



Standard Practice for Preparation of Uniform Free Films of Organic Coatings¹

This standard is issued under the fixed designation D 4708; 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 practice covers the preparation of free films of organic coatings for use in determining the physical properties of the coatings. Procedures are given for preparing free films on four alternative substrates. These substrates are dental foil, treated FEP (fluorinated ethylene-propylene) sheet, silicone coated paper, and halo-silane coated glass plates.

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.* A specific hazard statement is given in 6.1.

2. Referenced Documents

2.1 ASTM Standards:

- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1653 Test Methods for Water Vapor Transmission of Organic Coating Films²
- D 2370 Test Method for Tensile Properties of Organic Coatings²

3. Summary of Test Method

3.1 Free films are prepared by depositing a uniform wet coating of the test material on a release substrate. The applied films are dried or baked, cut into appropriate size for the intended physical property test, and then stripped from the release substrate.

4. Significance and Use

4.1 Free films are required for conducting tests to evaluate physical properties such as tensile and elongation (Test Method D 2370), moisture vapor permeability (Test Methods D 1653), and other physical properties of organic coatings where the substrate may interfere with the determination.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

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² *Annual Book of ASTM Standards*, Vol 06.01.

4.2 The tin foil/mercury amalgamation procedure should be used only in cases where other substrates may be affected by high-temperature baking or may affect the test results.

5. Apparatus and Materials

5.1 *Equipment*, for applying films of uniform thickness as described in Test Methods D 823.

5.2 *Micrometer Film Thickness Gage*, as described in Test Methods D 1005.

5.3 *Alternative Release Substrates*:

5.3.1 *Dental Tin Foil*, preferably 1-mil (25- μ m) thick.

5.3.2 *Sheet of FEP—(polyhexafluoropropylene)*,³ preferably 2-mils (50- μ m) thick, coated with a thin film of a dry lubricant.⁴

5.3.3 *Sheet of Silicone Coated Paper*, preferably 5-mil (125- μ m) thick.⁵

5.3.4 *Glass Plates*, coated with halosilane compound.

NOTE 1—Other substrates that may be suitable are 10-mil (250- μ m) thick polyethylene, photographic paper, polished steel, and fluoropolymer coated metal panels.

5.4 *Precision Specimen Cutter*, having a double blade with a foot to hold the sample.⁶

6. Hazards

6.1 *Mercury*—This practice involves the use of an OSHA-designated hazardous substance, mercury. For information on

³ Teflon FEP 2-mil film thickness (Card No. 03111, Item #29499) from E.I. du Pont de Nemours & Co., Inc., Wilmington, DE 19898, was found suitable for this purpose. However, it is no longer available. Other substrates that may be suitable are 10-mil (250- μ m) thick polyethylene, photographic paper, polished steel, and fluoropolymer coated metal panels.

⁴ The sole source of supply of dry lubricant (MS-122 Fluorocarbon Release Agent) known to the committee at this time is Miller-Stephenson Chemical Co., Inc., 55 Backus Ave., Danbury, CT 06810. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁵ The sole source of supply of silicone coated release paper, Form RP-1K, size 8% by 11¼ in., known to the committee at this time is the Leneta Co., 15 Whitney Rd., Mahwah, NJ 07430. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁶ The sole source of supply of the JDC Precision Sample Cutter, known to the committee at this time is the Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, PA 19154. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

the potential hazards and guidance relative to use, consult the supplier's Material Safety Data Sheet. Mercury is a toxic metallic liquid. Its vapors are extremely hazardous. Small amounts of spilled mercury can vaporize sufficiently at room temperature to exceed the TVL of the vapor. Use with adequate ventilation (in a hood) or wear a respirator, and clean up spills immediately. Wear gloves when handling mercury. Containers should be kept closed. Droplets of mercury can be picked up by using a small glass pipet connected to a suction flask with a rubber hose.

7. Procedure

7.1 Prepare the free films by one of the following procedures:

7.1.1 Dental Tin Foil Substrate:

7.1.1.1 Mount the uncoated foil on glass plate using a rubber squeegee to smooth the foil. If the foil will not stay in place, use a small amount of a high-boiling liquid between the foil and glass plate to increase adhesion of foil to the plate. Most films with a dry-film thickness of less than 2 mils (50 μm) are very difficult to handle. Apply a uniform wet film of the material to the foil by one of the procedures given in Test Methods D 823.

NOTE 2—Use a liquid that is known to have little or no effect on the test coating. In many cases a light mineral oil would be suitable.

NOTE 3—**Caution:** Exercise care at all times to prevent the high-boiling liquids used in mounting the foil from coming in contact with the test film as these liquids may soften the film. For instance, the foil should be cleaned before amalgamation since any liquid remaining on the back of the foil and the test film will float on the bath of mercury.

7.1.1.2 Dry the applied films at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, or bake under conditions mutually agreeable to the producer and the user. Dry film thicknesses must not vary by more than the specified or agreed-upon tolerance of the average film thickness and must not contain visible flaws. Measure the dry-film thickness nondestructively. Test Method D 1005 is suitable for this measurement.

7.1.1.3 When required for further treatment such as weathering, remount the coated foil on a convenient smooth, flat substrate such as a glass or steel panel. In this case, the high boiling liquid (see Note 2) may be used to hold the foil in close contact with the substrate over extended periods of time.

7.1.1.4 Age or expose the coated foil to conditions mutually agreed upon. At the end of the specified or agreed upon period, remove the foil-backed film from the glass or metal substrate and cut the specimens to size using a sharp knife or a precision specimen cutter.

NOTE 4—To prepare specimens for tensile property tests a precision specimen cutter must be used to ensure nick-free edges on the specimens. Even with this instrument, it is necessary to cut each specimen indepen-

dently, allowing at least $\frac{1}{2}$ in. (13 mm) of waste between specimens.

7.1.1.5 Place the foil-backed specimen film side up on a pool of mercury (see Note 3) to remove the foil by amalgamation. After amalgamation is completed, pick up an end of the floating film with tweezers, and carefully brush the unsupported film free of mercury and amalgam with a soft camel hair brush.

7.1.2 FEP Substrate:

7.1.2.1 Apply each material to be tested to a sheet of FEP that has been treated as follows: First, cover a smooth, flat polished glass plate with a sheet of FEP. Coat the sheet uniformly with a light coat of a dry fluorocarbon lubricant and allow to dry for 24 h at standard conditions. Then apply a uniform wet coating of the test material on the sheet by one of the procedures given in Test Methods D 823.

7.1.2.2 Dry the applied films in $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ humidity, or bake under conditions mutually agreeable to the producer and the user. Dry film thicknesses must not vary by more than the specified or agreed upon tolerances of the average film thickness and must be free of visible flaws. Most films with a thickness of less than 2.0 mil (50 μm) are very difficult to handle.

7.1.2.3 When required for further treatment such as weathering, remount the coated sheet on a convenient smooth, flat substrate such as a glass or steel panel.

7.1.2.4 Age or expose the coated sheet to conditions mutually agreed upon. At the end of the aging or exposure period, remove the sheet-backed film from the glass or metal substrate and cut the specimens to size using a sharp knife or the precision cutter (Note 4). Carefully strip the coating film from the sheet substrate.

7.1.3 Silicone Coated Paper:

7.1.3.1 Apply each material to a sheet of silicone coated paper. First, cover a smooth, flat substrate with a sheet of a silicone-coated release paper. Then apply a film of the material under test, dry or cure, expose or treat if required, and prepare test specimens as described in 7.1.2.

7.1.4 *Halosilane Coated Glass Plates*—Apply each material to a glass plate coated with a halosilane compound. First, coat a glass plate with a thin film of a halosilane compound and dry. Then apply a film of the material under test, dry or cure, expose or treat if required, and prepare test specimens as described in 7.1.2.

8. Keywords

8.1 dental tin foil substrate; film; halosilane coated glass plates; lacquer; organic coatings; paints and related coatings; polyhexafluoropylene (FEP) substrate; silicone-coated paper; substrates; surface preparation; tension (tensile) properties/tests; vapors; varnishes

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