, which may be determined in accordance with Test Method D 984.

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:

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product²

D 984 Test Methods for Reducible Sulfur in Paper²

D 1193 Specification for Reagent Water³

3. Summary of Test Method

- 3.1 A specimen of the sample is saturated with water and placed between two polished silver plates. The specimen is allowed to dry in contact with the plates at a temperature of 70°C for at least 3 h. At the end of the drying period, the plates are examined for evidence of tarnish or stain.
- 3.2 Water repellent or parchmentized papers and metalcoated papers will require certain modifications of the test procedure. In most cases, they may be tested without wetting the test specimen and by prolonging the time in contact with the silver to at least 24 h.
- 3.3 The principles involved in this test method are also suggested for use where wrapping paper is to be tested for its

staining or corrosion properties with respect to other metals, such as steel, copper, nickel, etc. In such cases, plates or forms of the particular metal or alloy in question shall be used in place of silver plates. Other modifications may be required for a specific purpose. For example, in the case of a metal that would rust, such as steel, the paper obviously could not be wetted; it would be necessary instead to heat in an atmosphere of high humidity.

3.4 Modifications to accommodate paperboard are also possible. One such modification is described in the Appendix.

4. Significance and Use

- 4.1 This test method covers the determination of the presence or absence of materials that will tarnish or stain silver in paper used for wrapping silverware. Freedom from tarnishing silver is an important end user requirement of such papers.
- 4.2 Possible modifications to other metals have specialized applications in metal plating, graphic arts, and related areas.

5. Apparatus

- 5.1 *Metal Surface*,⁴ solid silver or silver plated brass flat plates, approximately 15 by 15 cm (6 by 6 in.). Two plates are needed for each test and are probably sufficient for occasional tests of paper. Several sets are necessary where frequent testing is desired.
- 5.1.1 *Solid Silver Plates*, approximately 15 by 15 cm (6 by 6 in.).
- 5.1.2 Metal Plates—Flat plates approximately 15 by 15 cm (6 by 6 in.) from commercial rolled sheet brass 0.25 in. (6.35 mm) thick. One face of each plate should be buffed smooth and as free as possible from tool marks, then plated with pure silver to a thickness of about 0.125 mm, buffed again, and plated with another 0.125 mm of silver. In this way, a smooth continuous layer of pure silver about 0.25 mm thick will be obtained that can be cleaned and polished repeatedly. It is advisable to check the amount of silver by weighing the brass plates before and after plating. A layer of silver 0.25 mm thick should weigh

¹ This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

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² Annual Book of ASTM Standards, Vol 15.09.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ The metal surface required is not a stock item in many laboratory supply organizations, however the solid silver plates in 5.1.1 may be obtained upon order from some dealers in fine and precious metals. One such source is Hagstoz & Son, 709 Sampson St., Philadelphia, PA 19106, who can supply the plates in 5.1.1 in several thicknesses. There are other similar sources. The plates in 5.1.2 can be obtained by purchasing the brass plates as described and having them plated with silver to the specification in 5.1.2 by a commercial silver-plater.

about 0.25 g/cm². If the brass plates are plated with pure silver on both sides, one side of each plate will serve as a "blank" during the test, indicating whether the air in the oven will tarnish silver or not. The use of plates coated on both sides with silver (or solid silver plates) will obviate the use of extra strips of silver foil called for in the procedure.

- 5.2 Oven—A temperature-controlled air oven of the gravity-convection type to maintain a temperature of $70 \pm 5^{\circ}$ C.
 - 5.3 Forceps, wooden or plaster, for holding test specimens.

6. Reagents and Materials

- 6.1 Silver Polish—A good grade of silver-polishing powder or paste that will not scratch pure silver and contains no cyanides or protective-type coatings such as silicone. A paste composed of the finest grade of Tripoli powder, such as used for polishing metals for metallographic work, and distilled water containing a small amount of ammonium hydroxide (NH₄OH) will clean silver completely.
- 6.2 *Water*—Unless indicated otherwise, references to water shall be understood to mean reagent water as defined by Type I or II of Specification D 1193.
 - 6.3 Silver Foil.

7. Test Specimen

7.1 The material shall be sampled in accordance with Test Methods D 585. Do not touch the areas to be tested with the fingers. Instead use wooden or plastic forceps to protect the sample from atmospheric contamination. Select from each test unit of the sample at least five representative test specimens, each about 160 mm² (6.5 in.²).

8. Procedure

8.1 Thoroughly clean the silver surface of each plate and polish to a high luster. Then rinse the plate with alcohol followed by ether or 1,1,1-trichloroethane and dry. Holding one of the 160-mm square test specimens by means of wooden or plastic forceps, wet it with water as uniformly as possible, so that the paper is saturated but not dripping wet. This can be readily done by means of an all-glass atomizer operated by compressed air (not a rubber bulb) or by steaming over the outlet of a wash bottle that contains boiling water. As soon as the test specimen is saturated with water, place it on the polished silver surface of one plate, taking care to have as few

wrinkles or air bubbles as possible. Then lay the other plate, silver surface down, upon the paper.

8.2 Place the test assembly in the air oven, protect from access to laboratory fumes, and adjust the temperature so that the paper will dry completely in not less than 3 h (about 65 to 75°C; not more than 80°C).

Note 2—Wherever there is any doubt regarding the presence of sulfur compounds in the air, it is advisable to hang one or more small strips of clean, highly polished, pure silver foil in the oven while the test specimens are there. If tarnishing or staining of these strips occurs, the air entering the oven should be purified, or the oven should be placed where the air causes no tarnishing of the silver.

Note 3—Papers other than tissue require a longer time to dry. Paperboard 3 to 6 mm thick may require 48 h or more. Consequently, thick specimens should not be too wet when placed between the test plates. If paperboard is dried too rapidly, it frequently buckles sufficiently to lift the test plates out of contact with its surface. It may be necessary to clamp the test plates together with C clamps after inserting the wetted test specimen between them.

8.3 When the paper is dry, examine the silvered surface of each plate that was in contact with the specimen for evidence of tarnishing or staining. Note the color and the character of any stains found. If isolated spotting occurs, estimate the approximate size and count the number of the spots on each plate.

Note 4—A permanent record of the results may be made for future reference by placing tracing paper over the plate after the test and tracing the spots or stained areas. A photograph also provides an acceptable permanent record.

9. Report

- 9.1 Report the following information:
- 9.1.1 Number of specimens tested,
- 9.1.2 Color and character of tarnishing or staining, if any (whether spots, large areas, etc.) (see 8.3 and Note 4), and
- 9.1.3 Number of tarnished or stained areas, reported as maximum and minimum number found in any one test specimen and the total number found in all pieces tested.

10. Precision and Bias

10.1 No statement is made about the precision or bias of this test method since the results are simply a qualitative summary of tarnishing occurring under the test conditions specified in the test method.

APPENDIX

(Nonmandatory Information)

X1. SUGGESTED MODIFICATION FOR PAPERBOARDS

- X1.1 Heat a desiccator or glass container in a 38°C oven for at least 30 min. Interior surface area must be at least large enough to accommodate the metal test surface (6 by 6 in.).
- X1.2 Place the specimens between the silver plates using wooden or plastic forceps, building up a stack of five or six of these sandwiches with the top and bottom layers containing no specimens.
- X1.3 Place this stack on the desiccator stand in the glass container or desiccator. Place 12 to 15 mm ($\frac{1}{2}$ in.) of distilled water in the container. The stand should prevent the samples from actually coming into direct contact with the water.
 - X1.4 Place an approximately 2.5-kg weight (which has

been carefully covered with several layers of aluminum foil) on top of the specimen stack. A good contact between sample and the plate throughout the entire test must be assured. Use a small amount of stopcock grease to seal the cover to the test specimen container or desiccator.

- X1.5 Heat the complete test apparatus at 38° C ($100 \pm 3^{\circ}$ F) for 24 h. By using this closed system, a relatively pure, highly humid air source is consistently available. This reduces the possibility of laboratory air contaminating the test.
- X1.6 When the test is complete, remove the specimens and plates from the desiccator. Examine the plates as described in 8.3. Report data as described in Section 9.

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