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T _____ 444 _____

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WORKING GROUP
CHAIRMAN _____ Chris Czyryca _____

SUBJECT
CATEGORY _____ Physical Properties _____

RELATED
METHODS _____ See "Additional Information" _____

CAUTION:

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

**Silver tarnishing by paper and paperboard
(Five-year review of T 444 om-12)
(Changes from Draft 1 incorporated)
(Reclassified as a Standard Practice on Ballot 2)
(New classification: T 444 sp-XX)**

1. Scope

1.1 This practice is for identifying papers and boards that will tarnish or stain metal plates. The appearance of the tarnish, stain, or corrosion of metal by a test specimen of the sample is reported, together with the distribution of the tarnishing or staining. Reducible sulfur activity is a common cause of tarnishing.

1.2 This practice was formerly an Official Method specific to silver tarnishing, with other types of metal plates referenced as modifications. This practice expands the scope of the testing and clearly indicates that the procedure is intended to be a qualitative evaluation.

1.3 Certain sample types, such as water repellent or metal-coated papers, or board may require modification of the test procedure. In most cases, water repellent or metal-coated samples may be tested without wetting the

test specimen and by prolonging the time in contact between the sample and plate to at least 24 h. When such sample types are tested, state any modification of the practice in the report.

1.4 The principles involved in this standard practice may be used to test paper samples for the tarnish, stain, or corrosion properties with respect to a variety of metals, such as steel, copper, nickel, brass, etc. It is necessary to use plates or forms of the particular metal or alloy in question and to make other modifications as may be required for the particular purpose. For example, in the case of metal that would rust, the paper might not be wetted; it could instead be heated in an atmosphere of high humidity.

1.4 Specific procedures for testing for tarnishing, staining, or corroding paper samples are not included in this standard practice. This practice describes the procedural framework and the required included information of a report generated by employing this practice.

2. Significance

This test method provides a qualitative method to determine if paper or paperboard will cause tarnishing of metal surfaces in contact with the grade of paper examined.

3. Apparatus

3.1 *Metal plates*, solid flat plates, with area approximately 15 cm × 15 cm (6 in. × 6 in.) and between 6mm and 17mm thick. The mass of the plates must be sufficient to ensure full and consistent contact between the specimen and plate. Two plates are required for each test. Silver or silver plated, brass plates were specified in the Official Method version of this procedure, and are described in Section 3.1.1. Metal plates used must be composed of a known and reported alloy or plated with a known and reported alloy. Eight nominally identical plates are required to conduct this procedure. Two plates are sufficient for occasional tests of paper, using the alternate procedure for papers less than 160 g/m².

3.1.1 *Silver plated brass plates*, produced as follows: commercial rolled-sheet brass 6.35 mm (0.25 in.) thick is buffed smooth and as free from tool marks as practical, then plated with pure silver to a thickness of about 0.125 mm (0.005 in.), buffed again, and replated with another 0.125 mm of pure silver. A smooth, continuous layer of pure silver about 0.25 mm (0.010 in.) thick, which can be repeatedly cleaned and polished, should be obtained. To determine the amount of plated silver, weigh the brass plate before and after plating. A layer of silver 0.25 mm thick weighs about 2.5 kg/m². The brass plates may be plated on one or both sides.

3.2 *Oven*, forced draft, capable of maintaining a temperature of 38 ± 2°C about (100°F) or 70 ± 5°C (about 150°F) protected by isolation from laboratory fumes.

3.3 *Desiccator*, glass, or other container with a tightly fitting cover, and ceramic or glass stand.

3.4 *Forceps*, wooden or plastic.

3.5 *Weight*, approximately 2.5 kg (5 lb).

4. Reagents and materials

4.1 *Metal polish*, a polishing powder or paste, which will not scratch the surface of the plates and does not contain cyanides or protective coatings such as silicone. A paste made up of the finest grade of Tripoli powder, such as that used for polishing metals for metallographic work, and distilled water containing a small amount of NH_4OH is recommended. The polish used shall be noted in the report.

4.1.1 Tripoli powder is the common name for particulate SiO_2 used in polishes for a wide range of materials including wood, “rocks,” and metal surfaces. Tripoli powder comes in a wide range of particle sizes and purities between about 90% and 99%, major impurities being other oxides such as aluminum, calcium, and iron. The use of Tripoli powder in this test dates from the origination of the procedure in 1939, and there is no added information on the use of Tripoli powder for polishing the plates used than that in section 4.1. While a Tripoli powder paste can be prepared, commercial metal polishes are quite suitable, but the requirement that they not leave any cyanides or protective coating on the silver is critical. Any material deposited on the plate by the polish that would coat it in any way would eliminate its use in a test for tarnishing.

4.2 *Water*, distilled or deionized.

4.3 *Silver foil*

4.4 *Aluminum foil*, area to contact test specimen must be cleaned free of oil by wiping with clean cotton or lens paper soaked in a suitable solvent.

4.5 *Solvent*, a sulfur-free laboratory solvent to clean and remove residue from the test plates. Commercial metal cleaner, acetone, ethyl acetate, or a mixture of 50/50 acetone/isopropanol may be used. The solvent historically specified in the Official Method was 1, 1, 1-trichloroethane. **SAFETY CAUTION:** 1, 1, 1-trichloroethane represents a significant health and environmental hazardous material and should not be utilized. The solvent used shall be noted in the report.

5. Test specimen

5.1 Obtain the sample in accordance with TAPPI T 400 “Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, or Related Product.” Do not touch the areas to be tested with the fingers; instead, use clean forceps and protect the sample from contamination which may be in the atmosphere.

5.1.1 Whenever 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 silver foil in the laboratory in which the test pieces are prepared, and in the oven with the test specimens. 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.

5.2 From the sample, select at least five representative test specimens, each about 160 mm (6.5 in.) square.

6. Procedure

6.1 Thoroughly clean the surface of each plate and polish it to a high luster, then rinse the plate with alcohol, rerinse with a solvent, that does not leave a residue, and dry, first in the laboratory, and finally in the oven.

SAFETY CAUTION: Never place the plates in the oven until all traces of any organic solvent, some of which are flammable or explosive, have evaporated from the plates. Generally, drying the plates with a soft absorbent tissue, and then allowing the plates to remain in the laboratory air (see 5.1.1) for 60 minutes should be sufficient to remove solvent traces. When in doubt, allow the plates to dry for a longer time prior to placing in the oven.

6.2 *For paperboard and papers greater than 160 g/m².*

6.2.1 Heat the dessicator or glass container in the 38°C (about 100°F) oven for at least 1/2 hour.

6.2.2 Place the specimens between the plates, building up a stack of five or six of these sandwiches with the top and bottom layers containing no specimen.

6.2.3 Place this stack on the dessicator stand in the glass container or dessicator. Place 12-15 mm (¹/₂ in.) of distilled water in the container. The stand should prevent the samples from actually coming into direct contact with the water.

6.2.4 Place an approximately 2.5-kg weight (which has been carefully covered with several layers of aluminum foil) atop 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 dessicator.

6.2.5 Heat the complete test apparatus at 38°C about (100°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. See 5.1.1.

6.2.6 When the specimen is dry, examine the silvered surface of each plate for tarnishing or staining. Record the color and character of the stains. A digital image (digital photograph) is quite suitable for making this record, although a drawing may be made as part of the sample record. Alternately, a permanent record may be made for future reference by placing tracing paper over the plate after the test and tracing the spots or stained areas. If isolated spotting occurs, record the approximate size of the spots and count their number on each plate.

7. Report

Report the number of specimens tested; the color and character (e.g. spots, large areas, etc.) of tarnishing or staining, if any, and the number of tarnished or stained areas, as the maximum and minimum number found in any one test specimen and the total number found in all specimens tested. State if alternative cleaning procedures and/or chemicals were used.

8. Precision

No precision statement has been determined due to the qualitative nature of the determination of the results of the procedure.

9. Keywords

Paper, Paperboard, Metal tarnish tests, Reducible sulfur

10. Additional information

10.1 Effective date of issue: to be assigned.

10.2 See also TAPPI T 406 “Reducible Sulfur in Paper and Paperboard.”

10.3 The 2021 revision of this practice contained major revisions: the procedure was reclassified from an Official Method to a Standard Practice, the use of the solvent 1, 1, 1-trichloroethane was removed as unsuitable with laboratory safety, and the procedure was consolidated to reflect only the multiple-ply procedure.

10.4 Changes in the 2011 revision are editorial and include: safety precautions for use if working with certain organic solvents, moving certain “additional information” into the body of the document, removal of reference to a withdrawn ASTM method, moving the requirement for performing the method in the absence of certain air pollutants from a note to a numbered clause, addition of missing equipment (a 70°C oven), and other minor organizational changes.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■