**Designation: D 5095 – 91**

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# Standard Test Method for Determination of the Nonvolatile Content in Silanes, Siloxanes and Silane-Siloxane Blends Used in Masonry Water Repellent Treatments[1]

## 1. Scope

1.1 This test method describes a procedure for the determination of the nonvolatile content ($N_s$) of silanes, siloxanes, and blended silane-siloxane materials used in masonry water repellent treatments and is applicable to both solvent- and water-borne materials.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 7.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials[3]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[4]

## 3. Summary of Test Method

3.1 A designated quantity of test material is weighed into a preweighed aluminum dish containing the catalyst solution, mixed, allowed to stand for 60 min at room temperature, and then heated in an oven at 110 ± 5°C for 60 min. The nonvolatile content of the test material is calculated by subtracting the solids of the catalyst solution from the total solids by weight of the test solution.

## 4. Significance and Use

4.1 This test method is used to determine the nonvolatile content of silanes, siloxanes, and silane-siloxane blended materials used in masonry water-repellent treatments. It can be used for the purpose of calculating the volatile organic compound (VOC) content of these materials under specified test conditions.

## 5. Apparatus

5.1 *Aluminum Dishes*, 58-mm diameter by 18-mm high with a smooth (planar) bottom surface. Precondition the dishes for 30 min in an oven at 110 ± 5°C and store in a desiccator prior to use.

5.2 *Forced Draft Oven*, Type IIA or Type IIB as specified by Specification E 145.

5.3 *Syringes*, 1-mL and 5-mL.

5.4 *Analytical Balance*, capable of weighing to 0.1 mg.

## 6. Reagents

6.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

6.2 *p-Toluenesulfonic Acid*, monohydrate.[5]

6.3 *Alcohol*, technical grade ethanol or isopropanol.

## 7. Hazards

7.1 In addition to other precautions, provide adequate ventilation, consistent with accepted laboratory practice, to limit the accumulation of solvent vapors.

## 8. Procedure

8.1 *Catalyst Solution*—Prepare a catalyst solution containing a mixture of 0.5 % *p*-Toluenesulfonic acid in either ethanol or isopropanol. Thoroughly mix the solution. Prepare sufficient catalyst solution to perform all tests.

8.1.1 The nonvolatile content of the test material can be calculated only if the same catalyst solution is used throughout the test. Each time a new batch of catalyst solution is used, its nonvolatile content must be determined.

8.2 Determine the nonvolatile matter, in triplicate, of the catalyst solution as follows:

8.2.1 Weigh an aluminum dish to 0.1 mg. Record the weight as $W_1$.

8.2.2 Using a 5-mL syringe, weigh 3 ± 1.0 g, to 0.1 mg, by difference, of the catalyst solution into the preweighed aluminum dish. Record the weight of catalyst solution as $W_3$.

8.2.3 Heat the aluminum dish containing the catalyst solution in a forced draft oven for 60 min at 110 ± 5°C.

8.2.4 Remove the dish from the oven and immediately place in a desiccator. Seal the desiccator and allow the dish to cool to ambient temperature.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.47 on Masonry Treatments.

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[2] *Annual Book of ASTM Standards*, Vols 6.01 and 11.01.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Solutions of *p*-Toluenesulfonic acid in isopropanol may be obtained from King Industries, Science Road, Norwalk, CT 06852.

8.2.5 Reweigh the dish to 0.1 mg. Record the weight as $W_2$.

8.2.6 Calculate the nonvolatile matter of the catalyst solution, $N_c$, in accordance with 9.1.

8.3 Determine the percent nonvolatile content, in triplicate, of the test specimen as follows:

8.3.1 Thoroughly mix the test materials before use.

8.3.2 Weigh an aluminum dish to 0.1 mg. Record the weight as $W_4$.

8.3.3 Using a 5-mL syringe, weigh 3 ± 1.0 g, to 0.1 mg, by difference, of the catalyst solution into the preweighed aluminum dish. Record the weight as $W_6$.

8.3.4 Using a 1-mL syringe, weigh 1.0 ± 0.1 g, to 0.1 mg, by difference, of the test specimen into the weighing dish containing the catalyst solution. Record the weight of the test specimen as $S$.

8.3.4.1 Weighings must be done quickly to limit weight loss due to volatilization. If there is insufficient moisture present when testing solvent-borne silane materials, it is advisable to add up to 0.3 g of reagent grade water to the dish containing the mixture of catalyst and test solutions.

8.3.5 Gently swirl the dish to mix the materials. Allow the materials to stand at room temperature for 60 min.

8.3.6 Heat the dish containing the mixture of catalyst and test solutions in a forced-draft oven for 60 min at 110 ± 5°C.

8.3.7 Remove the dish from the oven and immediately place in a desiccator. Seal the desiccator and allow the dish to cool to ambient temperature.

8.3.8 Reweigh the aluminum dish to 0.1 mg and record the weight as $W_5$.

8.3.9 Calculate the nonvolatile content, $N_s$, of the test specimen in accordance with 9.2.

## 9. Calculation

9.1 Calculate the nonvolatile matter, $N_c$, in the catalyst solution as follows:

$$N_c = (W_2 - W_1)/W_3 \tag{1}$$

where:

$N_c$ = nonvolatile matter of catalyst solution expressed as a decimal fraction,
$W_1$ = weight of aluminum dish, g,
$W_2$ = weight of dish plus catalyst solution after heating, g, and
$W_3$ = weight of catalyst solution before heating, g.

9.2 Calculate the nonvolatile content, $N_s$, in the test specimen as follows:

$$N_s = 100\,\frac{[(W_5 - W_4) - (W_6)(N_c)]}{S} \tag{2}$$

where:

$N_s$ = nonvolatile content of test specimen, percent,
$W_4$ = weight of the aluminum dish, g,
$W_5$ = weight of aluminum dish plus the catalyzed test material after heating, g,
$W_6$ = weight of catalyst solution used in test specimen before heating, g,
$N_c$ = nonvolatile matter of catalyst solution, decimal fraction, (average of two determinations), and
$S$ = weight of test specimen before heating, g.

## 10. Report

10.1 Report the following information:

10.1.1 The average values obtained for the nonvolatile content of the catalyst solution, $N_c$.

10.1.2 The average values obtained for the percent nonvolatile content of the test specimen, $N_s$.

## 11. Precision and Bias

11.1 The precision estimated for this test method is based on an interlaboratory study in which one operator in each of five laboratories tested in triplicate on two different days four water repellent materials containing between 14 to 65 % nonvolatiles. The results were analyzed statistically in accordance with Practice D 3980. The intralaboratory standard deviation was found to be 0.283 % absolute with 17 df and the interlaboratory coefficient of variation 7.02 % relative with 16 df. Based on the standard deviation and coefficient of variation, the following criteria should be used for judging at the 95 % confidence level, the acceptability of results.

11.1.1 *Repeatability*—Two results, each the mean of triplicates, obtained by the same operator should be considered suspect if they differ by more than 0.84 % absolute.

11.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 7.02 % relative.

11.2 *Bias*—Bias has not been established for this test method.

## 12. Keywords

12.1 masonry water repellents; nonvolatile matter content; silanes; siloxanes

**Designation: D 5098 - 92**

# Standard Specification for Artists' Acrylic Emulsion Paints[1]

This standard is issued under the fixed designation D 5098; 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 specification establishes requirements for composition, physical properties, performance, and labeling of artists' acrylic emulsion paints.

1.2 This specification covers pigments, vehicles, and additives. Requirements are included for pigment identification, lightfastness, consistency, and drying time.

1.3 Table 1 lists some pigments meeting the lightfastness requirements in this specification. In order to identify other pigments that meet these requirements, instructions are given for test specimen preparation. Test methods for determining relative lightfastness are referenced.

1.4 The values stated in inch-pound units are to be regarded as standard. The SI units given in parentheses are for information only.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints[2]

D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller[3]

D 476 Specification for Titanium Dioxide Pigments[2]

D 602 Specification for Barium Sulfate Pigments[2]

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems[3]

D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature[2]

D 3168 Practice for Qualitative Identification of Polymers in Emulsion Paints[3]

D 4236 Practice for Labeling Art Materials for Chronic Health Hazards[4]

D 4303 Test Methods for Lightfastness of Pigments Used in Artists' Paints[4]

D 4838 Test Method for Determining the Relative Tinting Strength of Chromatic Paints[4]

E 284 Terminology of Appearance[3]

## 3. Terminology

3.1 *Definitions:*

3.1.1 *colour index name*—consists of the category (type of dye or pigment), general hue, and an assigned number given to a colorant in the Colour Index[5] as an international identification system.

3.1.1.1 *Discussion*—For example, the Colour Index Name of one phthalocyanine blue pigment is Pigment Blue 15 (PB 15).

3.1.2 *Colour Index Number*—a five-digit number given in the Colour Index that describes the chemical constitution of a colorant.

3.1.2.1 *Discussion*—For example, the Colour Index Number of one phthalocyanine blue pigment is 74160.

3.1.3 Appearance terms used in this specification are defined in Terminology E 284.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *acrylic emulsion paint*—paint containing a stable aqueous dispersion of polymers or copolymers of acrylic acid, methacrylic acid, esters of these acids, or acrylonitrile; sometimes termed latex, acrylic latex, or polymer emulsion paint.

3.2.2 *glycols*—general term for dihydric alcohols used to provide freeze-thaw stability in acrylic and other water-based vehicle systems.

## 4. Significance and Use

4.1 This specification establishes quality requirements and provides a basis for common understanding among producers, distributors, and users.

4.2 It is not intended that all paints meeting the requirements be identical nor of uniform excellence in all respects. Variations in manufacture, not covered by this specification, may cause some artists to prefer one brand over another, either of which may be acceptable under this specification.

## 5. Labeling Requirements

5.1 *Pigment(s) Identification:*

5.1.1 Every label shall include for each pigment contained in the paint the information underlined in Table 1 which includes the Common Name, Colour Index Name, and any additional terms necessary to identify the form of the pigment.

5.1.2 The complete pigment identification given in Table 1, which also includes the Colour Index Number and a simple chemical description, shall be given in an appropriate producer publication. Manufacturers are encouraged to put this complete identification on the container label when label size permits.

5.1.3 The Common Name shall be placed on the front of the label and shall be the name of the paint except as

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.02.

[5] *Colour Index*, 3rd ed., 5 Vols and Revisions. The Society of Dyers and Colourists, London, 1971–75. Available from the American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.

## TABLE 1 Suitable Pigment List

NOTE—Underlined information and the lightfastness rating in the table shall be included on every label.

Key:

*Lightfastness Category:*

Lightfastness I Excellent Lightfastness
Lightfastness II Very Good Lightfastness

*Abbreviations used in Colour Index Names:*

| | |
|---|---|
| PB | Pigment Blue |
| PBk | Pigment Black |
| PBr | Pigment Brown |
| PG | Pigment Green |
| PO | Pigment Orange |
| PR | Pigment Red |
| PV | Pigment Violet |
| PW | Pigment White |
| PY | Pigment Yellow |

*Pigment Notations:*

| | |
|---|---|
| (AR) | Alkali Resistant |
| (CC) | Concentrated cadmium pigments may contain up to 15 % barium sulfate for color control. Cadmium-barium pigments contain a much higher amount of barium sulfate. |
| (DL) | May darken in strong light |
| (LF) | Lightfast type |
| (NA) | Colour index name or number not assigned |
| (RS) | Red shade |
| (SM) | Sensitive to moisture in direct sunlight |
| (SS) | Sensitive to hydrogen sulfide |

| Colour Index Name | Lightfastness Category<br>Acrylic | Common Name and Chemical Class | Colour Index Number |
|---|---|---|---|
| | | **YELLOWS** | |
| PY 3 | II | Arylide Yellow 10G, with option of adding the name Hansa Yellow Light, arylide yellow | 11710 |
| PY 35 | I | Cadmium Yellow Light, concentrated cadmium zinc sulfide (CC) (SM) | 77205 |
| PY 35:1 | I | Cadmium-Barium Yellow Light, cadmium zinc sulfide coprecipitated with barium sulfate (SM) | 77205:1 |
| PY 37 | I | Cadmium Yellow Medium or Deep, concentrated cadmium sulfate (CC) (SM) | 77199 |
| PY 37:1 | I | Cadmium-Barium Yellow Medium or Deep, cadmium sulfide coprecipitated with barium sulfate (SM) | 77199:1 |
| PY 42 | I | Mars Yellow, with option of adding the name Yellow Iron Oxide, synthetic hydrated iron oxide | 77492 |
| PY 42 | I | Mars Orange, synthetic hydrated iron oxide | 77492 |
| PY 43 | I | Yellow Ochre, natural hydrated iron oxide | 77492 |
| PY 53 | I | Nickel Titanate Yellow, oxides of nickel, antimony and titanium | 77788 |
| PY 65 | I | Arylide Yellow RN, with option of adding Mansa Yellow RN, arylide yellow | 11740 |
| PY 73 | I | Arylide Yellow GX, with option of adding the name Hansa Yellow GX, arylide yellow | 11738 |
| PY 74 (LF) | I | Arylide Yellow 5Gx, with option of adding Hansa Yellow 5GX, arylide yellow | 11741 |
| PY 83 (HR70) | I | Diarylide Yellow HR70, diarylide yellow | 21108 |
| PY 97 | I | Arylide Yellow FGL, arylide yellow | 11767 |
| PY 98 | I | Arylide Yellow 10GX, with option of adding the name Hansa Yellow 10GX, arylide yellow | 11727 |
| PY 108 | I | Anthrapyrimidine Yellow, anthrapyrimidine | 68420 |
| PY 109 | I | Isoindolinone Yellow G, tetrachloroisoindolinone | NA |
| PY 110 | I | Isoindolinone Yellow R, tetrachloroisoindolinone | 56280 |
| PY 112 | I | Flavanthrone Yellow, flavanthrone | 70600 |
| PY 138 | I | Quinophthalone Yellow, quinophthalone | NA |
| PY 139 | I | Isoindoline Yellow, isoindoline | NA |
| PY 150 | I | Nickel Azo Yellow, nickel complex azo | NA |
| PY 151 | I | Benzimidazolone Yellow H4G, benzimidazolone | 13980 |
| PY 153 | I | Nickel Dioxine Yellow, dioxine yellow nickel complex | NA |
| PY 154 | I | Benzimidazolone Yellow H3G, benzimidazolone | 11781 |
| PY 175 | I | Benzimidazolone Yellow H6G, benzimidazolone | 11784 |
| | | **ORANGES** | |
| PO 5 | II | Dinitraniline Orange, dinitraniline (SM) | 12075 |
| PO 20 | I | Cadmium Orange, concentrated cadmium sulfo-selenide (CC) | 77202 |
| PO 20:1 | I | Cadmium-Barium Orange, cadmium sulfoselenide coprecipitated with barium sulfate | 77202:1 |
| PO 23 | I | Cadmium Vermilion Orange, concentrated cadmium mercury sulfide (CC) | 77201 |
| PO 23:1 | I | Cadmium-Barium Vermilion Orange, cadmium mercury sulfide coprecipitated with barium sulfate | 77201:1 |
| PO 36 | I | Benzimidazolone Orange HL, benzimidazolone | 11780 |
| PO 43 | I | Perinone Orange, perinone | 71105 |
| PO 48 | I | Quinacridone Gold, quinacridone | NA |
| PO 49 | I | Quinacridone Deep Gold, quinacridone | NA |
| PO 60 | I | Benzimidazolone Orange HGL, benzimidazolone | 11782 |
| PO 62 | I | Benzimidazolone Orange H5G, monoacetolone | 11775 |
| | | **REDS** | |
| PR 5 | II | Naphthol ITR, naphthol ITR | 12490 |
| PR 7 | I | Naphthol AS-TR, naphthol AS-TR | 12420 |
| PR 9 | I | Naphthol AS-OL, naphthol AS-OL | 12460 |
| PR 14 | II | Naphthol AS-D, naphthol AS-D | 12380 |

TABLE 1 *Continued*

| Colour Index Name | Lightfastness Category | Common Name and Chemical Class | Colour Index Number |
|---|---|---|---|
| | Acrylic | | |
| | | **REDS** (cont'd) | |
| PR 88 MRS[A] | I | Thioindigoid Violet, thioindigoid | 73312 |
| PR 101 | I | Indian Red, synthetic red iron oxide (bluish hue) | 77491 |
| PR 101 | I | Light or English Red Oxide, synthetic red iron oxide (yellowish hue) | 77491 |
| PR 101 | I | Mars Red, with option of adding the name Red Iron Oxide, synthetic red iron oxide | 77491 |
| PR 101 | I | Mars Violet, with option of adding the name Violet Iron Oxide, synthetic iron oxide (violet hue) | 77015 |
| PR 101 | I | Venetian Red, synthetic iron oxide (yellowish hue) | 77491 |
| PR 102 | I | Light Red, calcined yellow ochre | 77492 |
| PR 106 | I | Vermilion, mercuric sulfide (DL) | 77766 |
| PR 108 | I | Cadmium Red Light, Medium, or Deep, concentrated cadmium-seleno sulfide (CC) | 77202.1 |
| PR 108.1 | I | Cadmium-Barium Red Light, Medium or Deep, cadmium seleno-sulfide coprecipitated with barium sulfate | 77202:1 |
| PR 112 | II | Naphthol AS-D, naphthol AS-D | 12370 |
| PR 113 | I | Cadmium Vermilion Red Light, Medium or Deep, concentrated cadmium mercury sulfide (CC) | 77201 |
| PR 113.1 | I | Cadmium-Barium Vermilion Red Light, Medium or Deep, cadmium mercury sulfide coprecipitated with barium sulfate | 77201:1 |
| PR 119 | I | Naphthol Red, naphthol | NA |
| PR 122 | I | Quinacridone Magenta, $\gamma$ quinacridone | 73915 |
| PR 123 | II | Perylene Vermilion, perylene | 71145 |
| PR 149 | I | Perylene Red, perylene | 77137 |
| PR 168 | I | Brominated Anthranthrone, brominated anthranthrone | 58300 |
| PR 170 F3RK-70 | I | Naphthol Red, naphthol carbamide | 12475 |
| PR 170 F5RK | II | Naphthol Crimson, naphthol carbamide | 12475 |
| PR 171 | I | Benzimidazolone Bordeaux, monoazo benzimidazolone | 12512 |
| PR 175 | I | Benzimidazolone Maroon, benzimidazolone | 71513 |
| PR 179 | I | Perylene Maroon, perylene | 71130 |
| PR 181 | I | Thioindigoid Magenta, thioindigoid | 73360 |
| PR 188 | I | Naphthol AS, naphthol AS | 12467 |
| PR 190 | I | Perylene Red, perylene | 71140 |
| PR 192 | I | Quinacridone Red, $\gamma$ quinacridone red | NA |
| PR 194 | I | Pennone Red Deep, pennone | 71100 |
| PR 207 | I | Quinacridone Scarlet, quinacridone red | NA |
| PR 209 | I | Quinacridone Yellow Red, quinacridone red $\gamma$ | 73902 |
| PV 19 | I | Quinacridone Red, $\gamma$ quinacridone red | 73900 |
| | | **PURPLES** | |
| PV 15 | I | Ultramarine Red, complex silicate of sodium and aluminum with sulfur | 77007 |
| PV 15 | I | Ultramarine Violet, complex silicate of sodium and aluminum with sulfur | 77007 |
| PV 19 | I | Quinacridone Violet, quinacridone violet b | 73900 |
| PV 23 (RS) | II | Dioxazine Purple, carbazole dioxazine | 51319 |
| PV 31 | I | Isoviolanthrone Violet, isoviolanthrone | 60010 |
| | | **BLUES** | |
| PB 15 | I | Phthalocyanine Blue, copper phthalocyanine | 74160 |
| PB 16 | I | Phthalocyanine Blue, metal free phthalocyanine | 74100 |
| PB 22 | I | Indanthrone Blue, indanthrone | 69810 |
| PB 27 | II | Prussian Blue, with option of adding the name Milori Blue, Blue, ferri-ammonium ferrocyanide | 77510 |
| PB 28 | I | Cobalt Blue, oxides of cobalt and aluminum | 77346 |
| PB 29 | I | Ultramarine Blue, complex silicate of sodium and aluminum with sulfur | 77007 |
| PB 33 | I | Manganese Blue, barium manganate with barium sulfate | 77112 |
| PB 35 | I | Cerulean Blue, oxides of cobalt and tin | 77368 |
| PB 36 | I | Cerulean Blue, Chromium, oxides of cobalt and chromium | 77343 |
| PB 60 | I | Indanthrone Blue, indanthrone | 69800 |
| | | **GREENS** | |
| PG 7 | I | Phthalocyanine Green, chlorinated copper phthalocyanine | 74260 |
| PG 10 | I | Green Gold, with option of adding the name Nickel Azo Yellow, nickel chelated azo | 12775 |
| PG 17 | I | Chromium Oxide Green, anhydrous chromium sesquioxide | 77288 |
| PG 19 | I | Cobalt Green, oxides of cobalt and zinc | 77335 |
| PG 23 | I | Green Earth or Terra Verte, natural green | 77009 |
| PG 26 | I | Cobalt Green, cobalt chromite | 77344 |
| PG 36 | I | Phthalocyanine Green, chlorinated and brominated phthalocyanine | 74265 |
| PG 50 | I | Light Green Oxide, oxides of nickel, cobalt, and titanium | 77377 |
| | | **BROWNS** | |
| PBr 6 | I | Mars Brown, with option of adding the name Brown Iron Oxide, synthetic brown iron oxide or mixtures of synthetic iron oxides | 77499 |
| PBr 7 | I | Burnt Sienna, calcined natural iron oxide | 77492 |
| PBr 7 | I | Burnt Umber, calcined natural iron oxide containing manganese | 77492 |
| PBr 7 | I | Raw Sienna, natural iron oxide | 77492 |
| PBr 7 | I | Raw Umber, natural iron oxide containing manganese | 77492 |

TABLE 1 *Continued*

| Colour Index Name | Lightfastness Category | Common Name and Chemical Class | Colour Index Number |
|---|---|---|---|
| | Acrylic | | |
| | | **BLACKS** | |
| PBk 6 | I | Lamo Black, nearly pure amorphous carbon | 77266 |
| PBk 7 | I | Carbon Black, nearly pure amorphous carbon | 77266 |
| PBk 9 | I | Ivory Black, amorphous carbon produced by charring animal bones | 77267 |
| PBk 11 | I | Mars Black, with option of adding the name Black Iron Oxide, synthetic black iron oxide | 77499 |
| | | **WHITES** | |
| PW 4 | I | Zinc White, zinc oxide | 77947 |
| PW 6 | I | Titanium White, titanium dioxide (rutile or anatase) with option of including some barium sulfate or zinc oxide | 77891 |

[A] Applies only to Permanent Red Violet MRS, product of American Hoechst Corp., Coventry, RI 02816. Pigments described as thioindigoids have varying degrees of lightfastness.

described in 5.1.5 and 5.1.6. Other identification may be placed elsewhere on the container.

5.1.4 The Colour Index Name may be spelled out in full or abbreviated depending on the size of the label. Example: Pigment Blue 15, or Pig. Blue 15 or PB 15.

5.1.5 *Substituted Pigments*—In the case of substituted pigments, the word "Hue" in equal size letters shall follow in the title, on the front of the tube, immediately after the name of the pigment that has been simulated. Directly below the title, the Common Name of the significant pigment used shall be given in letters no less than the next type size smaller than the title. For example:

COBALT BLUE HUE
(ULTRAMARINE BLUE).

5.1.6 Proprietary names or optional names may be used provided the Common Name(s) given in Table 1 appears on the front of the label directly under the proprietary or optional name in letters no less than the next type size smaller than the proprietary or optional name.

5.1.7 *Mixed Pigments*—Artists' paints containing more than one colorant comply with this specification if all colored pigments used are on the suitable pigment list (Table 1) and provided the mixture itself has passed all other test requirements in this specification. The lightfastness category shall be that of the least lightfast pigment. This lightfastness category may be changed if these paints are tested for lightfastness in accordance with Test Methods D 4303 and results indicating a different category are submitted to ASTM Subcommittee D01.57 for evaluation.

5.2 Provide on the label identification of polymer used in the paint.

NOTE 1—The type of polymer can be identified by using Practice D 3168.

5.3 *Lightfastness*—The label shall contain the word "Lightfastness" followed by the appropriate rating, I or II, as given for each pigment in Table 1.

5.3.1 Lightfastness I pigments, when made into paint specimens as described in Section 7 and exposed, tested, and rated in accordance with Test Method D 4303, shall have a color difference ($\Delta E^*_{ab}$) of 4 or less CIELAB units between the specimens measured before and after exposure.

5.3.2 Lightfastness II pigments, when made into paint specimens as described in Section 7 and exposed, tested, and rated in accordance with Test Methods D 4303, shall have a color difference ($\Delta E^*_{ab}$) of more than 4.0 but not more than 8.0 CIELAB units between the specimens measured before and after exposure.

5.3.3 Pigments were placed in a lightfastness category on the basis of either known historical performance in art works or the ratings from four lightfastness tests conducted as described in Test Methods D 4303. Results from further tests on these, or other pigments, are solicited by ASTM Subcommittee D01.57.

5.3.3.1 The lightfastness category of a pigment shall be changed if results from several further tests conducted in accordance with Test Methods D 4303 and approved by ASTM Subcommittee D01.57, establish a different lightfastness category than the one given in Table 1.

5.3.3.2 Additional pigments shall be placed in Table 1 after they have been tested for lightfastness in accordance with Test Methods D 4303 and the test results submitted to ASTM Subcommittee D01.57 for evaluation, provided the results demonstrate that the pigments have the lightfastness ratings required for Lightfastness I or Lightfastness II, as described in 5.3.1 and 5.3.2.

5.3.4 For information and to establish nomenclature, pigments in Lightfastness III category are given in Table X1.1 in Appendix X1, but are not to be used in paint conforming to this specification. These pigments have a color difference before and after exposure of more than 8.0 but not more than 16.0 CIELAB units.

5.4 *Toxicity*—All products and labeling must conform to the Federal Hazardous Substances Act and to Practice D 4236.

5.5 *Statement of Conformance*—"Conforms to ASTM Specification D 5098," or "Conforms to ASTM D 5098," or "Conforms to the quality requirements of ASTM D 5098." This statement may be combined with other conformance statements, such as, "Conforms to the quality and health requirements of ASTM Specification D 5098 and Practice D 4236."

5.6 *Address*—Include on the label (*1*) the name and address of the manufacturer or importer and (*2*) the country of manufacture.

## 6. Quality Assurance for Artists' Acrylic Emulsion Paints

6.1 *Conditions not Covered in This Specification that Affect the Quality of Artists' Acrylic Emulsion Paints:*

6.1.1 *Substrate*—Factors such as the texture, gloss, effective pH, porosity, chemical composition, and condition of the substrate will affect gloss, gloss uniformity, drying time, adhesion, and the flexibility of the dried film.

6.1.2 *Environmental Conditions*—Factors such as temperature, humidity, air flow, and light conditions affect application properties, film formation, drying time, and adhesion.

6.1.3 *Storage*—Factors such as aging, and high and low temperatures may cause changes in consistency.

6.2 *Vehicles*—Only acrylic polymer emulsions or acrylic copolymer emulsions may be used (see Note 1).

6.3 *Pigments*—The pigments shall be limited to those recommended for use in acrylic emulsion paints in the list of suitable pigments in Table 1. Their lightfastness rating shall be the numeral given in the same row.

6.4 *Additives*—Surfactants, preservatives, defoamers, glycols, solvents, and thickeners may be used to achieve aging stability, to control foaming, to ensure freeze-thaw stability and film coalescence, and to obtain a desired consistency.

6.5 *Inerts*—Inerts shall be included only to adjust product gloss or sheen, or to produce desirable working qualities.

6.6 *Preparation of Sample*—Empty the contents of a previously unopened container onto a glass slab and mix thoroughly with a spatula to a homogeneous sample.

6.7 *Coarse Particles*—Paints shall be free of oversize particles and shall form a uniform film. The maximum content of coarse particles shall be 1 weight % as determined by Test Methods D 185.

6.8 *Fineness of Dispersion*—Determine the fineness of dispersion by Test Method D 1210. For paste paint: on a glass plate, using a spatula, mix the paint with an equal volume of water until homogeneous. The maximum allowable grind reading is 1.5 mils (40 μm).

6.9 *Consistency*—Paints shall be smooth and creamy. The paste type of paint shall not flow or level when applied with a palette knife.

6.10 *Freeze-Thaw Stability*—Using a freezer that has a temperature of 20°F (−7°C) or lower, subject the paint to five freeze-thaw cycles. A freeze-thaw cycle shall consist of freezing the paint to a solid state (minimum of 18 h) and then thawing the paint to room temperature (minimum of 5 h). The paint shall then meet the requirements of 6.7, 6.8, and 6.9.

6.11 *Drying*—Use a 6-mil (150-μm) clearance film applicator to make a uniform drawdown on a lacquer-sealed panel. At a relative humidity of 50 to 75 % and a temperature of 65 to 80°F (18 to 27°C), the dust-free drying time, determined in accordance with Test Method D 1640, shall be not less than 10 min.

6.12 Tinting strength requirements will be included in this specification as appropriate tinting strength standards for individual pigments are established. Test Method D 387 may be used to determine the tinting strength of pigments or paints when all ingredients are known. Test Method D 4838 can be used to determine the relative tinting strength of chromatic paints containing a single pigment and the same vehicle but where other ingredients are unknown.

## 7. Lightfastness Determination

7.1 If a pigment is not listed in Table 1, test specimens of a paint containing the pigment shall be prepared. These test specimens shall be tested in conformance with the requirements for exposure and evaluation given in Test Methods D 4303.

NOTE 2—A report of the results of these tests may be submitted to ASTM Subcommittee D01.57 for inclusion of the pigments in Table 1. The report shall include information on test conditions, instruments used, and be accompanied by the test specimens, which will be returned.

7.2 *Materials:*

7.2.1 *Aluminum Exposure Panels,*[6] 3 by 6 in. (75 by 150 mm).

7.2.2 *Posterboard,* lightweight, approximately 20 mils (0.5 mm) thick, having a glossy finish on one side.

7.2.3 *Titanium Dioxide Acrylic Emulsion Paint.*

7.2.4 *Spray Apparatus,* for applying ground coats.

7.2.5 *Paint Brush,* 2.5 in. (60 mm) wide for applying ground coats if spray apparatus is not available.

7.2.6 *Soft Hair Brush,* 1 in. (25 mm) wide for applying ground coats to aluminum panels. An oxhair artist's "stroke" brush is suitable.

7.3 *Specimen Preparation:*

7.3.1 *Ground Coats:*

7.3.1.1 For the first ground coat under acrylic emulsion paints, prepare the following enamel:

| | Weight % |
|---|---|
| Medium oil length soya alkyd, 50 % nonvolatile[7] | 20 |
| Rutile titanium dioxide[8] (conforming to Type II of Specification D 476) | 40 |
| Blanc fixe (conforming to Specification D 602) | 40 |
| Driers: 0.15 % zinc and 0.15 % zirconium as metal on the alkyd nonvolatile | |
| Sufficient mineral spirits for milling | |

7.3.1.2 Mill to a Hegman fineness of 7 as measured by Test Method D 1210. Thin with mineral spirits to appropriate viscosity for spraying or for flow coating by brush.

7.3.2 For the second ground coat for acrylic emulsion paints, prepare the following enamel:

| | Weight % |
|---|---|
| Medium oil length soya alkyd, 50 % nonvolatile[7] | 40 |
| Rutile titanium dioxide[8] (conforming to Type II of Specification D 476) | 30 |
| Colloidal silica[9] | |
| Driers: 0.15 % zinc and 0.15 % zirconium as metal on the alkyd nonvolatile | 30 |
| Sufficient mineral spirits for milling | |

7.3.2.1 The colloidal silica may be dispersed separately in mineral spirits using a high-speed mixer and added to the milled alkyd-titanium pigment paste. Mill to a Hegman fineness of 7 as measured by Test Method D 1210. Use sufficient mineral spirit for proper application.

NOTE 3—These soya alkyd enamels are used for the ground coats because of their color stability, nonabsorbency, adhesion of the specimen coats under humid conditions, and freedom from blistering that can occur with an acrylic ground coat under high humidity.

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[6] The No. A-36 aluminum panel manufactured by Q Panel Co., 26200 First St., Cleveland, OH 44145, has been found satisfactory for this purpose.

[7] The following alkyd resins have been found satisfactory for this purpose: Cargill, No. 5196; IMC McWhorter, DURAMAC 2419; Reichhold, No. 11-035; and Spencer-Kellog, AROPLAZ 1082 M50.

[8] DuPont R900 rutile titanium dioxide, available from E.I. du Pont de Nemours & Co., 1007 Market St., Wilmington, DE 19898, has been found satisfactory for this purpose.

[9] Syloid 308, available from Davison Chemical, Div. W.R. Grace Co., PO Box 2117, Baltimore, MD 21203, has been found satisfactory for this purpose.

7.3.3 *Application of Ground Coats:*

7.3.3.1 Degrease aluminum substrates before applying the ground coat.

7.3.3.2 Apply one coat of the enamel described in 7.3.1.1, followed by one coat of the enamel described in 7.3.2. To flow coat the aluminum panels for sun exposure use a 1-in. (25-mm) soft hair brush. Coat the posterboard to be used for laboratory exposure on the less absorbent, glossy side.

NOTE 4—Coating the posterboard is most conveniently done on the whole sheet before cutting to size.

7.3.3.3 Allow a minimum of five days drying time following the first coat and two weeks or more after applying the second coat and before applying the paint to be tested.

7.4 *Mixing Whites for Dilution of Colors:*

7.4.1 Use a white containing the same emulsion and additives as the formulation in which the pigment is incorporated if possible.[10] The following basic composition has been found satisfactory:

| | Weight % |
|---|---|
| Acrylic emulsion nonvolatile | 23 to 24 |
| Rutile titanium dioxide[8] (conforming to Type II of Specification D 476) | 34.6 |

7.4.1.1 Keep the viscosity of the mixing white to 250 P or slightly lower.

7.5 *Preparation of Test Paints:*

7.5.1 The pigment to be tested may be milled in a compatible paste for acrylic emulsion. If a prepared artists' paint of known composition is available, it may be used for this test instead of preparing a dispersion.

7.5.2 Dilute the pigment paste or paint with the white containing the same type of vehicle until the spectrophotometric measurement of the dried film shows 35 to 45 % relative reflectance at the wavelength of maximum absorption for that pigment. The wavelength of maximum absorption is located at the point of lowest reflectance on the spectral curve between 420 and 620 nm. If using a tristimulus filter colorimeter, the lowest of the three filter readings is the region of maximum absorption and the dilution should be adjusted so that a reading of 35 to 45 % reflectance is obtained with this filter. The diffuse white reference standard for all measurements should have an absolute reflectance between 97 and 100 %.

7.5.2.1 To obtain this reflectance, use the Kubelka-Munk Single Constant Method described in Appendix X2 or use a trial and error method.

7.5.3 Make instrumental readings with the specular reflectance included to minimize the effect on readings of any change in gloss and to minimize the effect of brush strokes.

7.5.4 Use an applicator with a 6-mil (150-µm) aperture to make a minimum size drawdown, 1½ by 1½ in. (40 by 40 mm), or other minimum size appropriate for the viewing area of the instrument used. Return all recoverable paint to the batch to allow for repeat mixes and measurements.

7.5.5 For acrylic emulsion paints, 80 g of white paint is needed since paint used in the drawdown is lost.

7.5.6 For the initial weights of white stated in 7.5.4 and 7.5.5, the weighing must be accurate to 0.05 g.

7.6 *Application of Paints to Panels:*

7.6.1 Prepare four specimen panels on appropriate substrates for each pigment under test. Two are used in the first lightfastness tests and two are retained in subdued light, one for visual comparisons with the exposed panels and one in case a third test is needed to supplement results from the first two tests, as described in Test Methods D 4303.

7.6.2 Apply the test paints by brush to the aluminum panel if exposure is to be under glass to the sun or by brush to the posterboard panel if exposure is to be to laboratory apparatus.

7.6.3 Using the No. 12 artist's flat bristle brush, brush the panels lengthwise, then crosswise, and again lengthwise, this time with a light touch to produce a film as smooth as possible. The mixing whites must be fluid enough to facilitate leveling, but not have excess vehicle that can affect the test. Do not use thinners. Apply two coats to all specimen panels to achieve complete opacity.

7.6.4 Allow specimens to dry between coats as described in the Procedure section (Dry-Through or Dry-to-Handle Time) of Test Methods D 1640. After recoating, allow specimens to dry hard, as described in the Procedure section (Dry-Hard Time) of Test Methods D 1640, before measuring them prior to exposure. Acrylic paints can be recoated the next day and read two days after recoating.

## 8. Exposure

8.1 Conduct exposure tests, calculate mean color difference, and assign pigments to lightfastness categories as described in Test Methods D 4303.

## 9. Keywords

9.1 artists' acrylic paints; lightfastness; quality requirements; test specimens

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[10] If the acrylic vehicle composition of the paint is unknown, Rhoplex AC-234, from Rohm & Haas Co., Independence Mall W., Philadelphia, PA 19105, is compatible with most acrylic emulsion paints.

# APPENDIXES

## (Nonmandatory Information)

## X1. LIGHTFASTNESS III

X1.1 The pigments in Table X1.1 are not sufficiently lightfast to be used in paints that conform to this specification. These pigments are listed here solely to establish common terminology. They may be satisfactory when used full strength (without dilution) or with extra protection from exposure to light.

**TABLE X1.1 Lightfastness III**

| Colour Index Name | Lightfastness Category<br>Acrylic | Common Name and Chemical Class | Colour Index Number |
|---|---|---|---|
| PR 17 | III | Naphthol Red AS-D, naphthol | 12390 |
| PR 146 | III | Naphthol Red, naphthoic arylide | 12485 |
| PV 23BS | III | Dioxazine Purple, carbazole dioxazine | 51319 |
| PG 8 | III | Hooker's Green, ferric-nitroso-beta-naphthol | 10006 |

## X2. KUBELKA-MUNK SINGLE CONSTANT METHOD FOR PREPARING PAINT FILMS 40 ± 5 % REFLECTANCE

X2.1 Prepare a mixture of white and colorant in proportions estimated to give a paint having a reflectance of 30 to 60 % at the wavelength of maximum absorption. Calculate the concentration of colorant in white in this batch as follows:

$$C_c = W_c / W_w + W_c$$

where:

$C_c$ = concentration of colorant, and

$W_w$ and $W_c$ = weight of white and colorant, respectively, used in the batch.

Draw or brush a film of this paint over contrast paper at a thickness sufficient to give complete visual hiding.

X2.1.1 After allowing the film to dry, measure the reflectance of this film at the wavelength of maximum absorption with a spectrophotometer or, if using a colorimeter, the reflectance with the filter that gives the lowest reading (see Note X2.1). Calculate the Kubelka-Munk Single Constant Value of this reflectance as follows:

$$(K/S)_m = [1 - (R_m - 0.04)]^2 / 2(R_m - 0.04)$$

where:

$(K/S)_m$ = Kubelka-Munk Value,

$R_m$ = reflectance measurement of the mixture, made with the specular component included expressed as a decimal value, and

0.04 = decimal value correction for surface reflectance.

NOTE X2.1—To speed drying, accelerated methods may be used. It is recommended that water-based acrylic paints be warmed to 50 to 55°C for 2 h after 1 h of air drying before measuring the reflectance. Placing the drawdowns of oil paints near fluorescent lamps, for example in the fluorescent light exposure racks described in Annex A1 of Test Methods D 4303, will speed their drying time.

X2.1.2 Calculate the Kubelka-Munk absorption coefficient for unit concentration as follows:

$$K_c = (K/S)_m / C_c$$

where $K_c$ = unit absorption coefficient.

X2.2 Based on the results of the trial mixture, calculate the concentration of colorant in white required to give the desired 35 to 45 % reflectance at the wavelength of maximum absorption as follows:

$$C_d = (K/S)_{40\ \%} / K_c = 0.4500 / K_c$$

where $C_d$ = concentration of colorant in white predicted to give a paint with the desired 35 to 45 % reflectance value.

X2.3 Adjust the concentration of the trial batch so that the concentration of colorant equals $C_d$. Determine the changes in colorant concentration, $\Delta C$ as follows:

$$\Delta C = C_d - C_c$$

These changes are positive if the trial batch has a reflectance greater than 40 % and negative if the reflectance is less than 40 %. If $\Delta C$ is positive, add more colorant to the trial batch to bring the concentration of colorant to $C_d$. If $\Delta C$ is negative, add more white to bring the concentration of colorant to $C_d$. Because it may be necessary to add large amounts of white to correct for concentrations of colorant giving reflectance values far below 40 %, it is usually best to prepare a trial batch expected to give a $\Delta C$ value that is moderately positive or only slightly negative.

X2.3.1 For the case where $\Delta C$ is positive, the weight of colorant needed to prepare a second batch is,

$$\Delta W_c = W_c - C_d(W_w + W_c)/C_d - 1 \times (LF)$$

where:

$\Delta W_c$ = weight of colorant to be added to the trial batch to obtain a corrected batch with a colorant concentration of $C_d$, and

$LF$ = "loss factor" = weight of paint from trial batch used to prepare the corrected batch/weight of paint prepared as trial batch.

The loss factor, $LF$, allows for paint removed to prepare drawdowns, loss by transfer to different containers, etc. This term may be neglected if losses are small compared to the batch size. The other terms are as defined in the previous sections of this appendix.

X2.3.2 For the case where $\Delta C$ is negative, the weight of white needed to prepare a corrected batch corresponds to,

$$\Delta W_w = W_c - C_d(W_w + W_c)/C_d \times (LF)$$

where $\Delta W_w$ = weight of white needed to adjust the colorant concentration in the batch to $C_d$. Other terms are as defined above or in previous sections of this appendix.

X2.4 Prepare a paint film from the adjusted batch. After drying, measure the reflectance at the wavelength of maximum absorption. If this value does not fall within 35 to 45 %, repeat X2.3 through X2.4, where $C_d$ now becomes the $C_c$ for the next batch correction. Normally only a single iteration is necessary and a second batch correction will not be needed.

# Standard Practice for
# Preparatory Surface Cleaning of Architectural Sandstone[1]

This standard is issued under the fixed designation D 5107; 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 non-abrasive surface cleaning of architectural sandstone to remove grease, dirt, loose material, and surface deposits such as soot, fly ash, hydrocarbon residues, and algae and other biological growth in preparation for the application of water repellent coatings. Procedures include broom cleaning, vacuum cleaning, air blast cleaning, water cleaning (and detergent water cleaning), and chemical cleaning.

1.2 *Limitations*—This practice is intended to clean architectural sandstone without damaging it or altering the surface profile.

1.3 *This standard does not purport to address all of the safety problems 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 4.

## 2. Referenced Documents

2.1 *ASTM Standards:*

C 119 Terminology Relating to Natural Building Stones[2]

D 4262 Test Method for pH of Chemically Cleaned or Etched Concrete Surfaces[3]

D 4263 Test Method for Indicating Moisture in Concrete by the Plastic Sheet Method[3]

D 4285 Test Method for Indicating Oil or Water in Compressed Air[3]

## 3. Significance and Use

3.1 Surface cleaning is necessary to prepare architectural sandstone surfaces for application of coatings intended for water repellent protection. Surface cleaning of the sandstone substrate helps to ensure proper adhesion of the coating.

3.2 Use of procedures described in this practice may not be adequate where protective systems will be used for continuous or intermittent immersion or mechanical loading.

## 4. Hazards

4.1 Moisture in the architectural sandstone may be detrimental to coating adhesion or (in some cases) cure. Moisture content shall be in compliance with coating manufacturer's recommendation. See also Test Method D 4263.

4.2 Localized staining (for example efflorescence and metallic staining) and previously applied coatings or preservative treatments not compatible with the treatment may require removal by other surface preparation methods.

4.3 Water cleaning, detergent water cleaning, and chemical cleaning should not be performed at temperatures below 40°F.

4.4 Many chemical cleaning products contain acids and should be handled according to manufacturers' recommendations. Use and disposal of materials should conform to established federal, state, local, and project requirements.

4.5 If pressure washing equipment is employed for water cleaning or for flushing the surface with detergent water cleaning or chemical cleaning, the minimum effective pressure should be used. Avoid excessive pressures that could damage the sandstone substrate.

## 5. Procedure

5.1 Air blast, water, scrubbing, sweeping, or vacuuming are acceptable cleaning methods. Chemical cleaning agents may be used to remove surface deposits such as soot, fly ash, and hydrocarbon residues not removed by any of the above methods. Chemical cleaning should be preceded and followed by a thorough fresh water rinse.

5.2 Existing conditions of the substrate will determine the selection of appropriate procedure(s). Prior to the initiation of cleaning, small test areas should be cleaned in inconspicuous areas by the selected procedure to determine effectiveness.

5.3 The six types of cleaning procedures are described below. One or more of the procedures may be required to remove contaminants from the sandstone surface:

5.3.1 *Broom Cleaning*—Removes most loosely adherent solid contaminants.

5.3.1.1 Working from top to bottom, sweep the surface with a clean industrial stiff-bristled broom or similar device. Remove sweepings from the immediate work area.

5.3.1.2 Clean broom-cleaned surfaces again using one or more of the surface preparation procedures specified in 5.3.2 through 5.3.6.

5.3.2 *Vacuum Cleaning*—Removes surface dust and other debris.

5.3.2.1 Vacuum the surface with a heavy-duty type industrial vacuum to provide an essentially dust-free surface.

5.3.3 *Air Blast Cleaning*—Removes debris, dust, dirt, loosely adherent architectural sandstone, and laitance from walls to provide an essentially sound, dust-free surface.

5.3.3.1 Clean surface with a compressed-air stream through a blasting nozzle held at an oblique angle approximately 2 ft (0.6 m) from the surface. Air stream pressure

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[1] This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.47 on Masonry Treatments.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 06.02.

should not exceed 100 psi (689 kPa).

5.3.3.2 Before initiating air-blast cleaning, verify that the air stream is free of oil in accordance with Test Method D 4285.

5.3.3.3 Surface cleanliness is dependent upon carrying off airborne dust before it is redeposited. Vacuum cleaning may be required to remove redeposited dust.

5.3.4 *Water Cleaning*—Removes dust, dirt, and water-soluble surface contaminants.

5.3.4.1 Clean the surface with a stream of clean potable water, aimed at an oblique angle approximately 2 ft (0.6 m) from the surface, having sufficient pressure to remove dust, dirt, and loose material without damaging the substrate. When necessary, hand scrub with a nonmetallic stiff-bristled fiber brush.

5.3.4.2 Prior to water cleaning, make provisions for the removal of wash water and contaminants generated.

5.3.4.3 If necessary, test the cleaned surface for moisture content in accordance with Test Method D 4263 prior to applying coatings.

5.3.5 *Detergent Water Cleaning*—Removes water-soluble surface contaminants and oils, grease, and other emulsifiable materials on the surface.

5.3.5.1 Scrape off heavy deposits of grease or oil and prewet the surface with potable water. Clean the surface with a nonmetallic stiff-bristled fiber brush, using an aqueous solution of detergent or nonsolvent emulsifier. Immediately after treatment, before the surface dries, remove residues of the cleaning agent by thoroughly flushing the surface with clean potable water. Repeat flushing until the pH of the surface water meets the acceptance criteria of Test Method D 4262.

5.3.5.2 Repeat 5.3.5.1 until water does not bead on the surfaces.

5.3.5.3 Prior to detergent water washing, make provisions for the removal of wash water and contaminants generated.

5.3.5.4 If necessary, test the cleaned surface for moisture content in accordance with Test Method D 4263.

5.3.6 *Chemical Cleaning*—This procedure is similar to detergent water washing, but involves the use of proprietary chemical cleaning compounds (generally hydrofluoric and phosphoric acids) for the removal of surface deposits such as soot, fly ash, and hydrocarbon residues.

NOTE—In some cases, removal of heavy soiling may require use of alkaline pretreatment prior to acidic cleaning.

5.3.6.1 Chemical cleaning using acidic products is generally not recommended for calcareous sandstones. (See Terminology C 119.)

5.3.6.2 Protect all adjacent materials and surrounding areas as recommended by the manufacturer of the proprietary chemical cleaning compound.

5.3.6.3 Prior to chemical cleaning, provide for application to small test area to determine effectiveness. Make provisions for the removal of water and contaminants generated during full-scale cleaning operations.

5.3.6.4 Before applying the chemical cleaning compound, prewet the surface thoroughly with potable water to prevent absorption of the cleaning solution within the pores of the masonry.

5.3.6.5 Apply a dilute solution of a proprietary cleaning compound to the pre-wet surface as recommended by the manufacturer. Leave the cleaning solution on the surface for a prescribed dwell period (usually less than 5 min).

5.3.6.6 Immediately following the dwell period, and before the surface dries, flush thoroughly to wash chemical cleaning compounds from the surface, then rinse thoroughly from bottom to top. (Rinsing from bottom to top helps to avoid surface streaking.) Repeat flushing may be necessary to remove cleaning residues.

5.3.6.7 *Evaluation*—Visually examine the prepared surface to evaluate removal of debris, dust, dirt, oil, grease, loosely adherent sandstone building stone, and other contaminants. Test the surfaces cleaned with proprietary chemical cleaning compounds for pH in accordance with Test Method D 4262 and if necessary test for moisture content in accordance with Test Method D 4263 prior to applying coatings.

5.4 Surface cleaning is intended to provide a clean, contamination-free surface without damaging or removing architectural sandstone from intact, sound surfaces. Acceptable surfaces shall be free of oil, grease, loosely adhering sandstone, and other contamination such as dirt, soot, fly ash, and hydrocarbon residues.

Designation: D 5108 – 90

# Standard Test Method for Organotin Release Rates of Antifouling Coating Systems in Sea Water[1]

This standard is issued under the fixed designation D 5108; 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 laboratory determination of the rate at which organotin expressed as tributyltin (TBT) is released from an antifouling (AF) coating in synthetic sea water using graphite furnace atomic absorption spectrophotometry (GF-AAS). This does not exclude the use of other analytical methodology for measurement of organotin in sea water such as gas chromatography.

1.2 The values stated in SI units are to be regarded as standard. The inch-pound units given in parentheses are for information only.

1.2 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety practices and to determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 7.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1141 Specification for Substitute Ocean Water[2]

D 1212 Test Methods for Measurement of Wet Film Thickness of Organic Coatings[3]

D 4138 Test Method for Measurement of Dry Film Thickness of Protective Coatings by Destructive Means[4]

## 3. Summary of Test Method

3.1 The candidate paint system is applied to cylindrical test specimens. The coated specimens are placed in a tank of synthetic sea water where the tin levels are kept low by circulating the sea water through a carbon filter. At specified intervals, each specimen is placed in 1500 mL of unused sea water and is rotated for 1 h. The rate of tributyltin release from the paint is determined by measuring tributyltin concentrations in the sea water.

3.2 Analysis of sea water for tributyltin is conducted by extracting the organotin with toluene, washing with sodium hydroxide, and measuring for total tin using (GF-AAS).

## 4. Significance and Use

4.1 This test method is designed to provide a laboratory procedure to measure changes in the release rates of solvent soluble tin (tributyl- and triphenyltin) that occur during a period of immersion under specified conditions of constant temperature, pH, salinity, and low heavy-metal concentrations in the surrounding sea water. Quantitative measurement of the release rate is necessary to help in selection of materials, in providing quality assurance, and in understanding the performance mechanism.

4.2 This test method serves only as a guide for organotin release rates in service. Organotin release rates of antifouling (AF) paint systems in service can vary over the life of the coating system depending on the formulation and the environment. Differences in berthing locations, operating schedules, length of service, condition of paint-film surface, temperature, pH, and salinity can affect results. Results obtained may not necessarily reflect actual tributyltin release rates that will occur in service, but provide reliable comparisons of the release rate characteristics of different antifouling formulations.

4.3 This test method will serve to characterize the early release rate pattern, as well as estimate the steady state release, of tributyltin from both self-polishing copolymer and free-association antifouling paints.

## 5. Apparatus

5.1 *Release-Rate Measuring Container*—A 2-L (nominal ½ gal) polycarbonate container,[5] approximately 13.5 cm (5.5 in.) in diameter and 19 cm (7.5 in.) high, fitted with three polycarbonate rods approximately 6 mm (nominal ¼ in.) in diameter to serve as baffles. Rods shall be evenly spaced on the inside circumference of the container to prevent swirling of water with the test cylinder during rotation. The rods will be secured to the container walls using acetone or methylene chloride.

5.2 *Constant Temperature Bath*—A temperature controlled water bath capable of maintaining a temperature of 25 ± 2°C into which one or more release rate measuring test containers can be placed.[6]

5.3 *Holding Tank*—A container of such dimensions so as to permit immersion of four or more test cylinders; must be equipped with a system to continuously circulate synthetic sea water in the tank through a carbon filter. The rate of water flow and the size of the carbon filter should be selected to maintain tributyltin concentrations below 100 µg/L. Flow

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.45 on Marine Coatings.

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[2] *Annual Book of ASTM Standards*, Vol 11.02.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.02.

[5] A Nalgene Container, available from Cole-Palmer, 7425 N. Oak Ave., Chicago, IL 60648, catalog number R-6761-20, or equivalent, has been found satisfactory for this purpose.

[6] Boekel Water Baths, Models 148003 and 148004 available from Boekel Industries Inc., 509-T Vine St., Philadelphia, PA 19106, or equivalent, have been found satisfactory for this purpose.

rates should generally be set to obtain 2 to 8 turnovers per h. The size and geometry of the tanks as well as the positioning of the inflow and outflow ports for the water circulation system should be selected to obtain a slow, relatively uniform flow of synthetic sea water past all test cylinders in the tank. Maintain the pH of the synthetic sea water between 7.8 and 8.2, and the salinity between 30 and 35 parts per thousand (ppt). The tank shall be provided with heaters to maintain the temperature between 21 and 27°C (70 and 81°F).

5.4 *Test Cylinders*—Approximately 6.4 cm (nominal 2½ in.) outside diameter polycarbonate pipe coated with a 10-cm band of AF paint around the exterior circumference of the test cylinder to provide 200 $cm^2$ of paint film that can be immersed and freely rotated in the release rate measuring container. Seal the bottom of the test cylinder with a polycarbonate disc using acetone, methylene chloride, or a polycarbonate cement so as to form a watertight joint. Do not coat the bottom 1 to 2 cm of the test cylinder. The test cylinder shall be of such height so that a rotating device can be attached to rotate the cylinder and the upper open end of the cylinder is above the level of the test container immersion liquid to prevent entry of the immersion liquid into the test cylinder.

5.5 *Test Cylinder Rotating Device*—The device shall be capable of rotating the test cylinder in the release rate measuring container at 60 ± 5 r/min. No part of the device shall be immersed in sea water.[7]

5.6 *Centrifuge Tubes*, 50-mL capacity, with screw closures[8] (or disposable bottles, culture tubes, separatory funnels, etc.) made of polycarbonate, TFE flourocarbon, or borosilicate glass.

5.7 *Mechanical Shaker*, with appropriate holders.

5.8 *Dispensers*, automatic or repeating, for reagents.

5.9 *Pipets*, with disposable polypropylene tips.

5.10 *Graphite Furnace*, atomic absorption spectrophotometer (GF-AAS) with automatic sampler.

5.11 *pH Meter*, with a mercury/mercurous chloride ($Hg/Hg_2Cl_2$) electrode.

5.12 *Appropriate Volumetric Flasks*.

## 6. Reagents and Materials

6.1 *Synthetic Sea Water*—Substitute ocean water in accordance with Section 6 of Specification D 1141 or a proprietary equivalent with a salinity of 30 to 35 ppt.

6.2 *Extraction Solvent*—Toluene, spectrograde or equivalent.

6.3 *Tributyltin Standards*—Prepare standards using a stock solution of tributyltin chloride (reagent grade, minimum 96 % pure) in methanol (suggested concentration of approximately 10 mg/L). The standards are acidified with acetic acid (less than pH 4) to obtain a stable solution.

6.4 *Hydrochloric Acid* (HCl) (10 % aqueous solution).

6.5 *Hydrochloric Acid* (HCl) (0.1*N*).

6.6 *Nitric Acid* ($HNO_3$) (10 % aqueous solution) can be used in place of HCl to clean labware.

6.7 *Sodium Hydroxide* (NaOH) (3 % aqueous solution).

6.8 *Sodium Hydroxide* (NaOH) (0.1*N*).

6.9 All reagents and cleaning agents used must be tin-free.

## 7. Hazards

7.1 **Warning**—Antifouling paints contain toxic materials that could cause skin and eye irritation on contact and adverse physiological effects if ingested or inhaled. In the preparation of test specimens and the application of various types of paints, the use of appropriate protective clothing and equipment is required consistent with local, state, and federal government regulations, and recognized industrial and technical standards. Spills, overspray, and unused material should not be flushed down the drain, but should be disposed of as hazardous waste.

7.2 See antifouling paint supplier's Material Safety Data Sheet.

## 8. Calibration and Standardization

8.1 Prepare three standards throughout the range of the quantification limit to 100 µg of tin per litre by dilution in toluene of a stock solution of tributyltin chloride (96 % pure) in methanol. Include one standard with a concentration of approximately 50 µg of tin per litre. An alternate range of concentrations may be used when appropriate.

8.2 Prepare synthetic sea water spiked with three concentrations of TBT in the range of 10 to 50 µg of tin per litre by spiking with stock solution of tributyltin chloride in methanol. When the concentration of tin extracted in toluene exceeds 100 µg/L appropriate dilution should be employed to keep it within the limits of the calibration curve (0 to 100 µg/L).

8.3 Operate the graphite furnace in accordance with manufacturer's instructions. Optional conditions are described in Appendix X1.

8.4 Analyze the following:

8.4.1 At the beginning of each instrument run, perform analysis of the toluene blank and standards in toluene in order to establish that the response of the instrument is linear. Plot separate calibration curves for each analysis of the standards (peak height absorbance versus tin concentration), and calculate the slope, intercept, and coefficient of determination for each curve using least squares regression or another appropriate procedure.

8.4.2 *Sea Water Blank*—Extract and analyze as specified for test samples to establish baseline.

8.4.3 *Spiked Sea Water Samples*—Extract and analyze as specified for test samples to determine extraction efficiency. Recovery must be 90 to 110 %.

8.4.4 If changing the graphite tube during a run is necessary, the blank and standards in toluene should be analyzed to ensure proper response and linearity before continuing the sample analysis.

8.5 Determine the tin concentration of individual test samples with reference to the 50 µg/L calibrating standard analyzed immediately after those test samples.

## 9. Procedure

9.1 Organotins have a strong tendency to adsorb on certain glass or plastic surfaces. Therefore, all labware (glass or polycarbonate) used for organotin release measurements

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[7] A six-paddle stirrer, Model 300, manufactured by Whitaker Medical Mfg. Co., Phipps and Bird Div., 8741 Landmark Rd., Richmond, VA 23228, or equivalent, has been found satisfactory for this purpose.

[8] Oak Ridge Tubes, available from Cole-Parmer, or equivalent, have been found satisfactory for this purpose.

must be treated as follows: clean thoroughly by soaking in 10 % HCl or $HNO_3$ for a minimum of 6 h. Rinse labware thoroughly with distilled water and allow to dry. Cleaning can also be accomplished by soaking in concentrated HCl for ½ h. Prepare all samples, blanks, and standards in labware treated in this manner. Disposable materials (pipet tips, centrifuge tubes, etc.) do not have to be acid-washed before use.

9.2 Prepare the exposure surfaces (200 $cm^2$) of three replicate test cylinders to provide a suitable surface for adhesion of the paint system to be applied. Mask the surfaces to remain uncoated (including the bottom 1 to 2 cm of the exterior circumferential surface of the test cylinder).

9.3 Paints shall be manufactured a minimum of seven days prior to testing. Also, test paints shall not be allowed to age beyond the manufacturer's recommended shelf life. Provide typical storage conditions during aging, that is, sealed in a container commonly used for sale and held at 20 to 30°C.

9.4 Apply one or more coats of antifouling paint to the exterior circumferential surface of a test cylinder to produce a band of AF paint with an exposure surface of 200 $cm^2$ and a minimum dry film thickness of 100 μm (4 mils). Follow manufacturer's instructions with respect to mixing and drying. At a minimum, mechanically shake until the paint appears homogeneous. Apply using a sponge applicator or spray as recommended by the manufacturer. If the paint is marketed only in spray cans, then apply as a spray. After the final application allow the paint to dry for 7 ± 1 days at 23 to 27°C.

9.5 Estimate the initial dry film thickness using a suitable nondestructive method such as Method D 1212. If the leaching tests exceed 6 months measure the film thickness at the conclusion of the test. Methodology for the final measurement can be either that used for the initial measurement of Test Method D 4138. If a nonstandard method is used, make several film thickness measurements for each cylinder and estimate the variability of the determination. Film thickness should remain greater than 50 μm throughout the test. For tests of long duration, the initial thickness may need to be greater than 100 μm to maintain a thickness greater than 50 μm throughout the test.

9.6 Place all cylinders in a single batch in a holding tank after the 7-day drying period. A batch consists of one or more sets of three replicate cylinders coated with a test paint and one control (unpainted) cylinder. The painted surface on the cylinders must be completely submerged. Cylinders must be stationary and positioned so that sea water moving through the tank will flow around each cylinder.

9.7 Monitor the pH (using a pH meter with a calomel electrode) and the temperature of the synthetic sea water in the holding tank daily. Adjust the pH if necessary using either 0.1*N* NaOH or 0.1*N* HCl. Quantitate salinity every 14 days and adjust if necessary. Determine TBT concentrations weekly. When TBT levels increase, change the carbon filter before the TBT concentration exceeds 100 μg/L. More frequent monitoring of synthetic sea water may be necessary in some instances to maintain the specified environmental conditions.

9.8 After 1, 3, 7, 10, 14, 21, 24, 28, 31, 35, 38, 42, and 45 days, transfer all cylinders in given batch into individual measuring containers containing 1500 mL of fresh synthetic sea water. Randomly assign cylinders (control and painted) to measuring containers at each leaching. When transferring cylinders, lift the cylinder out of the holding tank, allow sea water to drain off, install the cylinder into the rotating device, and submerge the painted area into the sea water. Immediately start rotation of the cylinder at 60 ± 5 r/min, and continue rotation for 60 min. When transferring the cylinders, do not touch or in any way damage the paint film, and do not allow the paint surface to dry. The transfer should be completed as quickly as possible (generally, in less than 5 min).

9.9 If testing beyond the minimum (45 days) length requirement is desired, the study may be extended to 73 days. During the extended test, remove the cylinders from the holding tank every 3 to 4 days to make a measurement of the leach rate.

9.10 At the completion of the cylinder rotation, immediately remove the cylinder from the measuring tank and return it to the holding tank. Pipet a 25-mL subsample of the sea water into a 50-mL centrifuge tube containing sufficient 10 % HCl to reduce the pH to ≤4.0. If the number of samples from the leach measuring steps exceeds the daily analysis capacity, the samples may be refrigerated and stored in the acidified state in a sealed container for up to 14 days. Clean the measuring containers using appropriate procedures (distilled water or acid wash) before reuse.

9.11 Partition acidified sea water samples with 10 mL of toluene (15 min shaking on a mechanical shaker). Remove most of the toluene and wash it with 5 mL of a 3 % aqueous NaOH solution (10 min shaking). Pipet off (or separate using a separatory funnel) the organic phase and analyze for total tin content by GF-AAS. The toluene extract can be stored sealed in the dark at 4°C for up to 24 h before analysis.

## 10. Calculation

10.1 Calculate the concentration of the TBT cation in the sea water of the measuring container as follows:

$$C_{TBT} = (C \times E \times F)/S$$

where:

$C_{TBT}$ = concentration of TBT cation (μg/L),
$C$ = concentration of tin in the toluene extraction (μg/L),
$E$ = volume of toluene = 10 mL,
$F$ = correction factor to convert tin to TBT = 2.5, and
$S$ = aliquot of sea water analyzed = 25 mL.

This equation can be simplified as follows if the prescribed volume of sea water and toluene are used:

$$\begin{aligned} C_{TBT} &= (C_{Sn} \times 10 \times 2.5)/25 \\ &= C_{Sn} \end{aligned}$$

10.2 Calculate the release rate by the following formula:

$$R = (C_{TBT} \times V \times D)/(T \times A)$$

where:

$R$ = release rate (μg/$cm^2$/day),
$D$ = 24 h/day,
$V$ = 1.5 L, volume of sea water in measuring container,
$T$ = 1 h during which cylinder is in measuring container, and
$A$ = 200 $cm^2$ surface area of paint film.

This formula can be simplified as follows:

$$
\begin{aligned}
R &= (C_{\mathrm{TBT}} \times 1.5 \times 24)/(1 \times 200) \\
&= C_{\mathrm{TBT}} \times 0.18
\end{aligned}
$$

10.3 Calculate the 14-day cumulative release of TBT cation as follows:

$$R = R_1 + (2 \times R_3) + (4 \times R_7) + (3 \times R_{10}) + (4 \times R_{14})$$

where:

$R$ = 14-day cumulative release, $\mu g/cm^2 \cdot 14$ days, and

$R_1$, $R_3$, $R_7$, $R_{10}$, and $R_{14}$ = release rates for sampling days 1, 3, 7, 10, and 14, respectively, $\mu g/cm^2$ per day.

10.4 Calculate the average release rate ($\mu g/cm^2$ per day) by averaging individual release rate measurements taken from day 21 through the last day of sampling (day 45 through day 73). If values at day 21 are high and it is suspected that the release had not reached pseudo steady-state conditions, then compare the release rate at day 21 to the mean for all release rates from day 21 through the termination of testing (45 to 73 days). If the release rate exceeds the mean by two or more standard deviations, then the release rate may be excluded from the average. If the day 21 release rate is excluded, the day 24 release rate may be evaluated by the same procedure.

## 11. Report

11.1 Report the following information:

11.1.1 Report the concentration in micrograms per litre of TBT cation in the sea water of the measuring tank and the rate of TBT cation release (micrograms per square centimetre per day) for each sampling time (give values for individual replicates as well as the mean). Plot the rate of TBT release as a function of time (use linear axes). Also report the 14-day cumulative release and the average release rate.

## 12. Precision and Bias

12.1 Precision and bias for this test method have not been determined.

## 13. Keywords

13.1 antifouling coating system; organotin; release rate; TBT; tributyl tin

## APPENDIX

### (Nonmandatory Information)

## X1. GRAPHITE FURNACE OPERATING INSTRUCTIONS

X1.1 Operate the graphite furnace in accordance with the manufacturer's instructions. Tables X1.1 and X1.2 provide guidelines that have been used by two operators.

**TABLE X1.1 Navy Equipment—for Toluene Extraction**

NOTE—Light Source: Tin electrodeless discharge lamp (EDL)
Wavelength: 224.6 nm
Background Corrector: On

| Step | Temperature, °C | Ramp Time, s | Hold Time, s | Gas Flow (mL/min) |
|---|---|---|---|---|
| 1. Dry | 100 | 5 | 10 | 300 |
| 2. Dry[A] | 110 | 5 | 5 | 300 |
| 3. Char[B] | 1000 | 10 | 2 | 300 |
| 4. Atomize | 2000 | 0 | 8[C] | 0 |
| 5. Clean | 2700 | 2 | 5 | 300 |
| 6. Cool | 20 | 2 | 15 | 300 |

[A] 286.3 nm tin hollow cathode.

[B] Gas stop stage must be used after Step 2 to prevent the carrier gas sweeping organotin out of the tube.

[C] Stop gas.

**TABLE X1.2 Perkin-Elmer 4000AS[8] Fitted With A Perkin-Elmer 500 Graphite Furnace With Temperature Programmer**

NOTE—Light Source: Hallow cathode lamp
Wavelength: 286.3 nm

| Step | Temperature, °C | Ramp Time, s | Hold Time, s |
|---|---|---|---|
| 1. Chart[A] | 250 | 7 | 30 |
| 2. Ash | 900 | 7 | 30 |
| 3. Atomize | 2600 | 0 | 5 |
| 4. Clean | 2700 | 1 | 3 |

[A] Purge gas used: argon.

## X2. AUTOSAMPLER

X2.1 *Perkin-Elmer AS40 Autosampler*[9]—This autosampler has been used with the Perkin-Elmer HGA 500 graphite furnace. The autosampler introduces aliquots of 20 µL into the graphite tube. Each sample is analyzed three times and an average given. The machine automatically recalibrates after every 12 samples.

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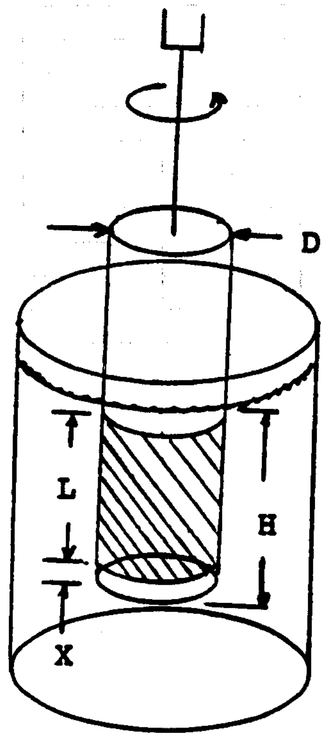
[9] Available from Perkin-Elmer Corp., 761-T Main Ave., Norwalk, CT 06859-0001, or equivalent, has been found suitable for this purpose.

## X3. SUGGESTED TABULAR REPORT FORM

| Aliquot | Day | Rotation Time, h | Measured Concentration of Tin, μg/L | | | | Avg TBT Release Rate, μg/cm$^2$ per day | Total Release Rate, μg/cm$^2$ per sample interval | Cumulative Total TBT Release Rate, μg/cm$^2$ |
|---|---|---|---|---|---|---|---|---|---|
| | | | Rep 1 | Rep 2 | Rep 3 | Average | | | |
| 1 | 1 | | | | | | | | |
| 2 | 3 | | | | | | | | |
| 3 | 7 | | | | | | | | |
| 4 | 10 | | | | | | | | |
| 5 | 14 | | | | | | | | |
| 6 | 21 | | | | | | | | |
| 7 | 24 | | | | | | | | |
| 8 | 28 | | | | | | | | |
| 9 | 31 | | | | | | | | |
| 10 | 35 | | | | | | | | |
| 11 | 38 | | | | | | | | |
| 12 | 42 | | | | | | | | |
| 13 | 45 | | | | | | | | |
| 14 | to 73 days (optional) | | | | | | | | |

## X4. DESCRIPTION OF PROPOSED TESTING APPARATUS

X4.1 A 200 $cm^2$ organotin antifouling paint film of a minimum 100 µm thickness is applied to the curved surface of a polycarbonate cylinder closed at one end. This cylinder is suspended with its closed and immersed within and concentric with a larger polycarbonate cylinder holding synthetic sea water. The coated internal cylinder is rotated about its axis at 60 ± 5 r/min in order to produce a peripheral speed of about 1 knot. In practice, the commercially available polycarbonate "multipurpose jars" described in this test method will hold 1500 mL of synthetic sea water.



NOTE 1—*Test Container Dimensions:*

| | |
|---|---|
| Capacity, L | 2 |
| Inside diameter, cm | 12.7 |
| Outside diameter, cm | 13.3 |
| Height (without cover), cm | 19 |

NOTE 2—*Rotating Test Cylinder Dimensions:*
*D* = approximately 7 cm (2½ in. nominal),
*H* = 12 cm min,
*L* = 10-cm coated section, and
*X* = 1-cm uncoated band.

**FIG. X4.1 Dimensions of Testing Apparatus (Container and Cylinder)**

**Designation: D 5139 – 90**

# Standard Specification for Sample Preparation for Qualification Testing of Coatings to be Used in Nuclear Power Plants[1]

This standard is issued under the fixed designation D 5139; 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 specification defines the size composition and surface preparation requirements for test samples used to evaluate coatings according to the following ASTM test procedures.

## 2. Referenced Documents

2.1 *ASTM Standards:*

A 36 Specification for Structural Steel[2]
C 33 Specification for Concrete Aggregates[3]
C 150 Specification for Portland Cement[4]
C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory[3]
C 260 Specification for Air-Entraining Admixtures for Concrete[3]
C 494 Specification for Chemical Admixtures for Concrete[3]
C 618 Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete[3]
D 3911 Test Method for Evaluating Coatings Used in Light-Water Nuclear Power Plants at Simulated Design Basis Accident (DBA) Conditions[5]
D 3912 Test Method for Chemical Resistance of Coatings Used in Light-Water Nuclear Power Plants[5]
D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser[6]
D 4082 Test Method for Effects of Radiation on Coatings Used in Light-Water Nuclear Power Plants[5]
D 4256 Test Method for Determination of the Decontaminability of Coatings Used in Light-Water Nuclear Power Plants[5]
D 4258 Practice for Surface Cleaning Concrete for Coating[5]
D 4259 Practice for Abrading Concrete[5]
D 4260 Practice for Acid Etching Concrete[5]
D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers[5]

2.2 *Other Standards:*

American Concrete Institute, ACI 301 Specifications for Structural Concrete for Buildings[7]
SSPC-SP-1, 2, 3, 5, 6, 7, 10, or 11[8]

## 3. Significance and Use

3.1 This specification provides uniform requirements for the preparation of test samples used for qualification testing of coatings used in nuclear power plant construction and maintenance.

## 4. Steel Samples

4.1 Sample size shall be a minimum of 2 in. wide by 4 in. long by ⅛ in. thick. Edges and corners may be rounded. A ¼ in. diameter hole suitably located may be in the test panel as appropriate for the test samples in Test Methods D 3911, D 3912, D 4082 and D 4256.

4.1.1 For Test Method D 4541 the minimum size shall be 3 in. wide by 5 in. long by ¼ in. thick.

4.2 All panels should be carbon steel, meeting the requirements of Specification A 36.

4.3 Surface preparation shall be in accordance with SSPC-SP 10 for qualification testing. Other surface preparation as required by the project or specific conditions may be used when testing for evaluation over surfaces other than SSPC-SP 10, such as SSPC-SP 1, 2, 3, 5, 6, 7, or 11.

## 5. Concrete Blocks

5.1 Applicable to Test Methods D 3911, D 3912, D 4082, D 4256 and D 4541. The minimum size shall be 2 in. by 2 in. deep by 4 in. long. The edges may be chamfered up to ¼ in. maximum.

5.2 Composition shall be as follows:

| | | |
|---|---|---|
| Cement | Specification C 150, Type II | 7 sacks/yd$^3$ |
| Gravel | Specification C 33, size ⅜ in. | 45 % by volume |
| Sand | Specification C 33 | 55 % by volume |
| Air-entraining admixture | Specification C 260 | As recommended 4 to 7 % |
| Water-reducing admixture | Specification C 494 | As recommended |
| Pozzolan | Specification C 618 | As recommended to 15 % amount |
| Water, demineralized or distilled | | 3 in. slump |

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[1] This specification is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.02 on Service and Material Parameters.
Current edition approved Dec. 12, 1990. Published January 1991.

[2] *Annual Book of ASTM Standards*, Vol 01.04.

[3] *Annual Book of ASTM Standards*, Vol 04.02.

[4] *Annual Book of ASTM Standards*, Vol 04.01.

[5] *Annual Book of ASTM Standards*, Vol 06.02.

[6] *Annual Book of ASTM Standards*, Vol 06.01.

[7] Available from American Concrete Institute, P.O. Box 19150, Detroit, MI 48219.

[8] Surface Preparation Standards are available from Steel Structures Painting Council, 4400 5th Ave., Pittsburg, PA 15213-2683.

| | | Nominal 1 Cubic Foot Batch |
|---|---|---|
| Cement | Specification C 150, Type II low alkali | 22.2 lb |
| Gravel | Specification C 33, Size ⅜ in. | 45.3 lb |
| Sand | Specification C 33 | 55.5 lb |
| Air-entraining admixture | Specification C 260 | 31 mL |
| Pozzolan | Specification C 618 | 2.2 lb |
| Water-reducing admixture (Type A) | Specification C 494 | As recommended by the manufacturer |
| Water, demineralized or distilled | | As required to produce 3-in. slump (approximately) |

5.3 Blocks shall be cast horizontally in forms using release agents that are compatible with the coatings to be used. The top surface, as cast, shall be given a broom finish, unless otherwise specified. The block surface is to be covered with plastic during the first 24 h to simulate water curing unless a curing compound is included in the testing.

5.4 The block shall be removed from the form after 24 h and wet cured in accordance with ACI 301 or Practice C 192.

5.5 If a curing compound is to be used, apply the compound to the broom finished surface immediately after finishing and to all other surfaces of the block within 2 h after its removal from the form.

5.6 Allow the block to cure for 28 days in accordance with ACI 301 before application of the coating system unless otherwise specified.

5.7 After curing, remove loose material on the broom finish surface by light wirebrushing. Remove loose material on the cast surfaces by blowing with air (80 to 100 psi) unless otherwise specified, such as Practices D 4258, D 4259, or D 4260.

5.8 All surfaces of the blocks shall be coated in accordance with Test Method D 3912. The upper and/or lower ends of the blocks shall be left uncoated for all other testing.

5.9 A suitable hanger compatible with the testing apparatus shall be affixed at the mid-point of the upper end of the block where applicable.

5.10 Large bug-holes, rock pockets and other coating defects may be simulated by drilling holes to test coating systems for patching surface defects.

## 6. Miscellaneous Materials

6.1 Follow 4.1 and 4.3 for metallic materials such as aluminum, galvanized steel, and other metals.

6.2 Follow 5.1 and 5.3 for castable materials such as grout, fireproofing, and other castables.

## 7. Abrasion Test Samples

7.1 Substrate material for abrasion testing shall be of a size in accordance with Test Method D 4060 and shall be aluminum or steel and of a thickness to maintain a flat surface after surface preparation.

7.2 Surface preparation shall be compatible with coating to be tested.

## 8. Application

8.1 All test samples shall be coated with the full coating system including any applicable fillers. The manufacturer's latest published application instructions shall be followed for mixing and coating application unless otherwise specified. Any special procedures or conditions shall be noted in the documentation including thinning, mixing, drying/curing times, force curing, aging between coats/systems, intermediate surface preparations or other special procedures or conditions.

8.2 The film thickness range shall be representative of the specified work for which the testing is being conducted.

8.3 Test samples for maintenance painting test programs may include simulated aging of the existing coating, intermediate surface preparation and wide ranges of coating film thicknesses. Field conditions should be duplicated as close as is possible including coating application methods that may differ from the manufacturer's published data.

## 9. Documentation

9.1 The procedures and conditions used for the test sample preparation shall be documented. As a minimum the documentation shall include preparation times and dates, surface preparation details, coating sequence and individual dry film thickness ranges of each coat, total thickness range, environmental conditions, and product batch identification.

9.2 Record all deviations from the procedures called for in this specification, that is, all items allowing "unless otherwise specified" that are different than those described.

## 10. Keywords

10.1 concrete blocks; nuclear power plants; qualification testing; sample preparation; steel samples; test samples

# Standard Guide for
# Use of Protective Coating Standards in Nuclear Power Plants[1]

This standard is issued under the fixed designation D 5144; 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.

## INTRODUCTION

Protective coatings (paints) have been used extensively in the nuclear industry to protect the surfaces of facilities and equipment from corrosion and contamination by radioactive nuclides in accordance with ALARA. In the absence of a standard method of selecting, testing, and evaluating coatings, many sites evaluated paints by empirical tests to determine which were useful in their particular operation. Understandably, the methods of testing were not uniform throughout the industry. It has been very difficult, consequently, to compare the results obtained at one site with those obtained at another. Standard tests whereby industrial (nuclear) users of paints systematically prepare specimens and subject them to selected evaluations, thus permitting uniform comparisons, are advantageous, internationally as well as domestically.

The designer of light water-moderated nuclear reactor systems must consider the possibility of a Design Basis Accident (DBA) and the subsequent events which might lead to the release or expulsion of a fraction of the fission-product inventory of the core to the reactor containment facility. Engineered safety features, principally a reactor containment facility, are provided to prevent the release of fission products to the biological environment during and after this improbable event. The design, fabrication, quality assurance, and testing of these engineered safety features ensure reliable operation and safety under all anticipated conditions.

Large areas of the reactor-containment facility are painted with a protective coating. If severe delamination, peeling, or flaking causes significant portions of the coating to be discharged into the common water reservoir (primary containment sump), the performance of the safety systems could be seriously compromised by the plugging of strainers, flow lines, pumps, spray nozzles, and core coolant channels.

The safety requirement for protective coatings is to ensure that failure of the coatings under DBA conditions does not adversely affect the performance of post-accident fluid systems. Therefore, coatings must be qualified to the environmental exposures anticipated during normal plant operation, and during DBA and other upset conditions.

This guide is the result of a comprehensive examination of the experience and data that have been developed on protective coatings in the nuclear industry over approximately 40 years. Standards pertaining to nuclear coatings have historically been covered by ANSI N5.12, N101.2, and N101.4. Responsibility for updating, rewriting, and issuing appropriate ANSI replacement standards has been transferred to ASTM, specifically ASTM Committee D-33, on Protective Coating and Lining Work for Power Generation Facilities.

Quality assurance in the nuclear industry is a mandatory requirement for all aspects of safety related nuclear coatings work. The objective of this guide is to provide a common basis on which to define and specify the performance requirements for the coatings which will be used in Service Level I and II areas of a nuclear facility. Proposed Regulation 10CFR50.65 (Draft Rule November 28, 1988) defines the requirements for an effective program for maintenance of nuclear power plants. This guide may be used both for coatings applied to new nuclear power plants and to facilitate implementation of the coating aspects of 10CFR50.65.

## 1. Scope

1.1 This guide provides a common basis on which protective coatings for the surfaces of light water-moderated nuclear power generating facilities may be qualified and selected by reproducible evaluation tests. This standard also provides guidance for application and maintenance of protective coatings. Under the environmental operating and accident conditions of nuclear power generation facilities, encompassing Pressurized Water Reactors (PWR's) and Boiling Water Reactors (BWR's), coating performance may be affected by exposure to any one, all, or a combination of the following conditions: ionizing radiation; contamination by radioactive nuclides and subsequent decontamination

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[1] This guide is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.02 on Service and Material Parameters.

Current edition approved Jan. 8, 1991. Published March 1991.

processes; chemical and water sprays; high-temperature high-pressure steam; and abrasion or wear.

1.2 The content of this guide includes:

1.2.1 In addition, this guide addresses technical topics within ANSI N5.12 and ANSI N101.2 that were covered by separate ASTM standards, for example, surface preparation, shop and field and coating application, and shop and field.

1.2.2 Applicable sections of this guide and specific acceptance criteria may be incorporated into specifications and other documents where appropriate.[2]

1.3 *This standard does not purport to address the safety problems 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 3843 Practice for Quality Assurance for Protective Coatings Applied to Nuclear Facilities[3]

D 3911 Test Method for Evaluating Coatings used in Light-Water Nuclear Power Plants at Simulated Design Basis Accident (DBA) Conditions[3]

D 3912 Test Method for Chemical Resistance of Coatings Used in Light-Water Nuclear Power Plants[3]

D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser[4]

D 4082 Test Method for Effects of Radiation on Coatings Used in Light-Water Nuclear Power Plants[3]

D 4227 Practice for Qualification of Journeyman Painters for Application of Coatings to Concrete Surfaces of Safety-Related Areas in Nuclear Facilities[3]

D 4228 Practice for Qualification of Journeyman Painters for Application of Coatings to Steel Surfaces of Safety-Related Areas in Nuclear Facilities[3]

D 4256 Test Method for Determination of the Decontaminability of Coatings Used in Light-Water Nuclear Power Plants[3]

D 4537 Guide for Establishing Procedures to Qualify and Certify Inspection Personnel for Coating Work in Nuclear Facilities[3]

D 4538 Definitions of Terms Relating to Protective Coating and Lining Work for Power Generation Facilities[3]

D 4541 Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers[3]

D 5139 Specification for Sample Preparation for Qualification Testing of Coatings to be Used in Nuclear Power Plants[3]

E 84 Test Method for Surface Burning Characteristics of Building Materials[5]

2.2 *Other Standards:*

ANSI N5.12 Protective Coatings (Paints) for the Nuclear Industry[6]

ANSI N101.2 Protective Coatings (Paints) for Light Water Nuclear Reactor Containment Facilities[6]

ANSI N101.4 Quality Assurance for Protective Coatings Applied to Nuclear Facilities[6]

10CFR50.65 Draft rule "Ensuring the Effectiveness for Maintenance Programs for Nuclear Reactor Plants", November 28, 1988[7]

Regulation Guide 1.54 Quality Assurance Requirements for Protective Coatings Applied to Water-Cooled Nuclear Power Plants[7]

USNRC Review Plan 6.1.2 Protective Coating Systems (Paints) Organic Materials[7]

10CFR20.1(C) Standards for Protection Against Radiation; Purpose[7]

USNRC Regulation Guide 8.8 Information Relevant to Ensuring that Occupational Radiation Exposures At Nuclear Power Stations Will Be As Low As Is Reasonably Achievable[7]

## 3. Terminology

3.1 *Definitions*—Definitions for use with this guide are shown in Definitions D 4538 or other applicable standards.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *ALARA*—the concept of reducing radiation exposure to personnel to levels "as low as is reasonably achievable," as defined in the USNRC Regulation Guide 8.8, and 10CFR20.1(C).

3.2.2 *Coating Service Level I*—areas where coating failure could adversely affect the operation of post-accident fluid systems and, thereby, impair safe shutdown. With few exceptions, *Coating Service Level I* applies to coatings inside primary containment.

3.2.3 *Coating Service Level II*—areas where coating failure could impair, but not prevent, normal operating performance. The function of *Coating Service Level II* coatings is to provide corrosion protection and decontaminability in those areas outside primary containment subject to radiation exposure and radionuclide contamination.

## 4. Significance and Use

4.1 This guide addresses the concerns of Regulation Guide 1.54 and Standard Review Plan 6.1.2, and the

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[2] Certain ASTM standards are available in compilation form (which includes this guide), as *Compilation of ASTM Standards for Use of Protective Coating Standards in Nuclear Power Plants* for expedient reference and usage by personnel involved in nuclear coating work.

[3] *Annual Book of ASTM Standards*, Vol 06.02.

[4] *Annual Book of ASTM Standards*, Vol 06.01.

[5] *Annual Book of ASTM Standards*, Vol 04.07.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[7] Available from the U.S. Government Printing Office, Washington, DC 20402.

replacement of ANSI Standards N5.12, N101.2, and N101.4. This guide covers coating work on previously coated surfaces as well as bare substrates. The entire document applies to all coating work in Service Level I areas. Applicable sections of this guide may be used to evaluate and select protective coatings for Service Level II areas.

## 5. Preparation of Test Specimens

5.1 All test samples used for qualification testing of coatings shall be prepared in accordance with Specification D 5139.

## 6. Radiation Tolerance Tests

6.1 Coating film resistance to radiation exposure shall be evaluated in accordance with Test Method D 4082.

## 7. Decontamination Test

7.1 The relative decontaminability of the coating shall be evaluated by Test Method D 4256.

7.2 Certain coatings may contaminate more readily than others, and the responses to decontamination treatments also vary. In some cases, the desired level of decontamination may be achieved merely by cleaning the coating surface; in other cases, decontamination may be achieved only by partial or complete removal of the coating. The decontamination test described gives a method of evaluating the relative ease of decontamination of a coating system. The higher the overall Decontamination Factor (DF), the easier the coating will be to decontaminate.

## 8. Physical Properties

8.1 *Adhesion*—Panels shall be tested for adhesion in accordance with Method D 4541. A minimum of two panels shall be tested for each coating system. If the size of the test specimen is less than 3 in. by 5 in., an annular bearing ring should be used to ensure full contact of the tester legs to the test specimen.

8.1.1 Minimum adhesion shall be 200 psi, consisting of three adhesion tests on each test panel.

8.2 *Abrasion Resistance*—Abrasion resistance characteristics of coating systems for floors and other surfaces where abrasion is a factor shall be determined in accordance with Test Method D 4060.

8.2.1 Weight loss shall not exceed 175 mg/1000 cycles when a CS-17 Wheel is used with a 1000-g load.

## 9. Chemical Resistance Tests

9.1 Test specimens shall be tested in accordance with Test Method D 3912.

9.2 The specific chemicals to be used should be selected to characterize the anticipated exposure; the chemicals indicated in Test Method D 3912 are shown only as examples and are not mandatory.

## 10. Fire Evaluation Tests

10.1 *Flame Spread Tests*—Flame-spread tests, when required, shall be conducted and evaluated in accordance with Test Method E 84. The permissible flame-spread and smoke generation, when tested on a noncombustible substrate, shall not exceed the limits set by the nuclear power generating facility.

10.2 The coating systems should be tested to cover the specified film thickness range (or greater) since the flame-spread and smoke density can vary with film thickness. Smoke density is significant where a coating is utilized in enclosed spaces and smoke generation can reduce visibility and prevent effective fire fighting operations. Historic test data indicates that most coatings applied at less than 25 mils dry film thickness over noncombustible substrates and tested in accordance with Test Method E 84 demonstrate flame-spread values below 25.

## 11. DBA Testing

11.1 The test specimen shall be tested and evaluated in accordance with Test Method D 3911.

## 12. Surface Preparation, Coating Application, and Inspection for Shop and Field Work

12.1 It is recommended that the *Manual of Coating Work for Light-Water Nuclear Power Plant Primary Containment and Other Safety-Related Facilities Guide*,[8] prepared by ASTM Subcommittee D01.43, be reviewed in the course of nuclear coating work.

12.2 Surface preparation for steel, concrete, and previously coated surfaces shall be at least as good as that used in the qualification testing of the coating system intended for use.

12.3 Coating application shall be in accordance with the job specifications and the coating manufacturer's latest published instructions. Journeymen painter qualification shall meet the requirements of the applicable quality assurance (QA) program, Practices D 4227 and D 4228. Coating dry film thickness shall be in the range used in the qualification testing of the coating system.

12.4 Coatings work shall be inspected by coatings inspectors qualified and certified in accordance with the applicable QA program and Guide D 4537. Inspections shall be documented to provide a record of the coatings work.

12.5 Maintenance painting work shall follow the requirements of 12.2, 12.3, and 12.4. The maintenance painting specifications shall take into consideration the plant environment in which the coating work must be accomplished. Maintenance painting work qualification testing should be based on proposed surface preparation, coating application methods, and film thickness ranges, all of which may be different than the original design qualification work.

## 13. Quality Assurance

13.1 A quality assurance program for Service Level I coating work shall be established in accordance with Practice D 3843. Quality assurance requirements may also be established for Service Level II coating work based on criticality.

## 14. Keywords

14.1 ANSI replacement standards; Design Basis Accident (DBA); decontamination; nuclear power plants; protective coating standards; qualification testing; quality assurance Service Level I and II; radiation; safety related

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[8] ASTM, 1979.

**Designation: D 5146 - 90$^{\epsilon 1}$**

# Standard Guide to Testing Solvent-Borne Architectural Coatings[1]

This standard is issued under the fixed designation D 5146; 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 ($\epsilon$) indicates an editorial change since the last revision or reapproval.

$^{\epsilon 1}$ NOTE—Editorial changes made throughout in November 1992.

## 1. Scope

1.1 This guide covers the selection and use of procedures for testing solvent-borne coatings to be used on exterior, interior or both types of surfaces (see Note 1). The properties that can be examined or, in some cases, the relevant test procedures are listed in Tables 1 and 2.

NOTE 1—The term "architectural coating" as used here combines the definition in Definitions D 16 with that in the FSCT Paint/Coatings Dictionary, as follows: "Organic coatings intended for on-site application to interior or exterior surfaces of residential, commercial, institutional, or industrial buildings, in contrast to industrial coatings. They are protective and decorative finishes applied at ambient temperatures. Often called Trade Sales Coatings." (See 2.3.)

NOTE 2—Architectural coatings that are designed to give better performance than most conventional coatings because they are tougher and more stain- and abrasion-resistant are covered by Guide D 3730.

1.2 The types of organic coatings covered by this guide are as follows:

(1) Type 1 Interior Low-Gloss Wall Finish, partly covered by Guide D 3323,

(2) Type 2 Interior Gloss and Semigloss Wall and Trim Enamels, partly covered by Guide D 3425,

(3) Type 3 Exterior House and Trim Coatings, now covered by Guide D 2932, and

(4) Type 4 Floor Enamel, Exterior and/or Interior, now covered by Guide D 3383.

1.2.1 Each is intended for application by brushing, rolling, spraying, or other means to the materials appropriate for its type, which may include wood, plaster, wallboard, masonry, steel, previously painted surfaces, and other architectural substrates.

1.3 *This standard does not purport to address all of the safety problems, 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 16 Terminology Relating to Paint, Varnish, Lacquer and Related Products[2]

D 93 Test Method for Flash Point by Pensky-Martens Closed Tester[3]

D 154 Guide for Testing Varnishes[4]

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints[4]

D 215 Practice for Chemical Analysis of White Linseed Oil Paints[2]

D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts[2]

D 358 Specification for Wood to be Used as Panels in Weathering Tests of Coatings[5]

D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings[2]

D 523 Test Method for Specular Gloss[2]

D 562 Test Method for Consistency of Paints Using the Stormer Viscometer[2]

D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive[2]

D 659 Test Method for Evaluating Degree of Chalking of Exterior Paints[6]

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints[2]

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints[2]

D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints[2]

D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints[2]

D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive[2]

D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood[2]

D 1014 Test Method for Conducting Exterior Exposure Tests of Paints on Steel[5]

D 1038 Terminology Relating to Veneer and Plywood[8]

D 1208 Test Methods for Common Properties of Certain Pigments[4]

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems[2]

D 1296 Test Method for Odor of Volatile Solvents and Diluents[7]

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[1] This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coating and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

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

[3] *Annual Book of ASTM Standards*, Vols 05.01 and 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.03.

[5] *Annual Book of ASTM Standards*, Vol 06.02.

[6] Discontinued; see *1990 Annual Book of ASTM Standards*, Vol 06.01.

[7] *Annual Book of ASTM Standards*, Vol 06.04.

[8] *Annual Book of ASTM Standards*, Vol 04.09.

D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes[5]

D 1475 Test Method for Density of Paint, Varnish, Lacquer and Related Products[2]

D 1543 Test Method for Color Permanence of White Architectural Enamels[2]

D 1554 Terminology Relating to Wood-Base Fiber and Particle Panel Materials[8]

D 1640 Test Methods for Drying, Curing or Film Formation of Organic Coatings at Room Temperature[4]

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials[2]

D 1849 Test Method for Package Stability of Paint[5]

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer[2]

D 2197 Test Method for Adhesion of Organic Coatings by Scrape Adhesion[2]

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates[2]

D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints[2]

D 2369 Test Method for Volatile Content of Coatings[2]

D 2370 Test Method for Tensile Properties of Organic Coatings[2]

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints[2]

D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints[2]

D 2486 Test Method for Scrub Resistance of Interior Latex Flat Wall Paints[5]

D 2621 Test Method for Infrared Identification of Vehicle Solids from Solvent-Reducible Paints[2]

D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings[2]

D 2698 Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High Speed Centrifuging[2]

D 2805 Test Method for Hiding Power of Paints by Reflectometry[2]

D 2932 Guide for Testing Exterior Solvent-Reducible House and Trim Coatings[5]

D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus[2]

D 3323 Guide for Testing Interior Solvent-Reducible Flat Wall Paints[5]

D 3359 Test Methods for Measuring Adhesion by Tape Test[2]

D 3383 Guide for Testing Solvent-Reducible Floor Paints[5]

D 3425 Guide for Testing Solvent-Reducible Interior Semigloss Wall and Trim Enamels[5]

D 3450 Test Method for Washability Properties of Interior Architectural Coatings[5]

D 3456 Practice for Determining by Exterior Exposure Tests Susceptibility of Paint Films to Microbiological Attack[2]

D 3730 Practice for Testing High-Performance Interior Architectural Wall Coatings[5]

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings[2]

D 3928 Test Method for Evaluation of Gloss or Sheen Uniformity[5]

D 4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method[2]

D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser[2]

D 4062 Test Method for Leveling of Paints by Draw-Down Method[5]

D 4213 Test Method for Wet Abrasion Resistance of Interior Paints[5]

D 4214 Test Methods for Evaluating Degree of Chalking of Exterior Paint Films[2]

D 4287 Test Method for High Shear Viscosity of Paints and Varnishes by the ICI Cone/Plate Viscometer[2]

D 4400 Test Methods for Sag Resistance of Paints Using a Multinotch Applicator[5]

D 4585 Practice for Testing Water Resistance of Coatings Using Controlled Condensation[2]

D 4707 Test Method for Measurement of Paint Spatter Resistance to Roller Application[5]

D 4828 Test Method for Practical Evaluation of Washability of Organic Coatings[5]

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry[9]

E 105 Practice for Probability Sampling of Materials[10]

E 313 Test Method for Indexes of Whiteness and Yellowness of Near-White Opaque Materials[2]

2.2 *U.S. Federal Standard:*

U.S. Federal Test Method Standard No. 141[11]

2112 Application by Roller
2131 Application of Sprayed Films
2141 Application of Brushed Films
3011 Condition in Container
4203 Reducibility and Dilution Stability
4401 Odor Test
4421 Absorption Test
4541 Working Properties and Appearance of Dried Film

2.3 *Other Document:*

Paint/Coatings Dictionary of the Federation of Societies for Coatings Technology[12]

## 3. Terminology

3.1 For definitions of terms in this guide refer to Terminology D 16, D 1554 and D 1038 and to the FSCT Paint/Coatings Dictionary.

## 4. Conditions Affecting Solvent-Reducible Coatings

4.1 *Interior and Exterior Coatings:*

4.1.1 *Substrate Type*—The substrate to be painted can affect not only the application properties of a coating, such as gloss and uniformity, but is also a factor in determining the

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[9] Discontinued; see *1992 Annual Book of ASTM Standards*, Vol 06.01.

[10] *Annual Book of ASTM Standards*, Vol 14.02.

[11] Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

[12] Available from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

TABLE 1 List of Standards in Sectional Order

| Property (or related test) | Section | ASTM Standard | Federal Test Method Standard 141 |
|---|---|---|---|
| Sampling | 6.2 | D 3925 | 1022 |
| Liquid Paint Properties | | | |
| Skinning | 7.1 | D 154 | 3021 |
| Condition in container | 7.2 | | 3011 |
| Coarse particles and foreign matter | 7.3 | D 185 | |
| Density or Weight per gallon | 7.4 | D 1475 | |
| Fineness of dispersion | 7.5 | D 1210 | |
| Flash point | 7.6 | D 93, D 3278 | |
| Odor | 7.7 | D 1296 | 4401 |
| Absorption | 7.8 | | 4421 |
| Colorant acceptance | 7.9 | | |
| Dilution stability | 7.10 | | 4203 |
| Package stability | 7.11 | D 1849 | |
| Coating Application and Film Formation | | | |
| Application properties | 8.1 | | 4541 |
| Brush application | 8.1.1 | | 2141 |
| Roller application | 8.1.2 | | 2112 |
| Roller spatter | 8.1.2.1 | D 4707 | |
| Spray application | 8.1.3 | | 2131 |
| Touch-up uniformity | 8.2 | D 3928 | |
| Rhelological properties | 8.3 | | |
| Consistency (Low-shear viscosity) | 8.3.1 | D 562 | |
| Rheological properties of non-Newtonian liquids | 8.3.2 | D 2196, D 4287 | |
| Sag resistance | 8.3.3 | D 4400 | 4494 |
| Levelling properties | 8.3.4 | D 4062 | |
| Drying properties | 8.4 | D 1640 | 4061 |
| Appearance of Dry Film | | | |
| Color differences by visual comparison | 9.1 | D 1729 | |
| Color differences using instrumental measurement | 9.2 | D 2244 | |
| Directional reflectance | 9.3 | E 97 | |
| Gloss | 9.4 | | |
| Gloss, 60° | 9.4.1 | D 523 | |
| Sheen (85° gloss) | 9.4.2 | D 523 | |
| Hiding power | 9.5 | D 344, D 2805 | |
| Yellowness index | 9.6 | E 313 | 6131 |
| Properties of Dry Film | | | |
| Interior and Exterior Coatings | 10.1 | | |
| Abrasion resistance | 10.1.1 | D 658, D 968, D 4060 | 6192 |
| Adhesion | 10.1.2 | D 2197, D 3359 | |
| Flexibility | 10.1.3 | D 522, D 2370 | 6221[A] |
| Resistance to household chemicals | 10.1.4 | D 1308 | |
| Interior Coatings | 10.2 | | |
| Color change of white enamels | 10.2.1 | D 1543 | 6192 |
| Washability and cleansability | 10.2.2 | | |
| Washability | 10.2.2.1 | D 2486, D 4213 | |
| Cleansability | 10.2.2.2 | D 3450, D 4828 | 6141[B] |
| Exterior Coatings | 10.3 | | |
| Blister resistance | 10.3.1 | D 4585 | |
| Exposure resistance | 10.3.2 | D 1006, D 1014 | |
| Chalking | 10.3.2.2 | D 659, D 4214 | |
| Checking | 10.3.2.3 | D 660 | |
| Cracking | 10.3.2.4 | D 661 | |
| Erosion | 10.3.2.5 | D 662 | |
| Flaking | 10.3.2.6 | D 772 | |
| Mildew resistance | 10.3.3 | D 3456 | [C] |
| Fume resistance | 10.3.4 | | |
| Coating Analysis | | | |
| Chemical analysis | 11.1 | D 215 | |
| Volatile content | 11.2 | D 2369 | |
| Nonvolatile volume content | 11.3 | D 2697 | |
| Water content | 11.4 | D 1208, D 4017 | 4081 |
| Pigment content | 11.5 | D 2371 | 4021 |
| Pigment analysis | 11.6 | D 215 | 7261 |
| Nonvolatile vehicle content | 11.7 | D 215 | 4053 |
| Vehicle separation | 11.8 | D 2372 | |
| Nonvolatile vehicle identification | 11.9 | D 2621, D 2245 | |

[A] Equivalent only to Method B of Test Method D 522.

[B] Except for scrub medium.

[C] 6271 is *not* equivalent.

type of coating to use. For instance, low-gloss wall finishes do not have the abrasion resistance required on floors, whereas finishes intended only for interior service probably do not have adequate resistance to weather factors. Other factors are the type and quality of wood or wood composite (plywood, particle board or hardboard), the type, quality and alkalinity of concrete, plaster and joint cement systems, and the type and condition of any previous coatings.

4.1.2 *Substrate Conditions*—Conditions such as porosity, hardness or, in the case of unpainted concrete, alkalinity determine the kind of coating that can be applied. The condition of previously painted substrates, such as degree of chalk, presence of grease, dirt, and mold, film adhesion and porosity, and the general condition of the previous coating, all influence the performance of coatings. Smoothness of the substrate affects the spreading rate, final appearance, and texture.

4.1.3 Preparation of previously painted substrates including detergent cleaning, solvent cleaning, and sanding.

4.1.4 Type and quality of primer or undercoat and time of drying before topcoating.

4.1.5 Environmental conditions such as temperature and humidity at the time of coating application and during drying.

4.2 *Exterior Finishes:*

4.2.1 *Substrate Aspects of the Building*—If construction defects or defects due to age are such that excessive moisture from the inside or the outside makes its way through the substrate or if the substrate is in direct contact with damp ground, blistering, flaking or peeling may result.

4.2.2 Environmental conditions after application, both general for the area and specific, such as under eaves, behind shrubbery, northside and southside exposure.

## 5. Selection of Tests

5.1 Because the conditions to which a coating is subjected vary with (*a*) the surface type: wall, floor, ceiling, and (*b*) the service environment: exterior or interior, specialized types of solvent-borne coatings have been developed for the different locations. The recommended test methods presented in Tables 1 and 2 cover practically all the properties of solvent-reducible coatings but all of them are not required with each type. Coatings intended for exterior use only or both exterior and interior use require certain properties not relevant to those for interior use only. Selection of the methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and the seller.

5.2 The purchaser should first determine the properties a coating should have and then select only those test methods that measure or evaluate those properties. After selecting the desired tests, the purchaser should then decide which properties are the most important and establish the requirements or specifications accordingly. Since coating properties frequently tend to oppose each other, such as low sheen versus good cleansability, some properties may need to be less emphasized if others are to be accentuated. This balance of properties must be considered when selecting the tests and establishing the requirements. The significance of the tests and the normal range of values are presented in the different sections, in most cases.

5.3 This guide does not indicate relative importance of the various tests nor does it recommend specific test values because properties very important to one purchaser may be less so to another.

## 6. Sampling

6.1 Prior to sampling, the condition of the container should be checked since damage to it may cause evaporation, skinning, or other undesirable effects on the coating.

6.2 Sample in accordance with Practice D 3925. Determine the density in pounds per gallon (kilograms/litre) in accordance with Test Method D 1475. Continue sampling and determining density until successive results agree within 0.1 lb (45 g) or as agreed upon between the purchaser and seller. Then take samples for testing.

6.3 Specify the amount required for a representative sample, the package sizes, and an identification code. A 1-U.S. gal (or 4-L) sample is usually sufficient for the recommended tests, but for guidance in selecting a sampling plan consult Practice E 105.

## 7. Liquid Coating Properties

7.1 *Skinning*—Coatings that contain a binder that dries by oxidation may be subject to skin formation in a partially-filled can. Since skins are insoluble in the material they must be removed before use. The referenced test in a partially-filled container indicates the tendency of the material to skin. A typical minimum time for skinning in accordance with this method is 48 h. Examine the original sample for skins, both on and below the surface. Using a well-mixed skin-free portion of the sample, perform a skinning test in accordance wtih Guide D 154.

7.2 *Condition in Container*—Thickening, settling, and separation are undesirable and objectionable if material that has been stored cannot be readily reconditioned and made suitable for application with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties after storage and lists characteristics that are undesirable and objectionable in a stored paint. Determine condition in the container in accordance with Method 3011 of Federal Test Method Standard No. 141. (See also 7.13, Package Stability.)

7.3 *Coarse Particles and Foreign Matter*—Liquid coatings must be free of coarse particles and foreign matter to be able to form uniform films of good appearance, a typical maximum being 0.5 weight % of the total material. The referenced method with a 325-mesh (45-μm) screen gives the percent of these particles. Determine content of coarse particles and foreign matter in accordance with Test Methods D 185.

7.4 *Density or Weight per Gallon*—The density measured in pounds per gallon (kilograms per litre = g/mL) is used to ensure product uniformity from batch to batch, provides a check against the theoretical weight calculated from the formula, and is useful for determining the similarity of two samples. The referenced method gives a procedure for measuring the density of the coating at a specified temperature. Most interior paints have densities of about 10 to 12 lb/gal (1.2 to 1.4 kg/L). Determine density in accordance with Test Method D 1475, using a calibrated weight per gallon cup.

7.5 *Fineness of Dispersion*—Generally, the more finely a pigment is dispersed, the more efficiently it is being utilized. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the liquid coating down a calibrated tapered groove varying in depth from 4 to 0 mils (100 to 0 μm) (0–8 Hegman units). The depth at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the wet film is taken as the fineness of dispersion value. Higher readings in Hegman units or lower readings in mils or micrometres indicate finer dispersion. Low sheen finishes may have a dispersion value of 2 mils (50 μm or 4 Hegman) while gloss enamels might be near zero (8 Hegman) indicating that the pigment agglomerates are too small to be detected by the referenced method. Determine fineness of dispersion in accordance with Test Method D 1210.

7.6 *Flash Point*—Organic solvents used in these coatings have characteristic temperatures at which they support combustion. This temperature is known as the flash point and is often used for danger classification in shipping by common carrier. It is also used to determine conditions of storage to meet fire regulations and the safety requirements of the U.S. Occupational Safety and Health Act (OSHA). Determine flash point in accordance with Test Methods D 93, Part B, or D 3278.

7.7 *Odor*—Some solvent combinations produce obnoxious odors, particularly when painting indoors with inadequate ventilation and at elevated temperatures. Interior solvent-borne coatings usually contain low-odor or odorless mineral spirits. Nevertheless they should be evaluated to ensure that they are acceptable. Test for odor in accordance with Method 4401 of Federal Test Method Standard No. 141. Although not specifically designed for liquid coatings Test Method D 1296 may be used with the solvent-reducible type.

7.8 *Absorption*—On porous surfaces, binder penetration can result in pigment volume concentration changes as the film dries. This may cause appearance to vary. The referenced method provides a rough measure of the wetting and penetrating properties of the binder on a porous surface. Determine the absorption in accordance with Method 4421 of Federal Test Method Standard No. 141.

7.9 *Colorant Acceptance*—Tintability of white bases with colorants of standardized tinting strength is a trade requirement. If tinting colors are not adequately compatible with tint bases, lighter, darker, or nonuniform shades of colors are produced. There is no accepted ASTM test method at present. Test methods may be agreed upon between the purchaser and seller.

7.10 *Dilution Stability*—Dilution with a specified thinner shows whether the materials are compatible and whether the reduced coating is stable. Consequently the suggested diluent should be readily incorporated into the coating without excessive stirring or shaking. The referenced method evaluates the stability of the material that has been reduced by a given amount or to a specified viscosity. Determine dilution stability in accordance with Method 4203 of Federal Test Method Standard No. 141.

7.11 *Package Stability*—Since paints cannot normally be used immediately after manufacture, they must remain stable in the can for some time. At normal temperatures most paints can be stored for over a year with little change in properties. Although indications of long term package stability can usually be obtained in several days or weeks at an elevated temperature, such as 122°F (50°C) or 140°F (60°C), occasionally the results of the accelerated test do not agree with those at prolonged normal storage conditions. The referenced method predicts the change in consistency and certain other properties of packaged paint when stored at temperatures above freezing. Determine package stability in accordance with Test Method D 1849.

## 8. Coating Application and Film Formation

8.1 *Application Properties*—Application or working properties of a paint are generally compared to a standard or described by requirements in the product specification. Determine working properties in accordance with Method 4541 of Federal Test Method Standard No. 141.

8.1.1 *Brush Application*—Brushed films should be smooth and free of seeds and on vertical surfaces should show no sagging, color streaking, or excessive brush marks. Brush drag should not be excessive although some degree of drag may be desirable for adequate film thickness application. Wall finishes are tested on vertical surfaces and floor enamels on horizontal surfaces, although evaluation of the latter on vertical surfaces may be necessary to determine performance on stair risers, railings, posts, etc. The referenced method covers a means for the determination of the brushing properties of a coating. Even though the test is subjective someone experienced in the art can produce quite consistent results, particularly in the evaluation of drag qualities. Determine the brushing properties in accordance with Method 2141 of Federal Test Method Standard No. 141.

8.1.2 *Roller Application*—Both wall and floor coatings are frequently applied by roller. This type of application tends to produce some stipple pattern. The referenced method covers the evaluation of a material's characteristics when applied by roller. Determine roller coating properties in accordance with Method 2112 of Federal Test Method Standard No. 141.

8.1.2.1 Some coatings spatter more than others when applied by roller. The degree to which a paint spatters when roller applied can be determined by the density of the spatter. In the referenced method a specially designed notched spool is rolled through a film of the test material that has been applied to a plastic panel. Any spatter generated falls upon a catch paper and after drying is rated against photographic standards. This procedure eliminates the influence of the roller cover, thus determining the spattering characteristics of the paint alone. Determine spatter resistance in accordance with Test Method D 4707.

8.1.3 *Spray Application*—Architectural coatings are sometimes applied by spray. Both air and airless spray are used on commercial work. Determine the spray application properties in accordance with Method 2131 of Federal Test Method Standard No. 141. Manual application is very subjective and should be performed only by an individual skilled in the art of using spray equipment.

8.2 *Touch-Up Uniformity*—After a paint has dried, areas where less material was applied sometimes become noticeable. If the paint has suitable touch-up properties, additional material can be applied to these areas only, instead of refinishing the complete wall. The color, gloss, and levelling of the touched-up areas and the previously coated area should be uniform. Differences in these properties are often caused by short wet-edge time, poor levelling on recoat, and pigment orientation or flotation during and after application. Determine touch-up properties in accordance with Test Method D 3928.

8.3 *Rheological Properties:*

8.3.1 *Consistency (Low-Shear Viscosity)*—Consistency is important, relating to application and flow, and should fall within a stated range for satisfactory reproduction of a specific formula. While consistency is an important property it does not determine the quality of a coating and should be used mainly to ensure product uniformity. In the referenced method, consistency is defined as the load in grams to produce a specified rate of shear. The load value is frequently converted to Krebs units (KU) and the Stormer Consistency reported on that basis. Although the consistency of most solvent-borne house and trim coatings is about 150 to 300 g/100 revolutions (72 to 95 KU), a much wider range is possible because of the great variation that may occur in the rheological properties of these paints. Enamels for professional painters are usually formulated at a higher consistency range than consumer enamels. Typical ranges are 75 to 90 KU for consumer enamels and 90 to 100 for professional painter enamels. Two paints of the same consistency may have quite different rheological properties during application. Determine the consistency in accordance with Test Method D 562.

8.3.2 *Rheological Properties of Non-Newtonian Materials*—Rheological properties are related to application and flow characteristics of the liquid coating. The referenced methods cover the determination of rheological properties and are particularly suited for coatings that display thixotropic characteristics. They actually measure viscosity under different shear rates. In Test Method D 4287 the rate is similar to that occurring during brush application so that the measured viscosity is related to brush drag, spreading rate and film build. Determine rheological properties in accordance with Test Methods D 2196 or D 4287, or both.

8.3.3 *Sag Resistance*—Some coatings sag and form curtains before the film sets. This is an important property particularly for semigloss and gloss enamels because sagging results in unsightly film appearance. Measure sag resistance in accordance with Test Methods D 4400.

8.3.4 *Levelling Properties*—Levelling is an important property when smooth, uniform surfaces are to be produced, as it affects hiding and appearance. The referenced method covers the relative levelling characteristics of liquid coatings. Determine levelling in accordance with Test Method D 4062.

8.4 *Drying Properties*—The drying time of a coating is important in determining when a freshly painted room, floor or stair may be put back in use. Slow drying may result in dirt or insect pickup resulting in a poor appearance or, if on an exterior surface, rain or dew may cause a nonuniform appearance. The drying time of a coating is determined by its composition and by atmospheric conditions during drying. Typical drying times for enamels are ½ to 2 h set-to-touch and 18 h dry hard under normal temperature and humidity conditions. Some coatings lose drying speed during storage in the container. Any of the several methods for determining the various stages of film formation in the drying or curing of organic coatings may be used. For example, if two coats are specified the determination of "dry-to-recoat" time is important. Determine appropriate drying time(s) in accordance with Test Methods D 1640.

## 9. Appearance of Dry Film

9.1 *Color Differences by Visual Comparison*—Visual comparison of colors is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric, and geometric characteristics of light source, illuminating and viewing conditions, sizes of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials relative to their standards. Determine color difference in accordance with Practice D 1729.

9.2 *Color Differences Using Instrumental Measurements*—The difference in color between a product and its standard can be measured by instrument. Generally the tolerance is agreed upon by the purchaser and seller and may also be required if a product specification is involved. Color measuring instruments provide numerical values that can be compared to subsequent measurements. The referenced method covers the calculation of instrumental determinations of small color differences observable in daylight illumination between nonfluorescent, nonmetameric, opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation (9.1) should be used to verify the results. Calculate in accordance with Test Method D 2244 the color differences that have been measured instrumentally.

9.3 *Directional Reflectance*—This property is a measure of the appearance of lightness of a coating. It is usually assigned a value in specifications for white and pastel shades, a typical minimum reflectance value being 86 % for white wall paint. In the referenced method the directions of illumination and viewing are specified so as to eliminate the effect of gloss. Determine daylight directional reflectance in accordance with Test Method E 97.

9.4 *Gloss*—This property is a measure of the capability of a coating surface to reflect light in a mirror-like (specular) manner, that is, light strikes the surface and is reflected at the equal but opposite angle. In the referenced method the numerical gloss units are the ratio of light reflected by a specimen to that reflected by the primary standard black glass that is assigned a gloss value of 100. The gloss of some coatings varies greatly with the angle of incidence so that a complete description of their gloss would require measurements over a wide range of angle. In practice, the gloss of architectural finishes is adequately characterized by measurements at 60° or 85°, or both, from a line perpendicular (normal) to the surface. The 85° angle is a very low ("grazing") angle (5°) of illuminating and viewing the surface and the gloss at this angle is called "sheen". Attempts to standardize the levels of gloss associated with the several descriptive terms have not been very successful since the gloss scale is continuous with no distinct boundaries. Hence, there is some overlap at the ends of some gloss classifications in common usage.

9.4.1 *Gloss, 60°*—Semigloss enamels are particularly sensitive to enamel hold-out of primers and undercoats. Low or uneven gloss readings are indicative of this defect. Oil house paints are typically in a 60° gloss range from 30 to 70 while trim enamels are from 70 to 90. Floor enamels generally have a high (90+) gloss reading when first applied but this decreases with time and traffic. Interior semigloss enamels after drying 48 h are typically in the range from 40 to 70 but measurements taken shortly after drying should be repeated after one week because the gloss can drop considerably in the first few days of drying. Determine the 60° gloss in accordance with Test Method D 523.

9.4.2 *Sheen (85° Gloss)*—Although low-gloss paints with good uniformity of appearance at low angles of viewing often have little sheen while those with good cleansability usually have moderate sheen, this is not always the case so that sheen should not be used as a measure of other paint properties. Most flat wall paints have a sheen of about 1 to 10. The referenced method, using the 85° geometry, is useful in characterizing the low-angle appearance of low-gloss coatings. Determine the sheen (85° gloss) in accordance with Test Method D 523.

NOTE 3—This property is of particular interest with flat to low-gloss finishes.

9.5 *Hiding Power*—Hiding power is a measure of the ability of a coating to obscure the substrate and is usually expressed as the spreading rate for a specified level of opacity. It is, however, dependent on uniformity of film thickness, which in practical applications is influenced by flow, levelling and application properties of the coating. Test Method D 2805 is precise and gives an absolute rather than a comparative result. Paint is applied with an applicator bar to minimize the effects of flow and levelling, film thickness is rigorously measured, and film opacity is determined instrumentally. Test Method D 344 is a practical test in which paint is applied with a brush, wet-film thickness is approximately controlled by spreading rate, and hiding power is evaluated visually by comparison with a standard paint, but results are affected by flow and levelling of the materials. Determine hiding power in accordance with Test Methods D 344 or D 2805.

9.6 *Yellowness Index*—The referenced method is used for white or near white specimens to determine color departure from white toward yellow when first applied. Determine the yellowness index in accordance with Method E 313. (See also 10.2.1.)

## 10. Properties of the Dry Film

10.1 *Interior and Exterior Coatings:*

10.1.1 *Abrasion Resistance*—Abrasion resistance is a measure of the ability of a dried film to withstand wear from foot traffic and marring from objects rolled or pulled across the surface. In the referenced methods, dry abrasive is applied to a coated panel using the force of gravity or a jet blast for free-flowing abrasive or a weighted wheel for abrasive embedded in a resilient rubber matrix. Determine dry abrasion resistance in accordance with Test Methods D 658, D 968 or D 4060. (See 10.2.2.1 for wet abrasion resistance.)

NOTE 4—Because of the poor reproducibility of abrasion test methods, testing should be restricted to only one laboratory when numerical abrasion resistance values are to be used. Interlaboratory agreement is improved significantly when rankings are used in place of numerical values.

10.1.2 *Adhesion*—Adhesion, the ability of a film to resist removal from the substrate, is an important property of a coating. Determine adhesion in accordance with Test Method D 2197 or D 3359, or both.

10.1.3 *Flexibility*—Elongation is a measure of the flexibility of a coating film. Generally, gloss house paints and trim enamels have no problems in passing a mandrel bend test at ⅛ in. (3.2 mm). However, interior flat and eggshell finishes may pass only a ½-in. (12.7-mm) bend. For exterior coatings Test Method D 2370 is a much more discriminating method.[13] Determine flexibility in accordance with Methods D 522 or elongation with D 2370.

10.1.4 *Resistance to Household Chemicals*—An important property of interior coatings is the ability to resist spotting, softening or removal when subjected to household chemicals or strong cleaners. Determine resistance to these chemicals in accordance with Test Method D 1308.

10.2 *Finishes:*

10.2.1 *Color Change of White Architectural Enamels*—Color permanence is an important characteristic for interior white enamels. Lack of permanence is usually caused by after-yellowing. The referenced method attempts to accelerate color change by exposing coatings to dry sulfur dioxide. Determine resistance to color change in accordance Test Method D 1543.

10.2.2 *Washability and Cleansability*—The capability of satisfactorily removing marks without damaging the film is essential for good performance of interior finishes. A coating may be washable, that is, unaffected by the detergent solution, but may not have good cleansability. Frequently the difference between the two terms "cleansability" and "washability", is not clearly understood so that there is confusion as to what is really being tested; for example, the title of Test Method D 3450. Cleansability is evaluated by applying one or more stains and soils and determining how readily they are removed. Washability is evaluated by determining the resistance of the film to wet erosion either by visual assessment or measured film loss. In general, the precision of both types of test is poor because several properties, such as hardness, water and detergent resistance, cohesion and adhesion, are involved and the end point, except for the wet abrasion method, is rather indefinite.

10.2.2.1 *Washability (Also referred to as Scrubbing or Wet Abrasion Resistance)*—The scrubbing method, Test Method D 2486, developed for interior latex flat wall paints can be applied to coatings of almost any type. In it the coating is applied to a black plastic panel that, during scrubbing with a nylon brush and abrasive cleaning agent, is raised by a narrow shim to concentrate the test area. The number of back-and-forth strokes (cycles) required to remove the film over the shim is determined. The wet-abrasion method, Test Method D 4213, is similar except that a sponge is used in place of the bristle brush while the shim is not used. This method also provides for the use of a nonabrasive medium with paints having very low abrasion resistance. The weight

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[13] H. E. Ashton, "Flexibility and its Retention in Clear Coatings Exposed to Weathering", *Journal of Coatings Technology*, Vol 51, No. 653, June 1979, p. 41.

or volume loss per 100 cycles to erode the film almost to exposure of the black substrate is the measure of scrub resistance. Evaluate washability, as described just, in accordance with Test Methods D 2486 or D 4213.

10.2.2.2 *Cleansability*—The older referenced method, Test Method D 3450, is similar to the wet-abrasion method, Test Method D 4213, except that the sponge is used with either the nonabrasive or abrasive cleaning agent to remove a carbon black-oil stain. The ability to remove the stain is expressed as the ratio (in percent relative) of the reflectance of the cleaned area to that of the area before application of the stain. In Test Method D 4828, referred to as a "practical" test, numerous staining and soiling agents found in service and commercial abrasive or nonabrasive cleaners as well as the standardized cleaning agents can be used. In the current edition the films are cleansed only manually with the result that the method is not suitable for interlaboratory testing. Evaluate ease of removability in accordance with Test Methods D 3450 or D 4828.

10.3 *Exterior Coatings:*

10.3.1 *Blister Resistance*—Blister resistance is the ability of a dry film on wood to resist the formation of blisters caused by water from the wood substrate. In practice water can come from either the interior of a home or from structural defects that permit entry of exterior water behind the wood. Moisture blister resistance can be qualitatively evaluated in a laboratory test. Determine resistance to moisture blistering in accordance with Practice D 4585.

10.3.2 *Exposure Resistance*—If the coating is intended for exterior use, evaluation of the resistance to weathering may be required. In conducting exterior exposures follow Practice D 1006 for wood substrates or Test Method D 1014 for steel.

10.3.2.1 In establishing exterior performance on wood, use the panels described in Specification D 358.

10.3.2.2 *Degree of Chalking*—Determine the rating using Methods D 659 or D 4214.

10.3.2.3 *Degree of Checking*—Determine the rating using Test Method D 660.

10.3.2.4 *Degree of Cracking*—Determine the rating using Test Method D 661.

10.3.2.5 *Degree of Erosion*—Determine the rating using Test Method D 662.

10.3.2.6 *Degree of Flaking*—Determine the rating using Test Method D 772.

10.3.3 *Mildew Resistance*—Virtually all exterior paints are subject to microbiological discoloration on the surface with time. This is especially true in warm, moist climates. Determine mildew resistance in accordance with Practice D 3456.

10.3.4 *Fume Resistance*—Some paints exhibit a change in appearance (usually color) when exposed to air containing certain sulfur compounds, notably hydrogen sulfide and sulfur dioxide. This type of atmosphere may be present near industrial or other polluted areas and can cause paint to yellow or darken in as little time as overnight. There are no ASTM or Federal test methods for evaluating this color change, but one procedure used by the industry is as follows:

10.3.4.1 Apply a sufficient number of coats of the paint to two glass plates to hide the surface completely, allow to dry for 6 h and expose one in a moist atmosphere of hydrogen sulfide for 18 h. Compare the color with the unexposed plate. The color difference should not exceed that between plates that have been coated with a paint made with titanium dioxide pigment, lead-free zinc oxide, raw or refined linseed oil, and sufficient cobalt added for drying, and similarly treated.

## 11. Coating Analysis

11.1 *Chemical Analysis*—If a specification requires certain raw materials or certain components in a given amount then analysis is needed to determine whether the specified components are present and in what amounts. Analysis is primarily a measure of uniformity and does not necessarily establish quality that can also be greatly affected by manufacturing techniques. No single schematic analysis is comprehensive enough to cover the wide variety of paint compositions. Select test procedures from Practice D 215 and other ASTM methods that are pertinent to the components of solvent-borne coatings.

11.2 *Volatile Content*—The percent of volatile matter is a measure of the amount of a film lost as it dries. This quantity is not necessarily indicative of the quality of the coating. It is useful, however, for determining the similarity of two batches. The referenced method covers the determination of the volatile content of solvent- and water-reducible coatings. The quantity determined subtracted from 100 % gives the nonvolatile content of the coating. Determine the volatile content in accordance with Test Method D 2369.

11.3 *Nonvolatile Content (Volume Percent)*—The nonvolatile content by volume is a useful figure in calculating coverage or spreading capacity per gallon (or litre) at a specified dry-film thickness. Determine nonvolatile content volume percent in accordance with Test Method D 2697.

11.4 *Water Content*—If too much water is incorporated in a solvent-reducible coating it may retard the drying at high relative humidities. The amount of water in a coating is required in the calculation of the volatile organic content (VOC) of coatings. One referenced method covers the determination of water in paint and related materials by distilling with a volatile solvent. The newer method utilizes the Karl Fischer reaction. Determine water in accordance with Test Methods D 1208 or D 4017.

11.5 *Pigment Content*—Pigment provides the hiding and color and influences many other properties of a coating. The referenced method describes the procedure for the quantitative separation of the vehicle from the pigment in solvent-reducible coatings. It is used to measure the weight percent pigment in the paint. Determine the percent pigment content in accordance with Test Method D 2371.

11.6 *Pigment Analysis*—The analysis of pigment may be required if the product is covered by a specification or upon agreement between the purchaser and seller. Analyze the pigment in accordance with selected test procedures from Practice D 215 and other appropriate ASTM methods.

11.7 *Nonvolatile Vehicle Content*—The nonvolatile vehicle is the film-forming portion of a coating excluding the pigment. Water, volatile thinner, and pigment are determined and their sum subtracted from 100 % to give the binder content in accordance with Practice D 215. The vehicle may be separated for further analysis.

11.8 *Separation of Vehicle*—The recommended procedure describes this separation for certain solvent-borne coatings. If desired or required, separate the vehicle in accordance with Method D 2372.

11.9 *Identification of Nonvolatile Vehicle*—The type of binder used in a coating has a great influence on its properties. The referenced method covers the qualitative characterization or identification by infra-red spectroscopy of separated nonvolatile vehicle. It is useful in detecting uniformity, batch to batch, and the presence of adulterants. Identify the nonvolatile vehicle in accordance with Method D 2621. The composition of this vehicle can be further broken down into the types of oils present. If desired determine the identity of oils in accordance with Method D 2245.

**TABLE 2 Alphabetical List of Properties**

| Property (or related test) | Section | ASTM Standard | Federal Test Method Standard 141 |
|---|---|---|---|
| Abrasion Resistance | 10.1.1 | D 658, D 968, D 4060 | 6192 |
| Absorption | 7.10 | | 4421 |
| Adhesion | 10.1.2 | D 2197, D 3359 | |
| Analysis, chemical | 11.1 | D 215 | |
| Application properties | 8.1 | | 4541 |
| Blister resistance | 10.3.1 | D 4585 | |
| Brush application | 8.1.1 | | 2141 |
| Chalking | 10.3.2.2 | D 659, D 4214 | |
| Checking | 10.3.2.3 | D 660 | |
| Cleansability | 10.2.2.2 | D 3450, D 4828 | 6141[A] |
| Coarse particles and foreign matter | 7.3 | D 185 | |
| Colorant acceptance | 7.11 | | |
| Color change | 10.2.1 | D 1543 | 6192 |
| Color difference by visual comparison | 9.1 | D 1729 | |
| Color difference using instrumental measurement | 9.2 | D 2244 | |
| Condition in container | 7.2 | | 3021 |
| Consistency | 7.6 | D 562 | |
| Cracking | 10.3.2.4 | D 661 | |
| Density or weight per gal | 7.4 | D 1475 | |
| Dilution stability | 7.12 | | 4203 |
| Drying properties | 8.4 | D 1640 | 4061 |
| Erosion | 10.3.2.5 | D 662 | |
| Exposure resistance | 10.3.2 | D 1006, D 1014 | |
| Fineness of Dispersion | 7.5 | D 1210 | |
| Flaking | 10.3.2.6 | D 772 | |
| Flash point | 7.8 | D 93, D 3278 | |
| Flexibility | 10.1.3 | D 522, D 2370 | 6221[B] |
| Fume resistance | 10.3.4 | | |
| Gloss | 9.4 | | |
| Gloss, 60° | 9.4.1 | D 523 | |
| Hiding power | 9.5 | D 344, D 2805 | |
| Levelling properties | 8.3 | D 4062 | |
| Mildew resistance | 10.3.3 | D 3456 | [C] |
| Nonvolatile vehicle content | 11.7 | D 215 | 4053 |
| Nonvolatile vehicle identification | 11.9 | D 2621, D 2245 | |
| Nonvolatile volume content | 11.3 | D 2697 | |
| Odor | 7.9 | D 1296 | 4401 |
| Package stability | 7.13 | D 1849 | |
| Pigment analysis | 11.6 | D 215 | 7261 |
| Pigment content | 11.5 | D 2371 | 4021 |
| Reflectance, directional | 9.3 | E 97 | |
| Resistance to household chemicals | 10.1.4 | D 1308 | |
| Rheological properties of non-Newtonian liquids | 7.7 | D 2196, D 4287 | |
| Roller application | 8.1.2 | | 2112 |
| Roller spatter | 8.1.2.1 | D 4707 | |
| Sag resistance | 8.4 | D 4400 | 4494 |
| Sampling | 6.2 | D 3925 | 1022 |
| Sheen (85° gloss) | 9.4.2 | D 523 | |
| Skinning | 7.1 | D 154 | 3021 |
| Spray application | 8.1.3 | | 2131 |
| Touch-up uniformity | 8.2 | D 3928 | |
| Vehicle separation | 11.8 | D 2372 | |
| Volatile content | 11.2 | D 2369 | |
| Washability | 10.2.2.1 | D 2486, D 4213 | |
| Water content | 11.4 | D 1208, D 4017 | 4081 |
| Yellowness index | 9.6 | E 313 | 6131 |

[A] Except for scrub medium.

[B] Equivalent only to Method B of D 522.

[C] 6271 is *not* equivalent.

**Designation: D 5150 – 92**

# Standard Test Method for Hiding Power of Architectural Paints Applied by Roller[1]

## 1. Scope

1.1 This test method measures the ability of a paint to hide or obscure a surface to which it has been applied by a practical application procedure. This test method covers the use of a paint roller, but the concept is expected to work equally well when the application tool is a paint brush or paint pad.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address the safety problems, 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 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products[2]

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products[2]

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates[2]

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials[2]

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings[2]

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry[3]

## 3. Terminology

3.1 *Definition:*

3.1.1 *hiding power—n,* the ability of a paint or paint material as used to hide or obscure a surface to which it has been uniformly applied (see Terminology D 16).

## 4. Summary of Test Method

4.1 The test paint is applied with a roller at a specified spreading rate onto a test chart consisting of a series of light to dark grey stripes. After drying, a second coat is applied in the same manner over half of the chart. After the second coat has dried, both the single- and double-coated areas are evaluated for hiding as judged by the darkest stripe in each area that is completely or almost completely obscured.

## 5. Significance and Use

5.1 Laboratory hiding power measurements of architectural coatings generally employ blade-type applicators that lay down films of highly uniform thickness. But practical applicators, such as rollers, pads, and brushes, typically apply films that lack uniformity due to incomplete leveling, resulting in the practical hiding power of most paints being less than that measured on films applied with a drawdown blade. This test method simulates practical application procedures and conditions so as to provide an indication of the actual hiding performance obtainable when a paint is applied by an experienced worker. It is not intended to duplicate painting as done by the average consumer.

5.2 Since the rheological characteristics of a paint and its interaction with the applicator are influencing factors, rank order correlation between this test and one done by drawdown might not be obtained.

## 6. Apparatus

6.1 *Balance,* accurate to 0.1 g.

6.2 *A Weight/Gallon Cup,* or any other type of pycnometer suitable for determining paint density to 0.1 lb/gal.

6.3 *Paint Roller,* 3-in. (75-mm), frame, preferably one per paint to be tested.

6.4 *Paint Roller Cover,* 3-in. (75-mm) length, with a short nap (3/16 to 1/4 in. (4.8 to 6.4 mm) constructed from a woven fabric.[4] New unused roller covers are preferred but used roller covers may be used but must be thoroughly cleaned so the resulting appearance is like a new roller cover. When two or more coatings are being tested with the latter type of roller cover, each should have been used approximately the same number of times and should be well dried out from previous applications.

6.5 *Paint Tray.*

## 7. Materials

7.1 *Practical Opacity Charts*—These are smooth surface, sealed paper test charts on which are printed a 6-step series of grey stripes of increasing darkness numbered from 1 to 6. The test area is 6 $ft^2$ (5575 $cm^2$). For more complete details see Fig. 1 and the manufacturer's description in the

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved June 15, 1992. Published August 1992. Originally published as D 5150 – 91. Last previous edition D 5150 – 91.

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

[3] Discontinued; see *1992 Annual Book of ASTM Standards,* Vol 06.01.

[4] A 403-FPS-90 paint roller cover, made by EZ Painter, 4051 S. Iowa Ave., Milwaukee, WI 53207, or Doo-Z made by Wooster Brush, Wooster, OH, and widely available at commercial outlets has been found suitable for this purpose.

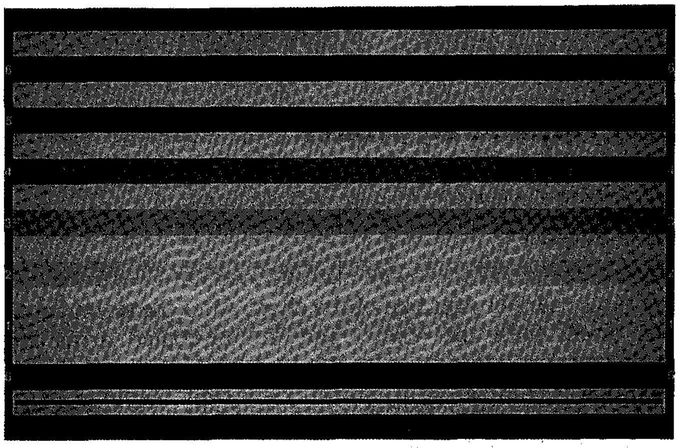

FIG. 1 Practical Opacity Chart in Accordance with Footnote 5 and Appendix X1

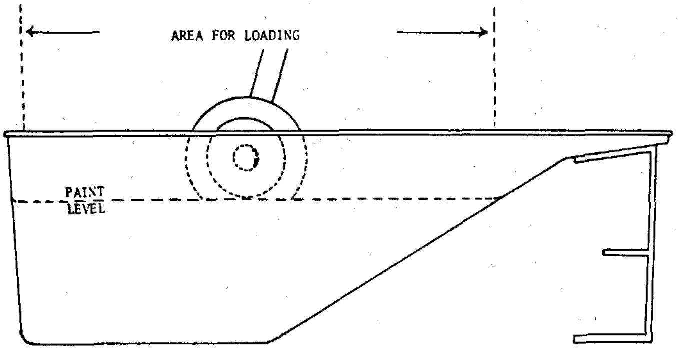

FIG. 2 Loading the Roller

appendix.[5]

7.2 *Masking Tape.*

## 8. Sampling and Conditioning

8.1 Sample the material in accordance with Practice D 3925.

8.2 Prior to testing, condition the samples in the standard atmosphere described in Specification D 3924. Make all tests under the same conditions.

## 9. Procedure

9.1 Determine the weight/gallon of each paint being tested to 0.1 lb in accordance with Test Method D 1475.

NOTE 1—The gallon unit referred to in this test method is the U.S. Gallon.

9.2 Use the following equation to determine the amount of paint required for each coat on 3 ft² of chart area:

$$\text{Grams for } 3\ \text{ft}^2\ (2787\ \text{m}^2) = \frac{1362 \times \text{Paint Density (lb/gal)}}{\text{Desired Spreading Rate (ft}^2\text{/gal)}}$$

The spreading rate may be either the manufacturer's recommended spreading rate for each paint, or the same spreading rate for all paints under test according to agreement between producer and user. A spreading rate of 450 ft²/gal (11.0 m²/L) is suggested if there is no other preference. Include spreading rate(s) used in the report.

9.3 At a convenient height, tape a test chart with the long direction horizontal, to a surface that is vertical or within 5° of vertical (top sloping away from the operator). The tape should be placed on the vertical, numbered border stripes that are outside the 6 ft² (5575 cm²) test area of the chart.

9.4 Prepare the paint roller for use in the test by the following procedure:

9.4.1 Load the roller cover with paint from the tray by rolling it through paint so that just the nap is submersed (see Fig. 2).

9.4.2 Roll out the loaded cover onto a scrap surface, in an upward and downward motion, within an area no larger than 1-ft (0.3-m) high by the 3-in. (75-mm) width of the roller cover.

9.4.3 Reload the roller cover and roll it out again in the same manner as 8.4.2 and over the same area. Do not increase the area except as necessary to control excess dripping of paint.

9.4.4 Repeat the procedure as necessary until the following conditions are met:

9.4.4.1 Reloading the paint roller cover does not result in increased paint pickup.

9.4.4.2 There is so much paint on the surface being used that the fully-loaded paint roller cover cannot transfer any more paint to the surface.

9.4.5 Now immediately perform the test.

9.5 Load the roller cover fully, just short of dripping and weigh. Either weigh the paint and roller before and after, or the paint and reservoir before and after. Do not weigh the paint on the chart, as even minimal evaporation results in significant inaccuracies. Apply to a small area of the left half of the chart, just to deposit the excess paint. Repeat this step as needed so that enough paint is on the chart for the desired spreading rate as calculated in 9.2. Use the roller to evenly spread the paint over the left half of the chart, reweighing to determine if the paint applied is within ±5 % of that desired. If not, add or remove paint as needed with the roller cover. Do this as quickly as possible to minimize the evaporation of volatiles. For this reason, only one adjustment is permitted and the paint out must be completed within 3 min.

9.6 Repeat 8.5 on the right half of the chart. Leave the chart in the (near) vertical position until the paint is dry to the touch. Let dry for 4 h or the manufacturer's recommended time, whichever is greater, before recoating.

9.7 With a soft lead pencil, lightly draw two lines 9 in. (230 mm) on either side of and parallel to the printed center line to outline a 3-ft² (2787-cm²) area in the middle of the chart. Repeat 9.5 on this area and again let dry as in 9.6. This results in four painted areas, two with one coat and two with two coats.

9.8 Visually determine the number of the darkest line fully hidden by application of the paint in each of the four painted areas. If the results of each pair of areas (one-coat and two-coated) do not agree exactly, the paint was not applied uniformly and the test must be repeated.

## 10. Report

10.1 Report the line numbers together with the spreading

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[5] Leneta Form CU-1 chart available from the Leneta Co., P.O. Box 86, Hohokus, NJ 07423, has been found suitable for this purpose.

rate used, as in the following example: Complete hiding of Line 2 with one coat and Line 5 with two coats at a spreading rate of 450 ± 20 $ft^2$/gal (11.0 ± 0.5 $m^2$/L)/coat is designated as 2-5/450.

## 11. Precision

11.1 *Precision*—In an interlaboratory study of this test method, one operator in each of five laboratories applied one and two coats of six paints varying widely in hiding power. Three of the operators evaluated both applications for hiding, one operator rated only the double-coat area, and one operator rated two of the paints for single coat and the other four paints for double-coat application. The pooled standard deviation for both single and double-coat application was found to be 0.5 rating units with 36 df. Based on these values the following criterion should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Reproducibility*—Two single results obtained by operators in different laboratories should be considered suspect if they differ by more than 1.5 rating units.

## 12. Keywords

12.1 architectural paints and coatings; hiding power; opacity; roller application of paints

## APPENDIX

### Nonmandatory Information

## X1. MANUFACTURER'S DESCRIPTION AND SPECIFICATIONS FOR THE PRACTICAL OPACITY CHARTS USED IN DEVELOPING THIS TEST METHOD[5]

X1.1 Description and Specifications are as follows:

X1.1.1 *Overall Size*—24 by 37½ in. (610 by 953 mm),

X1.1.2 *Test Area*—Size 24 by 36 in. (610 by 914 mm), 6 $ft^2$ (5575 $cm^2$),

X1.1.3 *Design*—Alternate stripes of white and neutral grey,

X1.1.4 *Stripe Dimensions*—$1\frac{13}{32}$ by 36 in. (36 by 914 mm),

X1.1.5 *Stripe Numbers*—The stripes are numbered 1 through 6 in order of increasing contrast, the numbers being printed on the two 24-in. borders. These borders are ¾-in. (19-mm) wide and are not normally considered a part of the test area.

X1.1.6 *Unnumbered Stripes*—The unnumbered stripes shown in Figure 1 are an integral part of the test area. Their purpose is to favor application uniformity on the numbered stripes by preempting the boundary locations that tend to be less uniformly coated than more centrally located areas.

X1.1.7 *Grey Stripe Reflectances*[5]—The reflectance values of the numbered stripes are selected with the objective that their color differences with respect to the white surround will be in the geometric sequence: 2, 4, 8, 16, 32, and 64 CIELAB units. The defining variable for the grey stripes is not the reflectance as such, but the ratio of the grey reflectance $G$ to the white reflectance $W$. Table X1.1 shows the relevant relationships and production tolerances.

NOTE X1.1—The term "reflectance" as used here refers to the luminous reflectance factor as defined in 4.2 of Method E 97, which may be expressed as a decimal fraction or as a percentage. The latter is numerically the same as the CIE Y-tristimulus value and is the scale reading on most if not all commercial colorimeters.

**TABLE X1.1 Relevant Relationships and Production Tolerances**

| Stripe Number | Color Difference,[A] % | Ratio G/W[B] | Reflectance G,[C] % |
|---|---|---|---|
| 1 | 2 ± 12.5 | 0.945 ± 0.007 | 75.6 ± 0.6 |
| 2 | 4 ± 12.5 | 0.893 ± 0.013 | 71.4 ± 1.0 |
| 3 | 8 ± 12.5 | 0.793 ± 0.024 | 63.5 ± 1.9 |
| 4 | 16 ± 12.5 | 0.617 ± 0.042 | 49.4 ± 3.4 |
| 5 | 32 ± 12.5 | 0.347 ± 0.055 | 27.8 ± 4.4 |
| 6 | 64 ± 12.5 | 0.067 ± 0.037 | 5.3 ± 3.0 |

[A] This is $\Delta E^*_{ab}$, the color difference with respect to the white stripes, expressed in CIELAB units as defined in 6.2 of Method D 2244.

[B] $G/W = (1 - \Delta E^*_{ab}/107.7)^3$. This equation is derived from 6.2.1 of Method D 2244 and assumes that for neutral grey versus white, the values $\Delta E^*_{ab}$ and $\Delta L^*$ are sufficiently close to be considered equal for the purpose of this test method.

[C] Values shown here are with a white stripe reflectance $W$ of 80 %. These values will vary somewhat with $W$, tolerances for the latter being ±2 % inter-batch and ±0.5 % intra-batch.

**Designation: D 5161 - 91**

# Standard Guide for Specifying Inspection Requirements for Coating and Lining Work (Metal Substrates)[1]

This standard is issued under the fixed designation D 5161; 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 guide is intended to aid the coating specification writer in selecting and specifying the appropriate inspection requirements. It indicates the inspection requirements that may be employed for each of four service environments including mild, moderate, severe, and immersion (see Table 1).

1.2 In order to aid the user in determining when to specify inspection requirements, a relationship between the consequence of failure and the suggested level of inspection is demonstrated (see Fig. 1).

1.3 It is not the intent of this guide to address the selection of protective coating systems, to specify surface preparation and application requirements, or to be a referenced document in a specification.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Metal Base[2]

D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base[2]

D 3276 Guide for Painting Inspectors (Metal Substrates)[3]

D 4285 Test Method for Indicating Oil or Water in Compressed Air[3]

D 4414 Practice for Measurement of Wet Film Thickness of Organic Coatings by Notch Gages[2]

D 4417 Test Method for Field Measurement of Surface Profile of Blast Cleaned Steel[3]

E 337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet and Dry-Bulb Temperatures)[4]

2.2 *NACE Standard:*

T6A-37 Recommended Practice for Discontinuity, (Holiday Testing of Protective Coating)[5]

2.3 *SSPC Standard:*

SSPC-Visl-89 Visual Standard for Abrasive Blast Cleaned Steel[6]

## 3. Terminology

3.1 *Service Environments Terms are as follows:*

3.1.1 *mild service*—indoor or protected outdoor areas not subject to rain, dew or corrosive elements, or both.

3.1.2 *moderate service*—areas subject to weather away from coastal or corrosive industrial environments, or both.

3.1.3 *severe service*—corrosive/erosive environments including coastal salt laden atmosphere, industrial atmosphere, and high intensity sunlight.

3.1.4 *immersion service*—wetted surfaces of tanks, containers, pits, etc and surfaces that are normally wet with condensation or exposed to other corrosive environments.

## 4. Significance and Use

4.1 The requirements for inspection should be addressed in all protective coating and lining work specifications.

4.2 This guide may be used by specification writers when selecting and establishing the inspection requirements for coating and lining specifications.

4.3 The instructions for using this guide are as follows:

4.3.1 Identify the service environment for the coating or lining system being specified. Read down the column under the appropriate service environment in Table I. Suggested levels of inspection listed on the left are identified with x's.

## 5. Inspection Requirements

5.1 *Pre-Surface Preparation:*

5.1.1 *Contaminants*—Visually verify that oil and grease are removed from the surface prior to surface preparation operations (see Guide D 3276).

5.1.2 *Surface Anomalies*—Visually verify that welds and sharp or jagged edges have been suitably prepared for the coating system being used (see Guide D 3276).

5.2 *Surface Preparation:*

5.2.1 *Air Supply*—Verify that air supply is clean and dry (see Test Method D 4285).

5.2.2 *Abrasives*—Verify cleanliness and proper size and type to achieve specified profile.

5.2.3 *Ambient Conditions*—Verify that air temperature, humidity, surface temperature, and due point spread are appropriate (see Test Method E 337).

5.2.4 *Surface Anomalies*—Identify any burrs, slivers, scabs, and weld spatter visible after blasting.

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[1] This guide is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.04 on Inspection.

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

[3] *Annual Book of ASTM Standards*, Vol 06.02.

[4] *Annual Book of ASTM Standards*, Vol 11.03.

[5] Available from National Association of Corrosion Engineers, P.O. Box 218340, Houston, TX 77218.

[6] Available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

**TABLE 1 Inspection Requirements**

| Suggested Inspection Requirements | Service Environments | | | |
|---|---|---|---|---|
| | Mild | Moderate | Severe | Immersion |
| Pre-Surface Preparation: | | | | |
| Visual for contaminants | X | X | X | X |
| Visual for surface anomalies | | | X | X |
| Surface Preparation: | | | | |
| Air supply for contaminants | | | X | X |
| Abrasives for type and cleanliness | | | X | X |
| Ambient conditions and dew point | | | X | X |
| Surface anomalies | | | X | X |
| Degree of cleanliness | X | X | X | X |
| Profile | | X | X | X |
| Visual final for contamination | | | X | X |
| Coating Application: | | | | |
| Materials | | X | X | X |
| Mixing | | | X | X |
| Equipment | | X | X | X |
| Air supply for contaminants | | X | X | X |
| Ambient conditions and dew point | | | X | X |
| Wet film thickness | | | X | X |
| Intercoat parameters | | X | X | X |
| Appearance between coats | | | X | X |
| Dry film thickness between coats | | | X | X |
| Final Acceptance: | | | | |
| Appearance | X | X | | |
| Visual runs and sags | | X | X | X |
| Visual holidays and pinholes | | | X | X |
| Dry film thickness | | X | X | X |
| Holiday tests | | | | X |
| Repairs | | | X | X |
| Final cure | | | X | X |

5.2.5 *Surface Cleanliness*—Verify degree of surface cleanliness (see SSPC-Visl-89).

5.2.6 *Profile*—Verify profile is within specified tolerance using appropriate instruments (see Test Method D 4417).

5.2.7 *Surface Contamination*—Verify that all visible surface contaminants including oil, grease, dust, etc have been removed.

5.3 *Coating Application:*

5.3.1 *Materials*—Verify that the coating materials and thinners are as specified, properly labeled and stored, and are within their shelf life.

5.3.2 *Mixing*—Verify that the coating materials are mixed in accordance with the manufacturer's instructions.

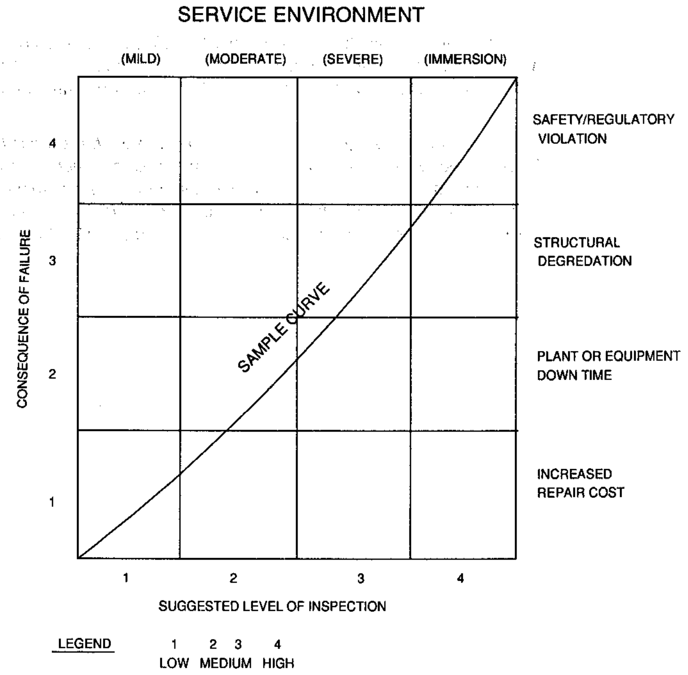
5.3.3 *Equipment*—Verify that proper application equipment is being utilized.

5.3.4 *Air Supply*—Verify that air supply is clean and dry (see Test Method D 4285).

5.3.5 *Ambient Conditions*—Verify that air temperature, humidity, surface temperature, and dew point spread are appropriate (see Test Method E 337).

5.3.6 *Wet Film Thickness*—Verify that the applicator is checking wet film thickness during the application process when required (see Practice D 4414).

5.3.7 *Intercoat Parameters*—Verify that parameters during recoat are observed and that no surface contaminants are present.



NOTE—This figure is provided as a visual aid to represent graphically the general relationship between the consequence of failure and the suggested level of inspection. The slope and configuration of the curve will vary due to the many factors affecting the consequence of failure, however, the suggested level of inspection should increase when the service environment is more severe.

**FIG. 1 Relationship Between the Consequence of Failure and the Suggested Level of Inspection**

5.3.8 *Appearance*—Visually inspect each coat for defects and uniform appearance (see Guide D 3276).

5.3.9 *Dry Film Thickness*—Verify that the dry film thickness is within the specified range after each coat (see Test Methods D 1186 and D 1400).

5.4 *Final Acceptance:*

5.4.1 *Appearance*—Visually inspect the surface for defects and uniform appearance (see Guide D 3276).

5.4.2 *Dry Film Thickness*—Verify that the total coating system's dry film thickness is within the specified range (see Test Methods D 1186 and D 1400).

5.4.3 *Holiday Test*—Verify that the surface meets the specified holiday/pinhole criteria (see NACE T6A-37).

5.4.4 *Repairs*—Verify that all identified repairs have been properly made. Repeat inspection requirements and testing as appropriate.

5.4.5 *Final Cure*—Verify that the coating is cured in accordance with the specified requirements of Guide D 3276.

## 6. Precautions and Limits

6.1 Specialty lining systems such as reinforced laminates may have additional inspection requirements to address hardness, strength, etc that are not addressed by this guide.

6.2 The quality of a project cannot be guaranteed by merely specifying inspection requirements, however, when the recommended inspections are properly implemented and documented, failures are minimized or eliminated.

## 7. Keywords

7.1 coating, inspection, specifications; consequence of failure; level of inspection; linings

# Standard Practice for Discontinuity (Holiday) Testing of Nonconductive Protective Coating on Metallic Substrates[1]

This standard is issued under the fixed designation D 5162; 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 procedures for determining discontinuities using two types of test equipment:

1.1.1 *Test Method A*—Low Voltage Wet Sponge, and

1.1.2 *Test Method B*—High Voltage Spark Testers.

1.2 This practice addresses metallic substrates. For concrete surfaces, refer to Practice D 4787.[2]

1.3 *This standard does not purport to address all of the safety problems, 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 Document

2.1 *ASTM Standard:*

D 4787 Practice for Continuity Verification of Liquid or Sheet Linings Applied to Concrete Substrates[2]

## 3. Terminology

3.1 *Description of Terms Specific to This Standard:*

3.1.1 *discontinuity, as used in this standard*—a void, crack, thin spot, foreign inclusion, or contamination in the coating film that significantly lowers the dielectric strength of the coating film. May also be identified as a holiday or pinhole.

3.1.2 *holiday, as used in this standard*—a term that identifies a discontinuity.

3.1.3 *holiday detector, as used in this standard*—a device that locates discontinuities in a nonconductive coating film applied to a conductive surface.

3.1.4 *pinhole, as used in this standard*—a film defect characterized by small porelike flaws in the coating which, when extended entirely through the film, will appear as a discontinuity. A pinhole in the finish coat may not appear as a discontinuity.

## 4. Significance and Use

4.1 A coating is applied to a metallic substrate to prevent corrosion, reduce abrasion or reduce product contamination, or both. The degree of coating continuity required is dictated by service conditions. Discontinuities in a coating are frequently very minute and not readily visible. This practice provides a procedure for electrical detection of minute discontinuities in nonconductive coating systems.

4.2 Electrical testing to determine the presence and number of discontinuities in a coating film is performed on a nonconductive coating applied to a conductive surface. The allowable number of discontinuities should be determined prior to conducting this test since the acceptable quantity of discontinuities will vary depending on coating film thickness, design, and service conditions.

4.3 The low voltage wet sponge test equipment is generally used for determining the existence of discontinuities in coating films having a total thickness of 20 mils (0.5 mm) or less. High voltage spark test equipment is generally used for determining the existences of discontinuities in coating films having a total thickness of greater than 20 mils (0.5 mm).

4.4 Coatings that are applied at a thickness of less than 20 mils (0.5 mm) may be susceptible to damage if tested with high voltage spark testing equipment. Consult the coating manufacturer for proper test equipment and inspection voltages.

4.5 To prevent damage to a coating film when using high voltage test instrumentation, total film thickness and dielectric strength in a coating system shall be considered in selecting the appropriate voltage for detection of discontinuities. Atmospheric conditions shall also be considered since the voltage required for the spark to gap a given distance in air varies with the conductivity of the air at the time the test is conducted. Suggested starting voltages are provided in Table 1.

4.6 The coating manufacturer shall be consulted to obtain the following information, which would affect the accuracy of this test to determine discontinuities:

4.6.1 Establish the length of time required to adequately dry or cure the applied coating film prior to testing. Solvents retained in an uncured coating film may form an electrically conductive path through the film to the substrate.

4.6.2 Determine whether the coating contains electrically

**TABLE 1 Suggested Voltages for High Voltage Spark Testing**

| Total Dry Film Thickness | | Suggested Inspection, V |
|---|---|---|
| mils | mm | |
| 8–12 | 0.20–0.31 | 1 500 |
| 13–18 | 0.32–0.46 | 2 000 |
| 19–30 | 0.47–0.77 | 2 500 |
| 31–40 | 0.78–1.03 | 4 000 |
| 41–60 | 1.04–1.54 | 5 000 |
| 61–80 | 1.55–2.04 | 7 500 |
| 81–100 | 2.05–2.55 | 10 000 |
| 101–125 | 2.56–3.19 | 12 000 |
| 126–160 | 3.20–4.07 | 15 000 |
| 161–200 | 4.08–5.09 | 20 000 |
| 201–250 | 5.10–6.35 | 25 000 |

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[1] This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.04 on Inspection.

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[2] *Annual Book of ASTM Standards*, Vol 06.02.

conductive fillers or pigments that may affect the normal dielectric properties.

4.7 This practice is intended for use with new coatings. It may also be applicable for coatings previously placed in service. However, considerations must be given to potential changes in the electrical properties of the coating due to in-service exposure.

## 5. Test Methods

### TEST METHOD A—LOW VOLTAGE WET SPONGE TESTING

### 5.1 Apparatus

5.1.1 *Low Voltage Holiday Detector*—an electronic device powered by a self-contained battery with voltages ranging from 5 to 90 V dc, depending on the equipment manufacturer's circuit design. It is used to locate discontinuities in a nonconductive coating applied to a conductive substrate. Operation includes the use of an open-cell sponge electrode wetted with a solution for exploring the coating surface, a ground connection, and an audible or visual indicator, or both, for signaling a point of coating discontinuity.

5.1.2 *Low Voltage Wet Sponge Tester*—a sensitivity device with the operating voltage being of little importance other than being part of the particular electronic circuit design.

5.1.3 *Wet Sponge Type Instrument*—a number of commercially available, industry-accepted, instruments are available. The following electronic principle describes two types of devices generally used; others may be available but are not described in this practice.

5.1.3.1 *Lightweight, Self-Contained, Portable Devices*—based on the electrical principle of an electromagnetic sensitive relay or a solid-state electronic relay circuit that energizes an audible or visual indicator when a coating discontinuity is detected. Generally this equipment is capable of being recalibrated in the field by the user.

5.1.3.2 *Lightweight, Self-Contained, Portable Devices*—also based on the principle of an electronic relaxation oscillator circuit that reacts significantly to the abrupt drop in electrical resistance between the high dielectric value of the coating film and the conductive substrate at the point of coating film discontinuity. This results in a rise in oscillator frequency as well as in the audible signal from the device. Generally, this equipment is incapable of being recalibrated in the field by the user.

### 5.2 Procedure

5.2.1 Sufficient drying or curing of the coating shall be allowed prior to conducting a test. The length of time required shall be obtained from the coating manufacturer. Solvents retained in the coating film could produce erroneous indicators.

5.2.2 The surface shall be clean, dry, and free of oil, dirt and other contaminates. Measure the film thickness of the coating with a nondestructive dry film thickness gage. If the coating film exceeds 20 mils (0.5 mm), use the procedures for high voltage spark testing described in Test Method B, High Voltage Spark Testing.

5.2.3 Test the instrument for sensitivity in accordance with 5.3.

5.2.4 Attach the ground wire from the instrument ground output terminal to the metallic substrate and ensure positive electrical contact.

5.2.5 Attach the exploring sponge lead to the other output terminal.

5.2.6 Wet the sponge with a solution consisting of tap water and a low sudsing wetting agent, combined at a ratio of not more than ½ fluid oz of wetting agent to 1 gal water. An example of a low sudsing wetting agent is one used in photographic development. The sponge shall be wetted sufficiently to barely avoid dripping of the solution while the sponge is moved over the coating. The wetting agent residue must be removed prior to executing repairs.

5.2.7 Sodium chloride (salt) shall not be added to the wetting solution because of the potential erroneous indications of discontinuities. The salt, after drying on the coated surface, may form a continuous path of conductivity. It will also interfere with intercoat adhesion of additional coats.

5.2.8 Contact a bare spot on the conductive substrate with the wetted sponge to verify that the instrument is properly grounded. This procedure shall be repeated periodically during the test.

5.2.9 Move the sponge over the surface of the coating at a moderate rate approximately 1 ft/s (0.3 m/s), using a double pass over each area. Apply sufficient pressure to maintain a wet surface. If a discontinuity is detected, turn the sponge on end to determine the exact location of the discontinuity.

5.2.10 Discontinuities that require repair shall be identified with a marker that is compatible with the repair coating or one that is easily removed.

5.2.11 To prevent telegraphing (current traveling through a moisture path to a discontinuity, giving an erroneous indication), take care to ensure that the solution is wiped dry from a previously detected discontinuity before continuing the test.

5.2.12 The wetting agent must be completely removed by rinsing the holiday area prior to repair.

5.2.13 Wet sponge holiday detection is not recommended between coats of a multicoat system. However, when a test is conducted between coats of a multicoat system, a wetting agent shall not be used and all residue left by the test water must be completely removed prior to applying additional coats.

### 5.3 Verifying Operation of Equipment

5.3.1 The instrument shall be tested for sensitivity prior to initial use and periodically thereafter, in accordance with the equipment manufacturer's instructions.

5.3.2 Test the battery for proper voltage output. Refer to the manufacturer's instructions.

5.3.3 Switch the instrument to the "on position," if applicable.

5.3.4 Wet the sponge with a wetting solution consisting of tap water and a wetting agent (see 5.2.6).

5.3.5 Connect the ground cable to the instrument ground output terminal.

5.3.6 Touch the ground cable alligator clip to the wetted sponge. The instrument signal should actuate in accordance with the instrument manufacturer's instructions.

5.3.7 If the instrument should fail to signal, it shall be considered defective.

### 5.4 Verifying Instrument Calibration

5.4.1 Verify instrument calibration in accordance with the manufacturer's latest published instructions. If out of calibration, the instrument shall be calibrated in accordance with the instrument manufacturer's latest published instructions, or returned for calibration.

## TEST METHOD B—HIGH VOLTAGE SPARK TESTING

### 5.5 Apparatus

5.5.1 *High Voltage Detector (in excess of 800 V)*—An electronic device used to locate discontinuities in a nonconductive protective coating applied to a conductive substrate. It consists of an electrical energy source, an exploring electrode, and a ground wire connection from the indicator signaling current flow through a coating film discontinuity to the substrate. The detector shall be equipped with a visual or audible indicator, or both.

5.5.2 *Exploring Electrode*, shall be of the type capable of maintaining continuous contact with the surface being inspected, such as bolts, raised areas, etc. It shall be kept clean and free of coating material.

5.5.3 *High Voltage Electrical Detector*, can be identified as either a pulse or direct current type. A pulse type detector discharges a cycling, high voltage pulse, while a direct current type discharges continuous voltage.

### 5.6 Procedure

5.6.1 Sufficient drying or curing of the coating shall be allowed prior to conducting a holiday test. The length of time required shall be obtained from the coating manufacturer. Solvents retained in the coating film could produce erroneous results, as well as a fire hazard.

5.6.2 The surface shall be clean, dry, and free of oil, dirt and other contaminates. Measure thickness of the coating with a nondestructive dry film thickness gage. If the coating film is less than 20 mils (0.5 mm), consider using procedures for low voltage testing (see Test Method A, Low Voltage Wet Sponge Testing). Although the high voltage spark tester is suitable for determining discontinuities in coating films of less than 20 mils (0.5 mm), it is recommended that the coating manufacturer be consulted before using this test. Certain coatings may be damaged if tested with this equipment.

5.6.3 Verify test instrument operation in accordance with 5.7.

5.6.4 Adjust the test instrument to the proper voltage for the coating thickness being tested. In selecting the inspection voltage, it is important to provide sufficient voltage to break the air gap that exists at the holiday. The air gap will vary depending on the total applied film thickness. The voltage required to break a given air gap may also vary due to atmospheric conditions such as relative humidity. Ensure that the voltage is high enough to break the air gap equivalent to the highest coating film thickness by separating the exploring electrode from the bare metal substrate using a nonconductive spacer equal to the maximum coating thickness. The voltage is set high enough to conduct the holiday test only if the spark will jump the gap formed by the spacer. Excessive voltage may produce a holiday in the coating film. The maximum voltage for the applied coating shall be obtained from the coating manufacturer. Table 1 contains suggested voltages that can be used as guides.

5.6.5 Attach ground wire from the instrument ground output terminal to the metal substrate and ensure positive electrical contact.

5.6.6 Make contact with the exploring electrode on the conductive substrate to verify that the instrument is properly grounded. This test shall be conducted periodically during the test. The spacer test described in 5.6.4 shall also be repeated if significant atmospheric changes take place during testing.

5.6.7 Move the exploring electrode over the surface of the dry coating at a rate of approximately 1 ft/s (0.3 m/s) using a single pass. Moisture on the coating surface may cause erroneous indications. If moisture exists, remove or allow to dry before conducting test.

5.6.8 Discontinuities that require repair shall be identified with a marker that is compatible with the repair coating or one that is easily removed.

### 5.7 Verifying Operation of Equipment

5.7.1 Test the energy source (battery) for proper voltage output. Refer to the manufacturer's instructions.

5.7.2 Connect the exploring electrode and grounding cable to the terminals of the detectors.

5.7.3 Switch the instrument to the "on" position.

5.7.4 Touch the exploring electrode to the ground cable alligator clip. The instrument signal should actuate in accordance with the instrument manufacturer's operating instructions.

5.7.5 If the instrument fails to signal, it shall be considered defective.

### 5.8 Verifying Instrument Calibration

5.8.1 Verify instrument calibration in accordance with the manufacturer's latest published instructions. If out of calibration, the instrument shall be calibrated in accordance with the instrument manufacturer's latest published instructions, or returned for calibration.

5.8.2 Perform field checking of the test voltage with the electrode placed against the surface of the lining since the exploring electrode voltage may be reduced by the slight current flow of the lining.

5.8.3 If required, compare measured voltage with the selected test voltage. Depending on the type of tester, adjust the selected voltage ±5 %.

## 6. Testing of Repaired Area

6.1 Sufficient drying or curing of the repair coating shall be allowed prior to retesting. The length of time required shall be obtained from the coating manufacturer.

6.2 Conduct the test following the procedures as previously outlined in this practice for the test instrument selected.

6.3 Retest only those areas that have been repaired, unless otherwise specified.

## 7. Keywords

7.1 discontinuity; holiday; holiday detectors; spark testers

# Standard Guide for Establishing Procedures to Monitor the Performance of Safety Related Coatings in an Operating Nuclear Power Plant[1]

## 1. Scope

1.1 This guide covers procedures for establishing a program to monitor Service Level I coating systems in operating nuclear power plants. Monitoring is an on going process of evaluating the condition of the in-service coating systems.

1.2 It is the intent of this guide to provide a recommended basis for establishing a coatings monitoring program, not to mandate a singular basis for all programs. Variations or simplifications of the program described in this guide may be appropriate for each operating nuclear power plant depending on their licensing commitments. Similar guidelines may be applicable for Service Level II and other areas outside containment.

1.3 *This standard does not purport to address all of the safety problems, 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 610 Test Methods for Evaluating Degree of Rusting on Painted Steel Surfaces[2]

D 714 Test Method for Evaluating Degree of Blistering of Paints[3]

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base[3]

D 3359 Test Methods for Measuring Adhesion by Tape Test[3]

D 4121 Practice for Photographic Documentation of Coating and Lining Failures and Defects[2]

D 4537 Guide for Establishing Procedures to Qualify and Certify Inspection Personnel for Coating Work in Nuclear Facilities[2]

D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers[2]

F 1130 Practice for Inspecting the Coating System of a Ship's Topside and Superstructure[4]

F 1131 Practice for Inspecting the Coating System of a Ship's Tanks and Voids[3]

F 1132 Practice for Inspecting the Coating System of a Ship's Decks and Deck Machinery[3]

F 1133 Practice for Inspecting the Coating System of a Ship's Underwater Hull and Boottop During Drydocking[4]

2.2 *ANSI Standard:*

N45.2.6—Qualification of Inspection, Examination, and Testing Personnel for Nuclear Power Plants[5]

2.3 *SSPC Standard:*

SSPC-PA2—Measurement of Dry Paint Thickness With Magnetic Gages[6]

2.4 *NACE Standard:*

RP0188-88—Standard Practice Discontinuity (Holiday) Testing of Protective Coatings[7]

## 3. Significance and Use

3.1 Establishment of an in-service coatings monitoring program permits planning and prioritization of coatings maintenance work as needed to maintain coat integrity and performance in nuclear Service Level I coating systems. For additional information on nuclear maintenance coating work, refer to ASTM Manual on Maintenance Coatings for Nuclear Power Plants.[8]

3.2 A coatings monitoring program enables early identification and detection of potential problems in coating systems. Some Service Level I coating systems may be known in advance to be suspect, deficient, or unqualified. Monitoring coating performance will assist in developing follow-up procedures to resolve any significant deficiency relative to coating work.

3.3 Degraded coatings may generate debris under design basis accident conditions that could adversely affect the performance of the post-accident safety systems. A coatings monitoring program may be required to fulfill safety analysis report commitments for Service Level I coating work in a nuclear power plant facility.

## 4. Responsibility

4.1 The owner/operator shall identify the department or group within the organization to be responsible for establishing the applicable requirements for activities or procedures covered by this guide and shall document the scope of their responsibility. Owner/operator delegation of this responsibility to other outside qualified organizations is per-

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[1] This guide is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.04 on Inspection.

Current edition approved Oct. 3, 1991. Published December 1991.

[2] *Annual Book of ASTM Standards*, Vol 06.02.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 01.07.

[5] Available from American National Standards Institute, 11 W. 42nd Street, 13th Floor, New York, NY 10036.

[6] Available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

[7] Available from National Association of Corrosion Engineers, P. O. Box 218340, Houston, TX 77218.

[8] Available from ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103.

mitted and shall be documented.

4.2 It is the responsibility of the owner/operator or his designee performing these activities to specify the detailed methods and procedures for meeting the applicable requirements of this guide.

4.3 The owner/operator or his designee shall assign a coordinator to be responsible for supervising coating inspection activities, data collection and documentation, and for ensuring that inspection personnel are adequately trained and qualified.

4.4 The owner/operator shall assign responsibility for evaluating the results of inspection activities carried out under the coatings performance monitoring program.

## 5. Frequency

5.1 Frequency of in-service coating inspection monitoring shall be determined by the owner/operator. In operating nuclear power plants certain monitoring activities may be restricted to major maintenance outages or refueling outages. It is a good practice to perform inspections during each refueling outage or during other major maintenance outages as needed.

## 6. Records and Past History

6.1 Coating performance will depend on the operating conditions experienced by the coating systems. Records of these conditions shall be obtained for each operating unit. These may include, but not be limited to, ambient conditions, temperatures, humidity, immersion, splash and spillage, chemical exposures, radiation exposures, previous decontamination procedures, abrasion and physical abuse, and start-up/shutdown frequency. Any change in service criteria or modifications of the physical design must be identified and dated.

6.2 The last two performance monitoring reports pertaining to the coating systems shall be reviewed prior to the monitoring process. Other past coatings history data to be reviewed may include:

6.2.1 Copies of coating specifications, manufacturer's product data sheets, and application procedures for in-place coatings.

6.2.2 Quality control documentation for the existing in-place coating systems and their application.

6.2.3 Copies of previous inspection or monitoring reports.

6.2.4 Documentation pertaining to any maintenance work performed on existing coating systems.

## 7. Premonitoring Procedure

7.1 Prior to conducting an inspection of the coating systems, the responsible organization shall ensure that the necessary services and equipment required for inspection are provided. Factors that must be considered while planning the inspection activities include, but are not limited to, lighting, access to coated surfaces, cleaning surfaces of any deposit or build up, ventilation and, where necessary, special underwater inspection requirements.

7.2 Station access procedures for Service Level I coating systems monitoring shall be followed. While access procedures may vary from plant to plant, specific station access procedures may include:

7.2.1 Security clearance for protected, radiation controlled, and vital areas, and escorted or unescorted clearance as required,

7.2.2 Radiological history including prior radiation exposure for all personnel involved,

7.2.3 Health physics classroom training in the use of radiation detection and monitoring devices and procedures for wearing anticontamination clothing,

7.2.4 A radiation work permit based on health physics radiological survey of the work location,

7.2.5 Compliance with radiation work permit requirements and other special radiation controls unique to each work location, and

7.2.6 Issuance of dosimetry.

7.3 The safety requirements of the facility owner/operator must be met when performing all inspection operations.

## 8. Personnel Requirements, Qualifications, and Training

8.1 The facility owner/operator shall specify the requirements and guidelines for qualification and training of inspection personnel involved in the program. However, inspectors and the coordinator shall be knowledgeable coatings personnel meeting the requirements of ASTM Guide D 4537 or ANSI N45.2.6. The coordinator shall, as a minimum, be a Level II Coatings Inspector.

8.2 The evaluator shall be a person knowledgeable and experienced in nuclear coatings work.

## 9. Inspection Plan

9.1 The owner/operator shall develop a plant specific inspection plan to accomplish the objectives of the monitoring program. A general visual inspection shall be conducted on all readily accessible coated surfaces during a walk-through. After the walk-through, thorough visual inspections shall be carried out on previously designated areas and on areas noted as deficient during the walk-through. A thorough visual inspection shall also be carried out on all coatings near sumps or screens associated with the Emergency Core Cooling System (ECCS). The inspection plan shall address the following based on specific owner/operator requirements:

9.1.1 A pre-inspection briefing to familiarize all inspection personnel with objectives of the inspections, procedures to be followed, and precautions to be taken,

9.1.2 Monitoring team(s) assignments to specific areas for inspection(s), and

9.1.3 Location maps dividing the plant into identifiable areas for inspection activities to be issued to inspection teams. The maps shall also identify items/areas requiring special testing, if any. The locations of all defects and of all tests performed shall be recorded on the maps so that additional testing, recoating, and further monitoring may be performed.

9.2 During walk-through, visually examine coated surfaces for any visible defects, such as blistering, cracking, flaking/peeling, rusting, and physical damage.

9.2.1 *Blistering*—Compare any blistering found to the blistering pictorial standards of coatings defects (refer to Test Method D 714) and record size and frequency. If the blisters are larger than those on the comparison photographs, measure, record size and extent, and photograph. Report if blistered portions are intact.

9.2.2 *Cracking*—Cracking can be limited to the one layer of coating or extend through to the substrate. Measure the length of the crack or if extensive cracking has occurred, measure the size of the area affected. Determine if the cracking is isolated or is part of a pattern. Record measurements and describe crack depth and pattern on the inspection report. Photograph the area affected.

9.2.3 *Flaking/Peeling/Delamination*—Measure the approximate size of the peels and note the pattern formed. Carefully test to see if lifting can be easily achieved beyond the obvious peeled area. Note all observations on the inspection report and photograph the area affected.

9.2.4 *Rusting*—Compare with the pictorial standards of Test Method D 610 to determine the degree of rusting. Try to determine the source of rusting (that is, is it surface stain caused by rusting elsewhere, or is it a failure of the coating allowing the substrate to rust). Photograph the affected area and record observations on the inspection report.

9.2.5 If no defects are found, mark "Coating Intact, No Defects" on the inspection report.

9.2.6 If portions of the coating cannot be inspected, note the specific areas on the location map-inspection report, along with the reason why the inspection cannot be conducted.

9.3 Written or photographic documentation, or both of coating inspection areas, failures, and defects shall be made and the process of documentation standardized by the facility owner/operator. Written documentation practice for inspection of coating system as given in Practices F 1130, F 1131, F 1132, and F 1133 may be adopted as a guideline. Practice D 4121 provides one method to obtain consistent, comparable close-up photographs.

9.4 For coating surfaces determined to be suspect, defective or deficient, one or more physical tests, such as dry film thickness (Test Methods D 1186 and SSPC-PA-2), adhesion (Test Methods D 3359 and D 4541), and continuity (NACE-RP0188-88), may be performed when directed by the evaluator. Samples may be gathered, and the size and extent of defective patterns may be described.

9.5 Instruments and equipment needed for inspection may include, but not be limited to, flashlights, spotlights, marker pen, mirror, measuring tape, magnifier, binoculars, 35-mm camera with or without wide angle lens, and polyethylene zip lock sample bags.

## 10. Reporting and Documentation

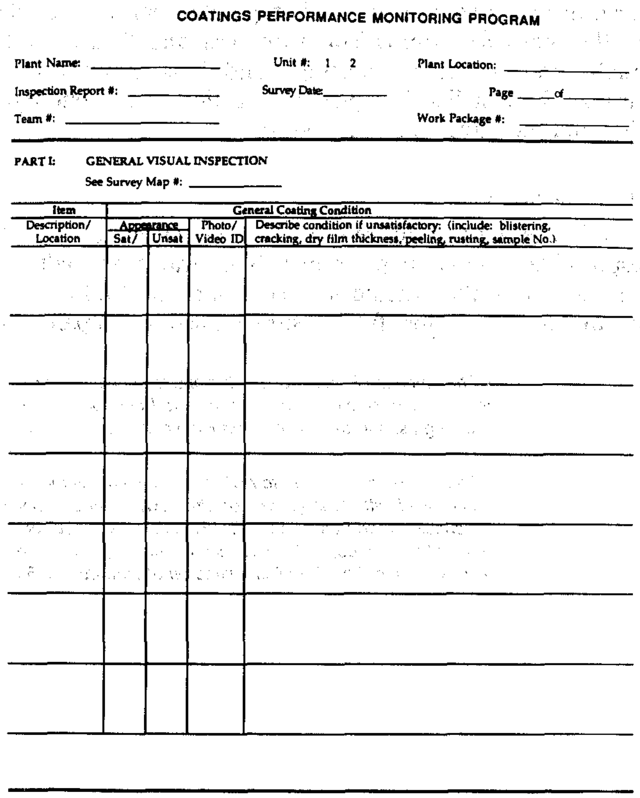
10.1 Inspection reports for submittal to responsible evaluation personnel based on specific owner/operator requirements should be prepared by a qualified Level II or Level III Coatings Inspector and should include at least the following information:

10.1.1 A list and location of all areas evidencing deterioration,

10.1.2 Prioritize the repair areas into areas that must be repaired during the same outage and areas where repair can be postponed to future outages, keeping the coating under surveillance in the interim period,

10.1.3 *Inspection Data Sheets*—A typical data sheet is given in Fig. 1, and

10.1.4 *Photographic Documentation*—A typical location map for photographic identification is given in Fig. 2. The



COATINGS PERFORMANCE MONITORING PROGRAM

Plant Name: ____________ Unit #: 1 2 Plant Location: ____________

Inspection Report #: ____________ Survey Date: ____________ Page ____ of ____

Team #: ____________ Work Package #: ____________

PART I: GENERAL VISUAL INSPECTION

See Survey Map #: ____________

| Item | General Coating Condition | | | |
|---|---|---|---|---|
| Description/ Location | Appearance Sat/ | Unsat | Photo/ Video ID | Describe condition if unsatisfactory: (include: blistering, cracking, dry film thickness, peeling, rusting, sample No.) |
| | | | | |
| | | | | |
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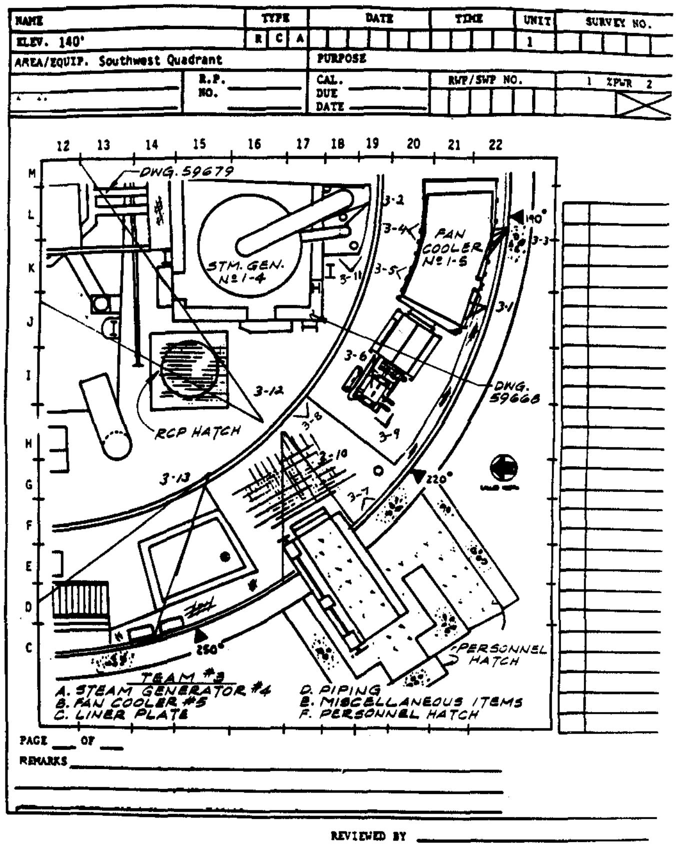
FIG. 1 Inspection Data Sheet



NAME | TYPE | DATE | TIME | UNIT | SURVEY NO.

ELEV. 140' | R C A | | | 1 |

AREA/EQUIP. Southwest Quadrant | PURPOSE

R.P. NO. ____ | CAL. DUE DATE ____ | RWP/SWP NO. | 1 XPWR 2

PAGE ___ OF ___

REMARKS ____________

REVIEWED BY ____________

FIG. 2 Photographic Documentation Location Map location and direction of all photographs shall be recorded on the maps for comparison with past and future photographs and to record existing conditions. Numbers 3-1 through 3-13 in Fig. 2 depict the location of the photographer and the directions of the view.

## 11. Evaluation

11.1 The inspection report shall be evaluated by the responsible evaluation personnel. The evaluator shall prepare a report that includes a summary of findings and recommendations for future surveillance or repair; this would include an analysis of the reasons or suspected reasons for failure. The repair work should be prioritized into major and minor defective areas. A recommended corrective plan of action must be provided for the major defective areas so that the plant can repair these areas, if appropriate, during the same outage.

## 12. Keywords

12.1 coatings monitoring program; coating performance; inspection; maintenance; nuclear power plant; safety related coatings; Service Level I; surveillance

**Designation: D 5181 – 91**

# Standard Test Method for Abrasion Resistance of Printed Matter by the GA-CAT Comprehensive Abrasion Tester[1]

This standard is issued under the fixed designation D 5181; 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 procedure for determining the abrasion resistance of printed matter using a GA-CAT Comprehensive Abrasion Tester.

1.2 This test method is applicable to packaging labels, book, catalog, and magazine covers, bar codes, corrugated boxes, and other containers having applied graphics on any flat substrate.

1.3 *This standard does not purport to address all of the safety problems, 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:*

E 171 Specification for Standard Atmospheres for Conditioning and Testing Materials[2]

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *abrasion resistance*—resistance against the act of scraping, smudging, or rubbing off.

3.1.2 *abrasiveness*—the degree to which a product tends to cause abrasion by the act of rubbing or scraping.

3.1.3 *receptor*—film or paper of standard abrasiveness onto which material removed from the specimen is deposited during the abrasion testing process. Alternatively, printed paper from which material is removed onto the specimen that has a higher degree of abrasiveness than the receptor (in case of testing abrasiveness).

## 4. Summary of Test Method

4.1 The test print and a receptor are sandwiched in the panel holder of the GA-CAT Comprehensive Abrasion Tester, clamped together with a known force, and made to slide over each other at a known frequency and over a known distance for a predetermined time period.

4.2 The test specimen is examined for degree of print degradation and the receptor for amount of ink or other material transferred from the specimen surface. Results may be rated relative to a comparative control run in the identical manner, or they may be quantified by comparison to a ranking scale numbered from zero to ten (zero being the most abrasion resistant and ten the least abrasion resistant).

## 5. Significance and Use

5.1 Abrasion resistance during transport and storage is essential to prevent marring of type matter, designs, or protective coatings on the exterior of labels and other printed materials. Recognizing that the actual amount of abrasion occurring in the field depends on relative humidity, temperature, tightness of packing, and a host of other variables, this test method provides a rapid means for comparing the abrasion resistance of test surfaces under laboratory conditions. It is useful for specification acceptance between the supplier and the customer.

5.2 This test method can also be used to evaluate the relative abrasion resistance of printed inks, varnishes, laminates and substrates, and the abrasiveness of inks.

## 6. Apparatus

6.1 *GA-CAT Comprehensive Abrasion Tester.*[3]

6.2 *Ranking Book,*[3] containing specimens whose degree of ruboff is ranked from 0 (no ruboff) to +10 (most ruboff).

## 7. Materials

7.1 *Standard Receptors*, approximately 4 by 4½ in. (102 by 114 mm), of an appropriate grade, as follows:

7.1.1 *C-1*[3] *(least abrasive)*—Glossy coated paper suitable for use with samples of low abrasion resistance.

7.1.2 *A-1, A-3, and A-4*[3,4] *(intermediate abrasiveness)*—Imperial lapping film with aluminum oxide abrasive particles of different sizes (9, 12, and 30 μm, respectively).

7.1.3 *A-6*[3,4] *(most abrasive)*, wet or dry, tri-um-ite 600.

7.1.4 *B-2*[3] *(intermediate abrasion resistance)*—Printed single color paper suitable for measuring abrasiveness of samples of average abrasiveness (corrugated).

NOTE 1—Other receptors may be substituted provided they have equal abrasiveness on the same test specimens. Further research and experience may indicate a need for additional grades of standard receptors.

7.2 *Foam Sheeting,*[3] 2 pieces, each approximately 2 mm in thickness and cut to 4 by 4½ in. (102 by 114 mm).

NOTE 2—Use of foam sheeting as a backing for the test specimen and the receptor is recommended to provide uniform pressure over the test surfaces and to prevent ink, varnish, or other particles from becoming imbedded in the sensitive surface of the panel holders.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved Oct. 15, 1991. Published December 1991.

[2] *Annual Book of ASTM Standards*, Vols 08.03 and 15.09.

[3] Available from Gavarti Assoc., Ltd., 9240 N. Sleepy Hollow Lane, Milwaukee, WI 53217.

[4] Available from 3M Co., St. Paul, MN 55119.

7.3 *Comparative Control,* a production or laboratory print preferably having known abrasion resistance. The comparative control must have the same substrate, color(s), and subject matter as the test print.

## 8. Test Specimen and Conditioning

8.1 The specimen size in the grain direction (or flute in the case of corrugated board) must not exceed 4½ in. (114 mm). The specimen size in the cross-grain direction is less critical; 4 in. (102 mm) is recommended. Care should be taken to avoid contaminating the test surface with fingerprints during specimen cutting and handling.

8.2 Condition the specimens at 73.5 ± 3.6°F (23 ± 2°C) and 50 ± 5 % relative humidity in accordance with Specification E 171.

## 9. Preparation of Apparatus

9.1 Set the abrasion tester on a sturdy bench, preferably in a room conditioned at the temperature and relative humidity prescribed in 8.2. Make sure that the three-prong power cord is correctly connected.

9.2 Periodically, check the liquid level in the hydraulic pressure system. If not full, follow the instructions in the manufacturer's manual.

9.3 If test conditions have not been specified, select an appropriate receptor by running the reference standard for the length of time required to achieve a visible level of degradation. Start with receptor C-1 (7.1.1). If the test time exceeds 2 min, use a receptor with a higher level of abrasiveness.

NOTE 3—Excessively long rubbing times are to be avoided because they introduce uncontrollable heat development that can alter the results.

## 10. Procedure for Abrasion Testing

10.1 *Loading the Instrument:*

10.1.1 Move the tray containing the four panel holders to the front black support shelf. Make sure that the inscribed "L" appears on the upper left side of the left panel holder and the "R" in the upper right side of the right panel holder (see Fig. 1). There should be space of at least ½ in. between the two center panel holders.

10.1.2 Face the test specimen on the receptor (selected in 9.3) and sandwich between the foam sheets. Place the sandwich between the two center panel holders so that the grain direction of the specimen is vertical. Take care that the sandwich does not extend below the bottom of the panel holders.

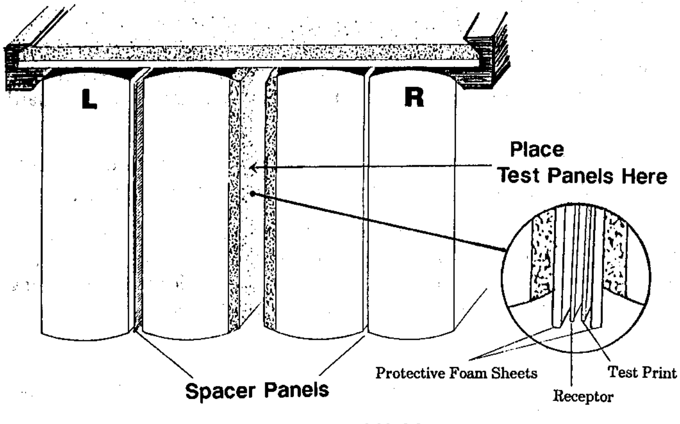

FIG. 1 Panel Holders

10.1.3 Push the four panel holders together into an upright parallel position. Make sure that the sandwich is not pinched at the bottom of the panel holders; otherwise, it will tend to slide down during the test. Grab the pins and move the tray inside the instrument so that the two retaining pins drop into the carriage holes. If the pins do not drop into their respective holes, recenter the panel holders on the sample tray.

10.1.4 Apply side pressure by turning the lower black spindle until the corresponding dial reads 20 pounds per square inch (psi). Do not apply top pressure at this time.

10.1.5 The carriage inside the instrument is meant to move between two limit switches (used for centering) approximately 2 in. (50.8 mm) apart. If the carriage is not situated between these limits, manually center the carriage.

10.2 *Testing Under the Preset Mode:*

10.2.1 Turn the power switch on by pressing the upper side of the flip switch at the right side of the instrument.

10.2.2 After the control box window (LCD) displays "GA-CAT ready," press CNTR; the LCD will read "check limits" to make sure the carriage is within the range of the limit switches. Press CNTR again and the LCD will read "release top pressure." Press CNTR once more and the carriage will move to the extreme right (limit switch) and then back to stop exactly in the center. At this point, the LCD will show that the carriage has been centered by showing a flashing square after "GA-CAT READY."

10.2.3 After the instrument is centered, apply the top pressure of 40 psi.

10.2.4 Push the "STRT" button. The LCD will read "computing" and after 3 s, the instrument will run under the following default values:

| | |
|---|---|
| Time | 5.0 s |
| Frequency | 2.0 Hz |
| Span | 1.0 in. |
| Offset | 0.0 in. |

10.2.5 When the instrument stops, release the top pressure, making sure that the top pressure plate is high enough to clear the panel holders. Push CNTR to re-center the carriage. Release the side pressure and pull the panel holders out of the carriage. Remove the sandwich and examine the test specimen and the receptor.

10.2.6 To continue running the test with the same settings, reload the instrument as in 10.1, apply the top pressure as in 10.2.3, and push STRT. When the instrument stops, follow the procedure in 10.2.5. Turn the switch off only at the conclusion of testing.

10.3 *Changing the Default Values:*

10.3.1 The default values may be changed to any of the following settings:

| | |
|---|---|
| Time | Continuous from 1 to 3600 s |
| Frequency | 0.2 to 3.0 Hz (cycles per s) with a resolution of 0.1 Hz |
| Span | 0.1 to 1.9 in. with a resolution of 0.1 in. |
| Offset | 0.1 to 0.4 in. (increments of 0.1 in.) |

10.3.2 In order to change a setting, press the "mode" button until the LCD displays the default value to be altered. Enter the new value on the number buttons and then press "enter." The LCD will read "new value?". If the value is correct, press "enter" a second time. If incorrect, enter another value or press "clear" and start over again.

NOTE 4—The instrument reverts to the original default values by pressing "reset" and any time the instrument is turned off.

10.3.3 When running thick samples such as corrugated material, follow the procedure in 10.1 through 10.2.1. If the center of the panel holders is not aligned with the center mark of the carriage, use the "offset" mode. Press the "mode" button until "offset" appears and then enter the new setting. A setting of 0.1 or 0.2 is recommended. An additional adjustment for overall width of the panel holders may also be necessary by removing one or two black separators.

10.3.4 Proceed further as in 10.2.2 until 10.2.6.

## 11. Interpretation of Results

11.1 Run the test material and the comparative control in the identical manner. Examine each print for degree of degradation and each receptor for amount of material transferred from the print. Report results for abrasion relative to the comparative control as equal, slightly or appreciably less, or slightly or appreciably greater.

11.2 A numerical record of degree of abrasion ranging from zero to ten can be obtained from a comparison of the test specimen with examples in the Ranking Book (6.2).

## 12. Precision and Bias

12.1 *Precision*—An interlaboratory study of this test method was conducted in which operators in 17 laboratories tested six prints in triplicate on each of two days. The prints consisted of three density levels of two different colors and were ranked for abrasion resistance from 1.0 to 4.0. The within-laboratory pooled standard deviation was 0.54 at 15 df and the between-laboratories pooled standard deviation was 0.58 at 18 df. Based on statistical analysis of the results, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1 *Repeatability*—Two results, each the mean of two determinations obtained by the same operator on different days, should be considered suspect if they differ by more than 1.5 ranking units.

12.1.2 *Reproducibility*—Two results, each the mean of results obtained on different days by operators in different laboratories, should be considered suspect if they differ by more than 2.2 ranking units.

12.2 *Bias*—Bias cannot be determined as there are no standard materials.

## 13. Keywords

13.1 abrasion; abrasiveness; printed matter; printing inks; smudging

**Designation: D 5235 - 92**

# Standard Test Method for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products[1]

## 1. Scope

1.1 This test method covers the measurement of dry film thickness of coatings applied to a smooth, textured or curved rigid substrate of wood or a wood-based product.

1.2 This test method covers the preparation of wood or wood-based specimens for the purpose of microscopical measurement of dry film thickness.

1.3 This test method suggests an interpretation of dry film thickness of coatings on wood or wood-based products when porous substrates are coated.

1.4 This test method suggests an interpretation of dry film thickness of coatings on wood or wood-based products when substrate attached or non-attached fibers occur in the dry film.

1.5 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address the safety problems, 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.* Specific hazard statements are given in Section 7.

## 2. Terminology

2.1 *Description of Terms Specific to This Standard:*

2.1.1 *dry film thickness*—that layer of thickness of dried coating above the microscopically visible board surface that also comprises attached fibers but excludes free fibers that are encapsulated in the layer itself.

2.1.2 *edge face*—That part of the specimen that is a plane perpendicular to the surface showing a cross section of the coating and substrate.

2.1.3 *soak in*—refers to a coating on a porous substrate where the coating does not lie essentially on the surface of the wood or wood-based product, but has penetrated into the fiber structure of the wood or wood-based material.

2.1.3.1 *Discussion*—Wood or wood-based products are generally of a porous nature; sometimes exhibiting uniform absorption of coatings. Frequently absorption of coatings is of a nonuniform nature and influenced by localized surface density differences or wood pore size. These conditions of coating absorption are commonly referred to as soak in.

## 3. Summary of Test Method

3.1 A specimen of coated wood or wood-based product is cut to convenient size and edge polished with sandpaper.

3.2 The polished edge of the specimen is viewed through a calibrated microscope in order to measure dry film thickness.

3.3 Suggestions regarding interpretation of dry film thickness on porous wood or wood-based material are offered.

3.4 Suggestions regarding interpretation of dry film thickness on wood or wood-based material that have attached or encapsulated fibers in the coating are offered.

## 4. Significance and Use

4.1 The dry film thickness of coatings on wood or wood-based products is specified in written product warranties for proper decorative and protective performance of coatings on wood or wood-based products.

4.2 The minimum and maximum dry film thickness of coatings is recommended by coating companies for satisfactory decorative and protective performance on wood or wood-based products.

4.3 The average dry film thickness of coatings on wood or wood-based material may be used by manufacturing companies to calculate theoretical cost of applied coatings. By comparison with actual cost, utilization efficiency may be calculated.

4.4 The ratio of peak to valley dry film thickness on textured products is used as an indication of coating uniformity.

4.5 Specific coated product requirements may dictate certain film thickness determinations to be made. Discussions between buyer and seller may be advisable to accommodate product needs relative to dry film thickness.

## 5. Apparatus

5.1 *Calibrated[2] Monocular Microscope*, equipped with an optical system providing sufficient resolution of 0.1-mil (2.54-μm) dry film thickness. One system consisting of a 16-mm objective and a 10-power filar micrometer eyepiece, resulting in a magnification of at least 100 diameters, has been found satisfactory. Other combinations of objectives and eyepieces and other magnifications may also be suitable, although magnifications above 200 diameters may result in distortion of the viewed cross section.

5.2 *Source of Oblique Illumination*, for the microscope.

5.3 *Cutoff Saw.*

5.4 *Belt or Disc Sander.*

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.52 on Factory-Coated Wood Building Products.

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[2] Consult the microscope manufacturer's operational manual for the correct calibration procedure.

5.5 *C-Type Clamp.*

## 6. Materials

6.1 *200 and 600-Grit Sand Paper.*

6.2 *Mold,* such as a paper cup, aluminum weighing dish, or a 2-in. (50.8-mm) or larger diameter plastic pipe that is at least 1-in. (25.4-mm) high.

6.3 *Source of Sanding Adhesive,* which is used as encapsulating medium such as:

6.3.1 *Hot Melt Glue,*

6.3.2 *Fast-Cure Acrylic Mounting Kit,*[3] and

6.3.3 *Epoxy.*[4]

6.4 *Solvent-based Tint Dispersion*[5], which is compatible with the sanding adhesive.

6.5 *Mineral Oil.*

6.6 *Automotive Red Transmission Oil.*

6.7 *Zinc Stearate Powder.*

## 7. Hazards

7.1 Use saws and sanders with goggles, dust mask, and proper machine safeguards to prevent injury to body limbs.

7.2 Solvent-based tint dispersions and adhesives may be flammable and contain toxic solvents. See manufacturer's instructions for use and proper disposal.

## 8. Procedure

8.1 *Specimen Preparation:*

8.1.1 Select the desired coated area of a wood or wood-based material that is to be measured for dry film thickness. With the cutoff saw, cut off a sample at least 1½-in. (38.1 mm) wide from this area.

8.1.2 Cut this specimen to a length that is at least ½-in. (12.7 mm) less than the inside diameter of the mold to be used.

8.1.3 Place the specimen, with the sample edge to be measured, face down and approximately centered in the mold.

8.1.4 Prepare the sanding adhesive according to the manufacturer's direction for use. A dispersed pigment[5] may be added to the adhesive for better microscopic contrast between the dry film and the adhesive.

8.1.5 Pour the sanding adhesive around the sample in the mold and allow to harden according to the manufacturer's directions.

8.1.6 Remove the mold from the hardened and encapsulated specimen edge.

8.1.7 Using a disc sander, belt sander or 200-grit sandpaper mounted on a glass plate, sand the edge face of the encapsulated specimen to be measured until the edge face is relatively smooth. Maintain the edge face of the specimen as flat as possible during sanding. Avoid heat buildup of the sanding adhesive by intermittent sanding if necessary.

8.1.8 Polish the edge face of the rough sanded specimen as follows:

8.1.8.1 Mount a piece of 600-grit sandpaper on a flat glass plate. Rub the edge face of the rough sanded specimen over the 600-grit sandpaper in one direction, then reverse direction by 180° for several more rubs. Zinc stearate powder can be sprinkled on the 600-grit sandpaper or the 600-grit sandpaper can be wetted with mineral oil to produce a highly polished edge face free of scratches.

8.1.9 To improve the microscopic contrast between the coating and the wood or wood-based product, wipe a light film of mineral oil or automotive red transmission oil across the polished edge face with a clean cotton rag or equivalent.

8.1.10 Some coatings and substrates are hard enough that encapsulation with a sanding adhesive is not necessary. Although in all cases, use of the encapsulating sanding adhesive will lead to the sharpest microscopic edge face and the highest degree of accuracy.

8.1.11 Some laboratories find that a mold for the specimen encapsulation with sanding adhesive is not necessary. In this case two specimens are prepared with the cut off saw. The sanding adhesive after proper mixing is generously applied to the coated face of each specimen, the specimens are placed together and a C-clamp is used to squeeze out some of the sanding adhesive. The C-clamp is not removed until the sanding adhesive has hardened.

8.2 *Measurement for the Microscopic Dry Film Thickness of the Polished Edge Face of the Specimen:*

8.2.1 Place the polished edge face under the microscope lens.

8.2.2 Adjust the illuminating light at a convenient oblique angle.

8.2.3 Focus the 10-power filar micrometer eyepiece on one side of the dry film thickness spot to be measured.

8.2.4 Advance the 10-power filar micrometer so as to reach the other side of the spot to be measured in 8.2.3.

8.2.5 Read the micrometer and calculate the dry film thickness by multiplying the distance in millimetres or inches per drum division on the micrometer by the number of drum divisions in the reading by the calibration factor.

8.2.6 Multiply the calculated number in 8.2.5 by 1000 for English units (39.37 for metric units), for conversion to a dry film thickness in mils.

8.2.7 Porous substrates tend to have the coating soaked into the open fiber structure of the wood or wood-based product. A clear demarcation line between substrate and coating is not discernible. In this case, it is suggested that film thickness is that layer of dry film thickness above the microscopically visible board surface that also comprises attached fibers but excludes free fibers that are encapsulated in the layer itself (see Figs. 1, 2 and 3).

8.2.8 Several microscope companies have advanced op-

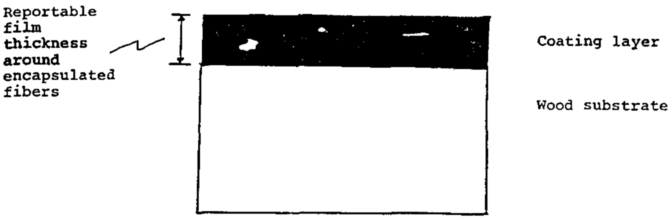

**FIG. 1 Illustrates Film Thickness Above the Wood Surface Where the Coating Layer Encapsulates Free Wood Fibers**

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[3] Fast-cure acrylic mounting kit available from Buehler Ltd., 41 Waukegan Rd., P. O. Box 1, Lake Bluff, IL 60044 has been found satisfactory for this purpose.

[4] Glue available from LePage's Inc., P. O. Box 291, Gloucester, MA 01930 has been found satisfactory for this purpose.

[5] Dispersion available from The Harshaw Chemical Co., 1945 East 97th St., Cleveland, OH 44106 has been found satisfactory for this purpose.

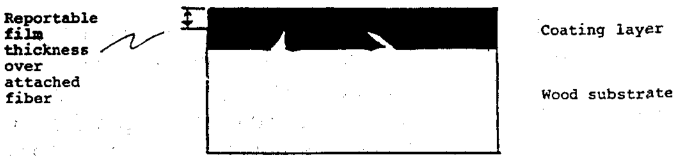

FIG. 2 Illustrates Film Thickness with Wood Fibers Attached to the Wood Surface and Surrounded by a Coating Layer

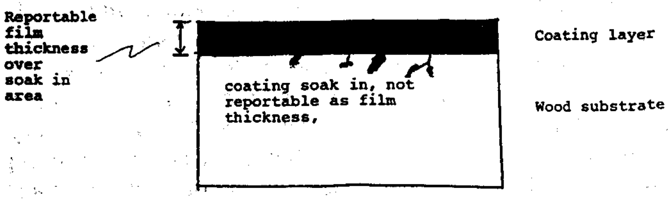

FIG. 3 Illustrates Coating Soak In, Not Reportable as Film Thickness

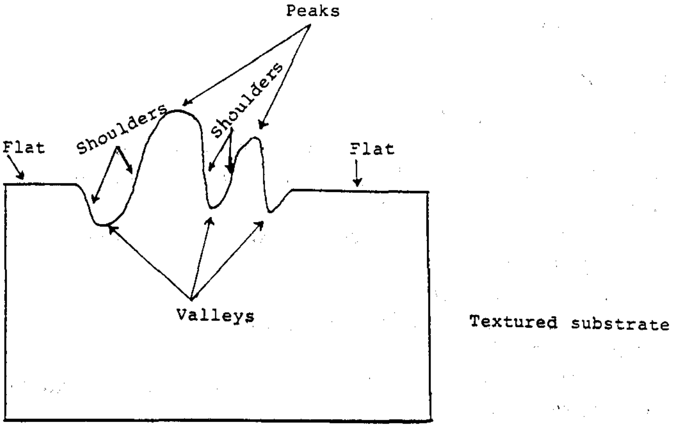

FIG. 4 Illustrates the Peaks, Shoulders (Slopes), Valleys and Flat Areas of a Textured Substrate

tical systems and useful accessories for measuring dry film thickness of coatings on properly prepared coated wood or wood-based specimens. The use of their equipment for the measurement of dry film thickness on wood or wood-based products is highly recommended. The directions for use of the equipment are specific to each microscope company. Closely follow the manufacturer's instructions.

## 9. Report

9.1 Report the dry film thickness of the spot to be measured in mils or microns.

9.2 For textured substrates, report the following information (see Fig. 4):

9.2.1 The dry film thickness in the valleys,

9.2.2 The dry film thickness on the shoulders or slopes,

9.2.3 The dry film thickness on the peaks,

9.2.4 The dry film thickness on the flat areas if any, and

9.2.5 Optionally some laboratories report the ratio of the peak to valley dry film thickness or the average of multiple dry film readings of one or all of these substrate areas.

9.3 For woods with large pores ignore the soak in of coating into the wood pore or cell and report only the dry film thickness above the microscopically visible board surface (see Fig. 5).

## 10. Precision and Bias

10.1 The precision and bias for this test method are primarily dependent upon each operator choosing exactly the same spot on the polished specimen for measurement.

10.2 The precision and bias statements will be developed in round-robin testing.

## 11. Keywords

11.1 coated wood or wood-based product; dry film thickness; microscopic measurement

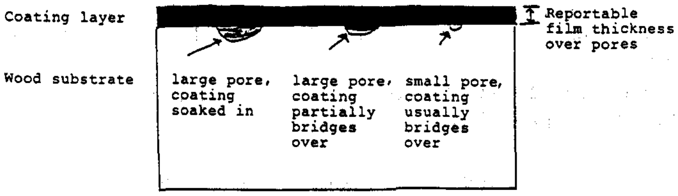

FIG. 5 Illustrates Film Thickness On Wood Substrates With Large Pores

# Standard Practice for Physical Characterization of Paint Brushes[1]

## 1. Scope

1.1 This practice covers the dimensions, terminology, materials and characteristics generally considered of importance to those within, or dealing with paint brushes, and describes methods of determining these parameters. This practice is not meant to be a definitive analytical method to deformulate brushes.

1.2 *This standard does not purport to address all of the safety problems, 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. Terminology

2.1 *Definitions:*

2.1.1 *chisel (or chiseled)*—a description of the configuration of the tip of the brush as being of a more or less convex shape across the thickness dimension.

2.1.2 *flat tip*—a description of the configuration of the tip of the brush as being flat or of uniform length across the thickness dimension.

2.1.3 *flagged*—a term describing the tip of a filament or bristle as being split resulting in a multiplicity of filament ends.

2.1.4 *tipped*—a term describing the tip of a filament or bristle as coming to a point.

2.1.5 *microtome*—a device for holding a number of filament or bristle materials in a rigid state for a cross sectional examination under a microscope (see Fig. 1).

## 3. Significance and Use

3.1 It is important to recognize that a brush can be a mixture of several chemically different filaments or bristles, or both, in several cross sections, thicknesses and lengths. The following procedures will be helpful in identifying brush composition.

## 4. Determination of Head Dimensions

4.1 *Width*—Determine the width by measuring the brushing material adjacent to the ferrule in the wide or broad dimension, using any accurate linear measuring device. Report dimensions to the nearest 1/8 in. (3 mm).

4.2 *Thickness*—Determine the thickness by measuring the brushing material adjacent to the ferrule, in the narrow dimension, using any accurate linear measuring device. Report dimensions to the nearest 1/16 in. (1 mm).

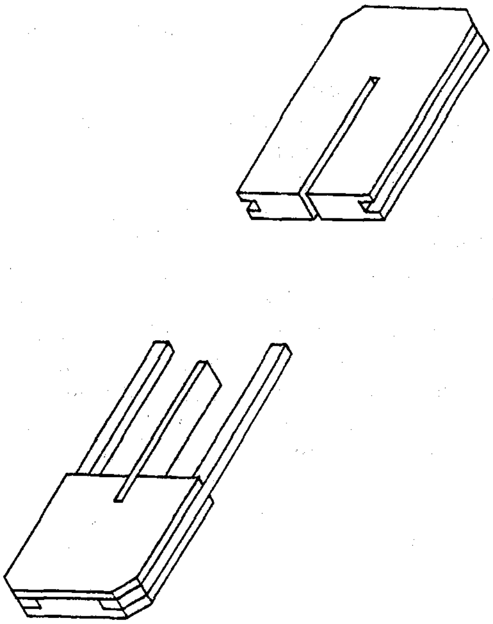

FIG. 1 Microtome

4.3 *Length Clear (Length Out)*—Determine the length by measuring the exposed filament or bristle length from the edge of the ferrule to its tip, using any accurate linear measuring device. Measure the brush on each end of the width and average the measurements so obtained to give the mean value. Report dimensions to the nearest 1/16 in. (1 mm).

NOTE 1—In the case of an angular sash brush, indicate whether the ferrule is angular or straight.

## 5. Brush Tip Configuration

5.1 Describe the configuration at the tip of the head as chiseled or flat (see 2.1.1 and 2.1.2).

## 6. Brushing Material Characteristics

6.1 *Cross-Sectional Shape*—Determine the cross-sectional shape of the filaments or bristles by inserting a representative sample of brushing material into the slot of a microtome, cutting the material flush with the surface of the microtome with a razor blade, and examining it visually with a 30× or greater microscope. Report the observations of filament shapes for example, round, solid, hollow or others.

6.2 *Filament or Bristle Length*—Determine the lengths of the filaments or bristles used in a brush by opening the ferrule and removing the filaments from that portion of the brush unaffected by the chisel. Measure a representative number of specimens to the nearest 1/16 in. (1 mm), and

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[1] This practice is under the jurisdiction of ASTM D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.61 on Paint Application Tools.

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report the length or various lengths.

6.3 *Filament or Bristle Thickness and Profile*—Remove a representative sample of filaments or bristles (10 to 20) from the brush as described in 6.2 and measure their thickness or diameter using a micrometer. Take measurements with a dial micrometer near each end. Those having the same measurement on each end are termed "level" and only one dimension is expressed; those having differences by 2 mils or more are termed "tapered" and the two dimensions are expressed as "thicker dimension/thinner dimension." Express dimensions in thousandths of an inch or hundredths of a millimeter.

6.4 *Filament or Bristle Color*—Determine how many different color filaments are present. This will assist in separating the filament sample, once taken, into the various filament types for proper analysis. Often, filaments of different composition are colored differently as well.

6.5 *Chemical Composition*—Material chemical composition may be determined by removing a representative sample of filaments or bristles and subjecting them to conventional analytical methods. Burning odor and behavior, solubility, density, melting point, and infra red spectroscopy are all useful techniques for estimating chemical composition. It is important when determining chemical composition that a uniform and homogeneous sample of filaments be used.

6.6 *Finishing (Processing)*—Observe finishing on the tips of the filaments or bristles, such as tipping or flagging, by visual examination with a 10× magnifying glass (see 2.1.3 and 2.1.4).

## 7. Ferrule Characteristics

7.1 *Ferrule Dimensions:*

7.1.1 Measure the shorter dimension (thickness) of the opening of the ferrule to the nearest 1⁄16 in. (1 mm).

7.1.2 Measure longer dimension (width) of the opening of the ferrule to the nearest 1⁄8 in. (3 mm).

7.1.3 Measure dimension across the closed part of the ferrule (height) to the nearest 1⁄16 in. (1 mm).

7.2 *Ferrule Material:*

7.2.1 Determine the surface material of the ferrule by conventional chemical analysis. Usual materials are tin plated steel, nickel plated steel, lacquer coated brass or copper plated steel and stainless steel.

7.2.2 Determine if the surface is lacquered by vigorously rubbing the ferrule using a cloth wetted with a solvent such as acetone or butyl acetate. If lacquered, the finish will dissolve.

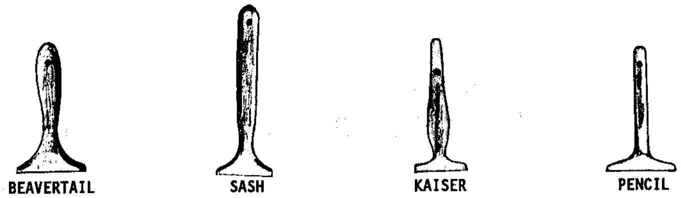

FIG. 2 Common Brush Handle Styles

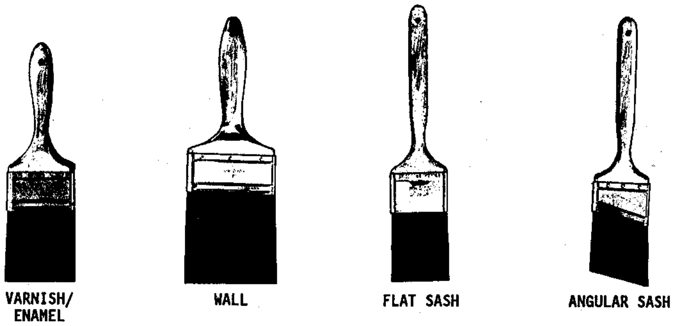

FIG. 3 Common Paint Brush Styles

7.3 *Ferrule Shape*—Describe the shape of the ferrule opening as rectangular (having corners), straight with rounded ends, oval, or other.

## 8. Handle

8.1 Determine the length of the handle by simple measurement from the handle tip to the ferrule expressed to the nearest 1⁄16 in. (1 mm).

8.2 Describe the handle material as wood, plastic, or other.

8.3 Describe the handle finish (sealed, lacquered, none).

8.4 Describe the handle style, for example, beavertail, flat sash, kaiser, pencil or others (see Fig. 2).

## 9. Handle Attachments

9.1 Describe the attachment method of the ferrule to the handle as stapled, nailed or crimped.

9.2 Describe the fit of the handle to the ferrule.

## 10. Brush Style

10.1 Report the style of the brush, such as varnish/enamel, wall, flat sash, angular sash or other (see Fig. 3).

## 11. Keyword

11.1 paint brush, physical characterization

# Standard Guide for Testing Water-Borne Architectural Coatings[1]

## 1. Scope

1.1 This guide covers the selection and use of procedures for testing water-borne coatings to be used on exterior, interior or both types of surfaces (Note 1). The properties that can be examined or, in some cases, the relevant test procedures are listed in Tables 1 and 2.

NOTE 1—The term "architectural coating" as used here combines the definition in Terminology D 16 with that in the *FSCT Paint/Coatings Dictionary*, as follows: "Organic coatings intended for on-site application to interior or exterior surfaces of residential, commercial, institutional, or industrial buildings, in contrast to industrial coatings. They are protective and decorative finishes applied at ambient temperatures. Often called Trade Sales Coatings" (see 2.3).

NOTE 2—Architectural coatings that are designed to give better performance than most conventional coatings because they are tougher and more stain and abrasion resistant are covered by Guide D 3730.

1.2 The types of organic coatings covered by this guide are as follows:

Type 1 Interior Latex Flat Wall Paints, partly covered by Guide D 2931
Type 2 Exterior Latex House Paints, partly covered by Guide D 3129
Type 3 Water-Borne Floor Paints, partly covered by Guide D 3358
Type 4 Interior Latex Semigloss and Gloss Paints, partly covered by Guide D 4540

1.2.1 Each is intended for application by brushing, rolling, spraying or other means to the material appropriate for its type, which may include plaster, masonry, wallboard, wood, steel, previously painted surfaces, and other architectural substrates.

1.3 *This standard does not purport to address all of the safety problems, 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 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products[3]
D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints[4]
D 215 Practice for the Chemical Analysis of White Linseed Oil Paints[3]
D 344 Test Methods for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts[3]
D 358 Specification for Wood to be Used as Panels in Weathering Tests of Coatings[5]
D 522 Test Methods for Mandrel Bend Test of Attached Organic Coatings[3]
D 523 Test Method for Specular Gloss[3]
D 562 Test Method for Consistency of Paints Using the Stormer Viscometer[3]
D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive[3]
D 660 Test Method for Evaluating Degree of Checking of Exterior Paints[3]
D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints[3]
D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints[3]
D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints[3]
D 869 Test Method for Evaluating Degree of Settling of Paint[5]
D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive[3]
D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood[3]
D 1014 Test Method for Conducting Exterior Exposure Tests of Paints on Steel[5]
D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems[3]
D 1296 Test Method for Odor of Volatile Solvents and Diluents[6]
D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes[5]
D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products[3]
D 1554 Terminology Relating to Wood-Base Fiber and Particle Panel Materials[7]
D 1640 Test Methods for Drying, Curing or Film Formation of Organic Coatings at Room Temperature[4]
D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials[3]
D 1736 Test Method for Efflorescence of Interior Wall Paints[5]
D 1849 Test Method for Package Stability of Paint[5]

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[1] This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct. 15, 1992. Published December 1992.

[2] Available from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.03.

[5] *Annual Book of ASTM Standards*, Vol 06.02.

[6] *Annual Book of ASTM Standards*, Vol 06.04.

[7] *Annual Book of ASTM Standards*, Vol 04.09.

D 2064 Test Method for Print Resistance of Architectural Paints[5]
D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer[3]
D 2197 Test Method for Adhesion of Organic Coatings by Scrape Adhesion[3]
D 2243 Test Method for Freeze-Thaw Resistance of Water-Borne Coatings[5]
D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates[3]
D 2369 Test Method for Volatile Content of Coatings[3]
D 2370 Test Method for Tensile Properties of Organic Coatings[3]
D 2486 Test Method for Scrub Resistance of Interior Latex Flat Wall Paints[5]
D 2574 Test Method for Resistance of Emulsion Paints in the Container to Attack by Microorganisms[3]
D 2805 Test Method for Hiding Power of Paints by Reflectometry[3]
D 2931 Guide for Testing Latex Flat Wall Paints[5]
D 3129 Guide for Testing Exterior Latex House Paints[5]
D 3168 Practice for Qualitative Identification of Polymers in Emulsion Paints[3]
D 3258 Test Method for Porosity of Paint Films[5]
D 3358 Guide for Testing Water-Borne Floor Paints[5]
D 3359 Test Methods for Measuring Adhesion by Tape Test[3]
D 3450 Test Method for Washability Properties of Interior Architectural Coatings[5]
D 3719 Test Method for Quantifying Dirt Collection on Coated Exterior Panels[5]
D 3723 Test Method for Pigment Content of Water Emulsion Paints by Low-Temperature Ashing[3]
D 3730 Guide for Testing High-Performance Interior Architectural Wall Coatings[5]
D 3792 Test Method for Water Content of Water-Reducible Paints by Direct Injection into a Gas Chromatograph[3]
D 3793 Test Method for Low-Temperature Coalescence of Latex Paint Films[5]
D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings[3]
D 3928 Method for Evaluation of Gloss or Sheen Uniformity[5]
D 3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings[3]
D 4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method[3]
D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser[3]
D 4062 Test Method for Levelling of Paints by Draw-Down Method[5]
D 4213 Test Method for Wet Abrasion Resistance of Interior Paints[5]
D 4214 Test Methods for Evaluating the Degree of Chalking of Exterior Paint Films[3]
D 4287 Test Method for High-Shear Viscosity Using the ICI Cone/Plate Viscometer[3]
D 4400 Test Methods for Sag Resistance of Paints Using a Multinotch Applicator[5]
D 4540 Guide for Testing Interior Latex Semigloss and Gloss Paints[5]
D 4585 Practice for Testing Water Resistance of Coatings Using Controlled Condensation[3]
D 4707 Test Method for Measurement of Paint Spatter Resistance to Roller Application[5]
D 4828 Test Method for Practical Evaluation of Washability of Organic Coatings[5]
D 4946 Test Method for Blocking Resistance of Architectural Paints[5]
D 4958 Test Method for Comparison of the Brush Drag of Latex Paints[5]
E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode[8]
E 105 Practice for Probability Sampling of Materials[9]
E 1347 Test Method for Color and Color Difference Measurement of Object-Color Specimens by Tristimulus (Filter) Colorimetry[3]

2.2 *U.S. Federal Test Method Standard No. 141:*[10]

2112 Application by Roller
2131 Application of Sprayed Films
2141 Application of Brushed Films
3011 Condition in Container
4541 Working Properties and Appearance of Dried Film
6301 Wet Adhesion (Tape Test)

## 3. Terminology

3.1 For definitions of terms in this guide refer to Terminology D 16 and D 1554.

## 4. Conditions Affecting Water-Reducible Coatings

4.1 *Interior and Exterior Coatings:*

4.1.1 *Substrate Type*—The substrate to be painted can affect not only the application properties of a coating, such as gloss and uniformity, but is also a factor in determining the type of coating to use. For instance, a primer-sealer may be required for porous substrates, such as new drywall, bare plaster, new wood or porous masonry. Other factors are the type and quality of metal, wood or wood composite (plywood, particle board or hardboard), the type, quality and alkalinity of concrete, plaster and joint cement systems, and the type and condition of any previous coatings.

4.1.2 *Substrate Conditions*—Conditions such as porosity and hardness determine the kind of coating that can be applied. The condition of previously painted substrates, such as degree of chalk, presence of grease, dirt, mold, and water-soluble or oily contaminants, film adhesion and porosity, all influence the performance of coatings. Smoothness of the substrate affects the spreading rate, final appearance, and texture.

4.1.3 Preparation of previously painted substrates including cleaning, solvent cleaning, and sanding.

4.1.4 Type and quality of primer or undercoat and time of drying before topcoating.

4.1.5 The application properties, even of interior water-reducible coatings, are affected by temperature and humidity at the time of application and during drying. As these

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[8] *Annual Book of ASTM Standards*, Vol 15.05.

[9] *Annual Book of ASTM Standards*, Vol 14.02.

[10] Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

**TABLE 1 List of Standards in Sectional Order**

| Property (or related test) | Section | ASTM Standard | Federal Test Method Standard 141 |
|---|---|---|---|
| Sampling | 6.2 | D 3925 | 1022 |
| Liquid Coating Properties | | | |
| Condition in container | 7.1 | | 3011 |
| Coarse particles and foreign matter | 7.2 | D 185 | |
| Density or weight per gallon | 7.3 | D 1475 | |
| Fineness of dispersion | 7.4 | D 1210 | |
| Odor | 7.5 | D 1296 | 4401 |
| Colorant acceptance | 7.6 | | |
| pH | 7.7 | E 70 | |
| Package stability | 7.8 | | |
| Heat stability | 7.8.1 | D 1849 | |
| Freeze-thaw stability | 7.8.2 | D 2243 | |
| Settling | 7.8.3 | D 869 | |
| Microorganism resistance | 7.8.4 | D 2574 | |
| Coating Application and Film Formation | | | |
| Application properties | 8.1 | | 4541 |
| Brush application | 8.1.1 | | 2141 |
| Brush drag | 8.1.1.1 | D 4958 | |
| Roller application | 8.1.2 | | 2112 |
| Roller spatter | 8.1.2.1 | D 4707 | |
| Spray application | 8.1.3 | | 2131 |
| Touch-up uniformity | 8.2 | D 3928 | |
| Low-temperature coalescence | 8.3 | D 3793 | |
| Rheological properties | 8.4 | | |
| Consistency (Low-shear viscosity) | 8.4.1 | D 562 | |
| Rheological properties of non-Newtonian materials | 8.4.2 | D 2196, D 4287 | |
| Sag resistance | 8.4.3 | D 4400 | 4494 |
| Levelling properties | 8.4.4 | D 4062 | |
| Drying properties | 8.5 | D 1640 | 4061 |
| Appearance of Dry Film | | | |
| Color differences by visual comparison | 9.1 | D 1729 | |
| Color differences using instrumental measurements | 9.2 | D 2244 | |
| Directional reflectance | 9.3 | E 1347 | |
| Gloss | 9.4 | | |
| Gloss, 60° | 9.4.1 | D 523 | |
| Sheen (85° gloss) | 9.4.2 | D 523 | |
| Hiding power | 9.5 | D 344, D 2805 | |
| Properties of Dry Film | | | |
| Interior and Exterior Coatings | 10.1 | | |
| Abrasion resistance | 10.1.1 | D 658, D 968, D 4060 | 6192 |
| Adhesion | 10.1.2 | D 2197, D 3359 | |
| Wet adhesion | 10.1.3 | | 6301 |
| Flexibility | 10.1.4 | D 522, D 2370 | 6221[A] |
| Resistance to household chemicals | 10.1.5 | D 1308 | |
| Efflorescence from the film | 10.1.6 | D 1736 | |
| Efflorescence from the substrate | 10.1.7 | | |
| Interior Finishes | 10.2 | | |
| Block resistance | 10.2.1 | D 4946 | |
| Print resistance | 10.2.2 | D 2064 | |
| Film porosity | 10.2.3 | D 3258 | |
| Washability and cleansability | 10.2.4 | | |
| Washability | 10.2.4.1 | D 2486, D 4213 | |
| Cleansability | 10.2.4.2 | D 3450, D 4828 | 6141[B] |
| Exterior Coatings | 10.3 | | |
| Adhesion to chalky surfaces | 10.3.1 | | 6301 |
| Dirt pick-up | 10.3.2 | D 3719 | |
| Fume resistance | 10.3.3 | 10.3.3.1 | |
| Blister resistance | 10.3.4 | D 4585 | |
| Exposure resistance | 10.3.5 | D 1006, D 1014 | |
| Chalking | 10.3.5.2 | D 4214 | |
| Checking | 10.3.5.3 | D 660 | |
| Cracking | 10.3.5.4 | D 661 | |
| Erosion | 10.3.5.5 | D 662 | |
| Flaking | 10.3.5.6 | D 772 | |
| Fade resistance | 10.3.5.7 | D 2244 | |
| Stain resistance | 10.3.6 | | |
| Coating Analysis | | | |
| Chemical analysis | 11.1 | D 215 | |
| Volatile content | 11.2 | D 2369 | |
| Volatile organic content | 11.3 | D 3960 | |
| Water content | 11.4 | D 3792, D 4017 | |
| Pigment content | 11.5 | D 3723 | |
| Pigment analysis | 11.6 | D 215 | 7261 |
| Nonvolatile vehicle identification | 11.7 | D 3168 | |

[A] Equivalent only to Method B of Test Method D 522.

[B] Except for scrub medium.

materials contain water, surfaces do not have to be completely dry before application. However, low temperature during drying may cause poor film formation.

4.2 *Exterior Finishes:*

4.2.1 *Substrate Weathering*—Weathering of wood before painting will probably adversely affect the performance of exterior coatings. Some weathering of masonry surfaces may have beneficial effects on the performance.

4.2.2 *Substrate Aspects of the Building*—If construction defects or defects due to age are such that excessive moisture from the inside or the outside makes its way through the substrate or if the substrate is in direct contact with damp ground, blistering, flaking or peeling may result.

4.2.3 Environmental conditions after application, both general for the area and specific, such as under eaves, behind shrubbery, northside and southside exposure.

## 5. Selection of Tests

5.1 Because the conditions to which a coating is subjected vary with (*a*) the surface type: wall, floor, ceiling, and (*b*) the service environment: exterior or interior, specialized types of water-borne coatings have been developed for the different locations. The recommended test methods presented in Tables 1 and 2 cover practically all of the properties of water-reducible coatings but all of them are not required with each type. Coatings intended for exterior use only or both exterior and interior use require certain properties not relevant to those for interior use only. Selection of the methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and the seller.

5.2 The purchaser should first determine the properties a coating should have and then select only those test methods that measure or evaluate those properties. After selecting the desired tests, the purchaser should then decide which properties are the most important and establish the requirements or specifications accordingly. Since coating properties frequently tend to oppose each other, such as low sheen versus good cleansability, some properties may need to be less emphasized if others are to be accentuated. This balance of properties must be considered when selecting the tests and establishing the requirements. The significance of the tests and the normal range of values are presented in the different sections, in most cases.

5.3 This guide does not indicate relative importance of the various tests nor does it recommend specific test values because properties very important to one purchaser may be less so to another.

## 6. Sampling

6.1 Prior to sampling, the condition of the container should be established since damage to it may cause evaporation, skinning, or other undesirable effects on the coating.

6.2 Sample in accordance with Practice D 3925. Determine the density in pounds per gallon (kilograms/litre) in accordance with Test Method D 1475. Continue sampling and determining density until successive results agree within 0.1 lb (45 g) or as agreed upon between the purchaser and seller. Then take samples for testing.

6.3 Specify the amount required for a representative sample, the package sizes, and an identification code. A 1-U.S. gal (or 4-L) sample is usually sufficient for the recommended tests, but for guidance in selecting a sampling plan consult Practice E 105.

## 7. Liquid Coating Properties

7.1 *Condition in Container*—Thickening, pigment settling, and liquid separation are undesirable and objectionable if material that has been stored cannot be readily reconditioned and made suitable for application with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties after storage and lists characteristics that are undesirable and objectionable in a stored paint. Determine condition in the container in accordance with Method 3011 of Federal Test Method Standard No. 141. (See also 7.10 Package Stability.)

7.2 *Coarse Particles and Foreign Matter*—Liquid coatings must be free of coarse particles and foreign matter to be able to form uniform films of good appearance, a typical maximum being 0.5 weight % of the total material. The referenced method with a 325-mesh (45-μm) screen gives the percent of these particles. Determine content of coarse particles and foreign matter in accordance with Test Method D 185.

7.2.1 Another test method used in industry to determine whether coarse particles are present in the dry film of a low-gloss finish is to scrape the surface of the film with a spatula or metal edge of a ruler. Any particles larger than 325 mesh can be clearly seen after the surface has been scraped.

7.3 *Density or Weight per Gallon*—The density measured in pounds per gallon (kilograms per litre = g/ml) is used to ensure product uniformity from batch to batch, provides a check against the theoretical weight calculated from the formula, and is useful for determining the similarity of two samples. The referenced method gives a procedure for measuring the density of the coating at a specified temperature. Most paints have densities of about 10 to 12 lb/gal (1.2 to 1.4 kg/L). Determine density in accordance with Test Method D 1475, using a calibrated weight per gallon cup.

7.4 *Fineness of Dispersion*—Generally, the more finely a pigment is dispersed the more efficiently it is being utilized. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the liquid coating down a calibrated tapered groove varying in depth from 4 to 0 mils (100 to 0 μm) (0 to 8 Hegman units). The depth at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the wet film is taken as the fineness of dispersion value. Higher readings in Hegman units or lower readings in mils or micrometres indicate finer dispersion. Most interior semigloss and gloss latex coatings have a fineness of about 1.5 to 0.3 mils (5 to 7.5 Hegman or 40 to 7 μm) while lower gloss finishes do not generally require a dispersion finer than 3 to 2.5 mils (2 to 3 Hegman). Some interior flat latex paints have finenesses as low as 3.5 mils (1 Hegman or 90 μm). Determine fineness of dispersion in accordance with Test Method D 1210.

7.4.1 The referenced method was designed primarily for coatings with good fineness of dispersion, such as high gloss finishes. Some interior flat paints contain pigments so coarse that it is impractical to measure the fineness with a grind gage because the agglomerates are carried along by the scraper.

NOTE 3—The fast drying of latex paints makes it difficult to make measurements of this type.

7.5 *Odor*—One of the advantages of latex paints is that they contain little if any organic solvent. Thus interior latex paints do not have odors characteristic of solvent-borne coatings. However, other ingredients, such as ammonia, may be used that might also be objectionable in confined spaces. Hence, interior latex paints should be tested for odor acceptability. Although there is no specific ASTM test method for evaluating odor of water-borne coatings, the industry does attempt to measure this property. Determine whether the paint has an unpleasant or irritating odor as agreed upon between the purchaser and seller, taking adequate precautions to ensure the safety of the operator. Test Method D 1296 may be suitable as the basis for a test.

7.6 *Colorant Acceptance*—Tintability of white bases with colorants of standardized tinting strength is a trade requirement. If tinting colors are not adequately compatible with tint bases, lighter, darker, or nonuniform shades of colors are produced. Suitable test methods should be agreed upon between the purchaser and the seller.

7.7 *pH*—Latex paints with low (acidic) pH may corrode metal containers. To avoid this problem, the pH is normally stabilized within the range from about 5 to 10, depending upon the type of latex used and the general formulation. The pH does not determine the quality of a latex paint and should be used only to ensure product uniformity. However, a change in pH during storage may indicate poor stability and an unacceptable change in the properties of a latex paint. Determine pH in accordance with Test Method E 70.

7.8 *Package Stability*—Since paints are normally not used immediately after manufacture, they must remain stable in the can for some time. At normal temperatures most water-borne coatings can be stored for over a year with little change in properties. However, exposure in uninsulated warehouses or during shipping to high temperatures in the summer or to low temperatures in the winter may cause unacceptable changes in these products. Other unsatisfactory conditions that may occur during storage are excessive settling and microbiological attack.

7.8.1 *Heat Stability*—Exposure in service to high temperatures can be used to test for the stability of a packaged coating that frequently encounters such conditions in service, or as an accelerated test to predict stability when stored at temperatures above freezing. Although inductions of long term package stability can usually be obtained in several days or weeks at an elevated temperature, such as 125°F (50°C) or 140°F (60°C), occasionally the results of the accelerated test do not agree with those at prolonged normal storage conditions. In the referenced method the changes in consistency and certain other properties of the accelerated aged material are compared to those occurring in a control kept at normal temperatures for a longer period. When testing for heat stability, as such, changes in viscosity, flow, gloss, pH, foam resistance, color uniformity, and wet adhesion are usually checked. Determine heat stability in accordance with Test Method D 1849.

7.8.2 *Freeze-Thaw Stability*—Water-borne coatings may be subjected to freezing conditions during shipping and storage. Suitably stabilized products can resist several cycles of freezing and thawing without showing deleterious changes such as coagulation, graininess (seeding), or excessive viscosity increase. Many latex paints that increase in viscosity can still be considered usable, if other properties that may be affected by a higher viscosity, such as levelling and brushability, are satisfactory. Determine freeze-thaw stability in accordance with Test Method D 2243.

7.8.3 *Settling*—Latex paints are generally resistant to hard settling, but do at times show separation and soft settling. The referenced method covers the degree of pigment suspension in and ease of remixing of a shelf-aged specimen to a homogeneous condition suitable for the intended use. Determine settling in accordance with Test Method D 869.

7.8.4 *Microorganism Resistance*—Microorganisms in a water-borne coating can cause gassing, putrefactive or fermentative odors, and loss of viscosity. Determine if the paint contains living bacteria and if it is resistant to attack by bacteria in accordance with Test Method D 2574.

## 8. Coating Application and Film Formation

8.1 *Application Properties*—Application or working properties of a paint are generally compared to a standard or described by requirements in the product specification. Determine working properties in accordance with Method 4541 of Federal Test Method Standard No. 141.

8.1.1 *Brush Application*—Brushed films should be smooth and free of seeds and on vertical surfaces should show no sagging, color streaking, nor excessive brush marks. Brush drag should not be excessive although some degree of drag may be desirable for adequate film thickness application. Wall finishes are tested on vertical surfaces and floor coatings on horizontal surfaces, although evaluation of the latter on vertical surfaces may be necessary to determine performance on stair risers, railings, posts, etc. The referenced method covers a means for the determination of the brushing properties of a coating. Even though the test is subjective, someone experienced in the art can produce quite consistent results. Determine brushing properties in accordance with Method 2141 of Federal Test Method Standard No. 141.

8.1.1.1 *Brush Drag*—As the brush drag (resistance encountered when applying a coating by brush) increases, any natural tendency of the painter to overspread the paint is reduced. All other factors being constant, increased brush drag results in greater film thickness with consequent improvements in hiding and film durability. Conversely, increasing brush drag too much can cause difficulties in spreading the paint easily and uniformly, leading to excessive sagging, prolonged drying time and, in highly pigmented latex paints, possibly to "mud-cracking" due to excessive thickness. The referenced method covers the determination of relative brush drag of a series of coatings applied by brush by the same operator. It has been established that the subjective ratings thus obtained correlate well with high shear viscosities obtained instrumentally using Test Method D 4287 (see 8.4.2), provided that the paints differ in viscosity by at least 0.3 poise (0.03 Pa·s). Determine brush drag ratings in accordance with Test Method D 4958.

8.1.2 *Roller Application*—Both wall and floor coatings are frequently applied by roller. This type of application tends to produce some stipple pattern. The referenced method covers the evaluation of a material's characteristics when applied by roller. Since foaming often occurs when water-borne coatings are roller applied, the amount of foam produced, and the number of craters that remain after the bubbles have broken should be determined during the test. Determine roller coating properties in accordance with Method 2112 of Federal Test Method Standard No. 141.

8.1.2.1 Some coatings spatter more than others when applied by roller. The degree to which a paint spatters when roller applied can be determined by the density of the spatter. In the referenced method a specially designed notched spool is rolled through a film of the test material that has been applied to a plastic panel. Any spatter generated falls upon a catch paper and after drying is rated against photographic standards. This procedure eliminates the influence of the roller cover, thus determining the spattering characteristics of the paint alone. Determine spatter resistance in accordance with Test Method D 4707.

8.1.3 *Spray Application*—Architectural coatings are sometimes applied by spray. Both air and airless spray are used on commercial work. Determine spray application properties in accordance with Method 2131 of Federal Test Method Standard No. 141. Manual application is very subjective and should be performed only by an individual skilled in the art of using spray equipment.

8.2 *Touch-Up Uniformity*—After a paint has dried, areas where less material was applied sometimes become noticeable. If the paint has suitable touch-up properties, additional material can be applied to these areas only, instead of refinishing the complete wall. The color, gloss, and levelling of the touched-up areas and the previously coated area should be uniform. Differences in these properties are often caused by short wet-edge time, poor levelling on recoat, and pigment orientation or flotation during and after application. Determine touch-up properties in accordance with Test Method D 3928.

8.3 *Low-Temperature Coalescence*—If a latex paint is applied at too low a temperature it will not form a coherent film. The referenced test method determines how well the latex particles fuse together or coalesce to form a continous film at low temperatures. Determine low-temperature coalescence of a series of coatings or reformulations in accordance with Test Method D 3793.

NOTE 4—Because of the poor reproducibility of this method with numerical values, it cannot be used to compare such results from different laboratories. Interlaboratory agreement is improved significantly when rankings are used.

8.4 *Rheological Properties:*

8.4.1 *Consistency (Low-Shear Viscosity)*—Consistency is important, relating to application and flow, and should fall within a stated range for satisfactory reproduction of a specific formula. While consistency is an important property it does not determine the quality of a coating and should be used mainly to ensure product uniformity. In the referenced method, consistency is defined as the load in grams to produce a specified rate of shear. The load value is frequently converted to Krebs Units (KU) and the Stormer consistency reported on that basis. Although the consistency of most latex paints is about 150 to 300 g/100 revolutions, a much wider range is possible because of the great variation that may occur in the rheological properties of these paints. Two paints of the same consistency may have quite different rheological properties during application. Determine consistency in accordance with Test Method D 562.

8.4.2 *Rheological Properties of Non-Newtonian Materials*—Rheological properties are related to application and flow characteristics of the liquid coating. The referenced methods cover the determination of rheological properties and are particularly suited for coatings that display thixotropic characteristics. However, they measure viscosity under different shear rates. In Test Method D 4287 there is only one rate but it is similar to that occurring during brush application so that the measured viscosity is related to brush drag, spreading rate and film build. Test Method D 2196 includes procedures for measuring viscosity at several shear rates to determine the amount of shear thinning and the viscosity change at low shear rates. The results can be used to evaluate sag resistance and levelling ability. Determine rheological properties in accordance with Test Methods D 2196 or D 4287 or both.

8.4.3 *Sag Resistance*—Some coatings sag and form curtains before the film sets. Resistance to this type of flow is an important property particularly for semigloss and gloss finishes because of the unsightly film appearance. Determine sag resistance in accordance with Test Method D 4400.

8.4.4 *Levelling Properties*—Levelling is an important property when smooth, uniform surfaces are to be produced, because it affects hiding and appearance. Brush marks and imperfections are much more conspicuous in semigloss and gloss finishes than they are in low gloss materials. In the referenced method a series of ridges is produced using a levelling rod and after drying they are compared to levelness standards. Determine levelling in accordance with Test Method D 4062.

8.5 *Drying Properties*—The drying time of a coating is important in determining when a freshly painted room, floor or stair can be put back in use. Slow drying may result in dirt or insect pickup causing a poor appearance or, if on an exterior surface, rain or dew may cause a nonuniform appearance. The drying time of a coating is determined by its composition and by atmospheric conditions during drying. Most latex paints dry to touch in 1 to 2 h when the water has evaporated from the film. Low gloss finishes can usually be recoated from within a few hours to 18 h. Because of the glycols present in semigloss and gloss latex coatings it is prudent not to recoat before at least 18 h drying. Curing to obtain the ultimate properties may take only a few days for some latex paints while others may require 1 to 2 weeks, depending upon the composition. Any one of the several methods for determining the various stages of film formation in the drying or curing of organic coatings may be used. For example, if two coats are specified the determination of "dry-to-recoat" time is important. Determine appropriate drying time(s) in accordance with Test Method D 1640.

## 9. Appearance of Dry Film

9.1 *Color Differences by Visual Comparison*—Visual comparison of colors is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric, and geometric characteristics of light source, illuminating and viewing conditions, sizes of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials relative to their standards. Determine color difference in accordance with Practice D 1729.

9.2 *Color Differences Using Instrumental Measurements*—The difference in color between a product and its standard can be measured by instrument. Generally the tolerance is agreed upon by the purchaser and seller and may also be required if a product specification is involved. Color measuring instruments provide numerical values that can be compared to subsequent measurements. The referenced method covers the calculation of instrumental determinations of small color differences observable in daylight illumination between nonfluorescent, nonmetameric, opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation (9.1) should be used to verify the results. Calculate in accordance with Test Method D 2244 the color differences that have been measured instrumentally.

9.3 *Directional Reflectance*—This property is a measure of the appearance of lightness of a coating. It is usually assigned a value in specifications for white and pastel shades, a typical range being 76 to 86 % for white finishes. In the referenced method the directions of illumination and viewing are specified so as to eliminate the effect of gloss. Determine daylight directional reflectance in accordance with Test Method E 1347.

9.4 *Gloss*—This property is a measure of the capability of a coating surface to reflect light in a mirror-like (specular) manner, that is, light strikes the surface and is reflected at the equal but opposite angle. In the referenced method the numerical gloss units are the ratio of light reflected by a specimen to that reflected by the primary black glass that is assigned a gloss value of 100. The gloss of some coatings varies greatly with the angle of incidence so that a complete description of their gloss would require measurements over a wide range of angle. In practice, the gloss of architectural finishes is adequately characterized by measurements at 60° or 85° (or both) from a line perpendicular (normal) to the surface. The 85° angle is a very low "grazing" angle (5°) of illuminating and viewing the surface and the gloss at this angle is called "sheen". Attempts to standardize the levels of gloss associated with the several descriptive terms have not been very successful since the gloss scale is continuous with no distinct boundaries. Hence, there is some overlap at the ends of some classifications in common usage.

9.4.1 *Gloss, 60°*—Semigloss finishes are particularly sensitive to poor enamel hold-out of primers and undercoats. Low or uneven gloss readings are indicative of this defect. Low gloss finishes range from 0 to 20 while exterior latex house paints may vary from 5 to 60. A range from 20 to 40 is typical of water-borne floor finishes after drying for a few days. Interior semiglosses vary from 35 to 70 but measurements taken shortly after drying should be repeated after one week because the gloss can drop considerably in the first few days of drying. Determine the 60° gloss in accordance with Test Method D 523.

9.4.2 *Sheen (85° Gloss)*—Although low-gloss paints with good uniformity of appearance at low angles of viewing often have little sheen while those with good cleansability usually have moderate sheen, this is not always the case so that sheen should not be used as a measure of other paint properties. The referenced method, using the 85° geometry, is useful in characterizing the low-angle appearance of low-gloss coatings. Most flat wall paints have a sheen of 1 to 10 whereas velvets or eggshells range from 15 to 35. Determine the sheen (85° gloss) in accordance with Test Method D 523.

NOTE 5—This property is of particular interest with flat to low-gloss finishes.

9.5 *Hiding Power*—Hiding power is a measure of the ability of a coating to obscure the substrate and is usually expressed as the spreading rate for a specified level of opacity. It is, however, dependent on uniformity of film thickness, which in practical applications is influenced by flow, levelling and application properties of the coating. Test Method D 2805 is precise and gives an absolute rather than a comparative result. Paint is applied with an applicator bar to minimize the effects of flow and levelling, film thickness is rigorously measured, and film opacity is determined instrumentally. Test Method D 344 is a practical test in which paint is applied with a brush, wet-film thickness is approximately controlled by spreading rate, and hiding power is evaluated visually by comparison with a standard paint, but results are affected by flow and levelling of the materials. Determine hiding power in accordance with Test Methods D 344 or D 2805.

## 10. Properties of the Dry Film

10.1 *Interior and Exterior Coatings:*

10.1.1 *Abrasion Resistance*—Abrasion resistance is a measure of the ability of a dried film to withstand wear from foot traffic and marring from objects rolled or pulled across the surface. In the referenced methods, dry abrasive is applied to a coated panel using the force of gravity or a jet blast for free-flowing abrasive or a weighted wheel for abrasive embedded in a resilient rubber matrix. Determine dry abrasion resistance in accordance with Test Methods D 658, D 968 or D 4060. (See 10.2.4.1 for wet abrasion resistance.)

NOTE 6—Because of the poor reproducibility of abrasion test methods, testing should be restricted to only one laboratory when numerical abrasion resistance values are to be used. Interlaboratory agreement is improved significantly when rankings are used in place of numerical values.

10.1.2 *Adhesion*—Adhesion, the ability of a film to resist removal from the substrate, is an important property of a coating. Determine adhesion in accordance with Test Method D 2197 or D 3359 or both.

10.1.3 *Wet Adhesion*—It is essential that a finish adhere tightly to a given substrate or primer under the wet conditions of washing or scrubbing. There is no adequate test method published by ASTM. Determine the wet adhesion of exterior latex paints in accordance with Method 6301 of Federal Test Method Standard No. 141.

10.1.4 *Flexibility*—Elongation is a measure of the flexibility of a coating film. Most semigloss and full gloss water-borne coatings can be bent over a 1/8-in. (3.2-mm) mandrel without affecting the film. However, interior flat and eggshell finishes usually pass at 1/4-in. (6.4-mm). For exterior coatings Test Method D 2370 is a much more discriminating method.[11] Determine flexibility in accordance with Test Methods D 522 or elongation with D 2370.

10.1.5 *Resistance to Household Chemicals*—An important property of some finishes is their ability to resist spotting, softening or removal when subjected to household chemicals or strong cleaners. Determine resistance to these chemicals in accordance with Test Method D 1308.

10.1.6 *Efflorescence from the Film*—Salt formation is produced by specific conditions of temperature and humidity if a paint contains sufficient solid water-soluble material to cause a noticeable deposit on the film. However, because of the improvements in latex and latex paint formulations few interior latex paints effloresce. The referenced method measures efflorescence that comes from the paint itself, not from the substrate. Determine efflorescence resistance in accordance with Test Method D 1736.

10.1.7 *Efflorescence from the Substrate*—Cementitious substances may contain sufficient solid water-soluble materials to cause a surface deposit through leaching and evaporation. Currently there is no adequate test method published by ASTM.

10.2 *Interior Finishes:*

10.2.1 *Block Resistance*—This property is important for interior semigloss and gloss finishes since it governs the resistance of surfaces of dried coatings to sticking together when stacked or placed in contact with each other. An interior finish often comes in contact with itself, especially on doors, windows and drawers where it sometimes sticks to itself (blocks) depending on the hardness of the coating, the pressure, temperature, humidity, and time that the surfaces are in contact. The referenced method covers an accelerated blocking resistance procedure developed especially for architectural coatings. Determine blocking resistance in accordance with Test Method D 4946.

10.2.2 *Print Resistance*—The ability of a coating to resist printing is important because its appearance is adversely affected if the surface texture is modified by contact with another surface, particularly one with a pattern. Interior gloss and semigloss systems on window sills and other horizontal surfaces often have flower pots placed on them that may tend to leave a permanent impression from the pressure. This tendency for a paint film to "print" is often a function of the hardness of the coating, the pressure, temperature, humidity, and time that the two surfaces are in contact. Test for print resistance in accordance with Test Method D 2064.

10.2.3 *Film Porosity*—The more porous a paint film is, the worse its cleansability and enamel holdout. In the referenced method a special, colored penetrating medium is applied to the coating and the change in reflectance indicates the degree of porosity. Determine film porosity in accordance with Test Method D 3258.

10.2.4 *Washability and Cleansability*—The capability of satisfactorily removing marks without damaging the film is essential for good performance of interior finishes. A coating may be washable, that is, unaffected by the detergent solution, but may not have good cleansability. Frequently the difference between the two terms, "cleansability" and "washability" is not clearly understood so that there is confusion as to what is really being tested; for example, the title of Test Method D 3450. Cleansability is evaluated by applying one or more stains and soils and determining how readily they are removed. Washability is evaluated by determining the resistance of the film to wet erosion either by visual assessment or measured film loss. In general, the precision of both types of test is poor because several properties, such as hardness, water and detergent resistance, cohesion and adhesion, are involved and the endpoint, except for the wet abrasion method, is rather indefinite.

10.2.4.1 *Washability (Also referred to as Scrubbing or Wet Abrasion Resistance)*—The scrubbing method, Test Method D 2486, developed for interior latex flat wall paints can be applied to coatings of almost any type. In it the coating is applied to a black plastic panel that, during scrubbing with a nylon brush and abrasive cleaning agent, is raised by a narrow shim to concentrate the test area. The number of back-and-forth strokes (cycles) required to remove the film over the shim is determined. Interior latex flat paints can vary in scrub resistance from less than 100 to more than 1000 cycles. The wet-abrasion method, Test Method D 4213, is similar except that a sponge is used in place of the bristle brush while the shim is not used. This method also provides for the use of a nonabrasive medium with paints having very low abrasion resistance. The weight or volume loss per 100 cycles to erode the film almost to exposure of the black substrate is the measure of scrub resistance. Evaluate washability, as just described, in accordance with Test Methods D 2486 or D 4213.

10.2.4.2 *Cleansability*—The older referenced method, Test Method D 3450, is similar to the wet-abrasion method, Test Method D 4213, except that the sponge is used with either the nonabrasive or abrasive cleaning agent to remove a carbon black-oil stain. The ability to remove the stain is expressed as the ratio (in percent relative) of the reflectance of the cleaned area to that of the area before application of the stain. In Test Method D 4828, referred to as a "practical" test, numerous staining and soiling agents found in service and commercial abrasive or nonabrasive cleaners as well as the standardized cleaning agents can be used. In the current edition the films are cleansed only manually with the result that the method is not suitable for interlaboratory testing. Evaluate ease or removability in accordance with Test Methods D 3450 or D 4828.

10.3 *Exterior Coatings:*

10.3.1 *Adhesion to Chalky Surfaces*—Latex paints generally have little ability to penetrate powder substrates. Consequently, adhesion to previous coatings that have chalked is poor unless the latex paint has been modified to penetrate and bind the chalk layer to the old coating. However, certain latices do exist that are designed to adhere well to chalky surfaces so do not require modification. There are no directly applicable ASTM or Federal Test Method Standard No. 141 test methods for adhesion to chalky surfaces, although work is still going on. The industry generally uses a pressure-sensitive tape to test for this property. The tape is pressed firmly onto the dried latex film (fresh dry films do not adhere as well as aged dry films) and then removed rapidly by pulling back upon itself. Method 6301 of Standard 141

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[11] Ashton, H. E., "Flexibility and its Retention in Clear Coatings Exposed to Weathering," *Journal of Coatings Technology*, Vol 51, No. 653, June 1979, p. 41.

describes a similar method but includes water exposure.

10.3.2 *Dirt Pickup*—Low-gloss exterior latex paints generally have good resistance to dirt pickup. Gloss or semigloss latex paints may be more subject to this type of disfigurement. Exterior exposure, particularly under an overhang (soffit), should indicate in a relatively short time (about 1 year) a paint's tendency to this defect. Determine degree of dirt collection in accordance with Test Method D 3719.

10.3.3 *Fume Resistance*—Some paints exhibit a change in appearance (usually color) when subjected to air containing certain sulfur compounds, notably hydrogen sulfide and sulfur dioxide. This type of atmosphere may be present near industrial or other polluted areas and can cause a paint to yellow or darken in as little time as overnight. There are no ASTM or Federal test methods for evaluating this color change, but one procedure used by the industry is as follows:

10.3.3.1 Apply a sufficient number of coats of the paint to two glass plates to hide the surface completely, allow to dry for 6 h and expose one in a moist atmosphere of hydrogen sulfide for 18 h. Compare the color with the unexposed plate. The color difference should not exceed that between plates that have been coated with a paint made with titanium dioxide pigment, lead-free zinc oxide, raw or refined linseed oil, and sufficient cobalt added for drying, and similarly treated.

10.3.4 *Blister Resistance*—Blister resistance is the ability of a dry film on wood to resist the formation of blisters caused by water from the wood substrate. In practice water can come from either the interior of a home or from the structural defects that permit entry of exterior water behind the wood. Moisture blister resistance can be qualitatively evaluated in a laboratory test. Determine resistance to moisture blistering in accordance with Practice D 4585.

NOTE 7—Latex paints are frequently promoted on the basis of their ability to allow moisture to escape without causing blisters, so they should be tested for this property.

10.3.5 *Exposure Resistance*—If the coating is intended for exterior use, evaluation of the resistance to weathering may be required. In conducting exterior exposures follow Practice D 1006 for wood substrates or Test Method D 1014 for steel.

10.3.5.1 In establishing exterior performance on wood, use the panels described in Specification D 358 or as agreed upon between the purchaser and the seller.

10.3.5.2 *Degree of Chalking*—Determine the rating using Test Method D 4214.

10.3.5.3 *Degree of Checking*—Determine the rating using Test Method D 660.

10.3.5.4 *Degree of Cracking*—Determine the rating using Test Method D 661.

10.3.5.5 *Degree of Erosion*—Determine the rating using Test Method D 662.

10.3.5.6 *Degree of Flaking*—Determine the rating using Test Method D 772.

10.3.5.7 *Fade Resistance*—Exterior latex paints usually have good color retention because of their good chalk resistance. However, the use of improper latex, pigment volume concentration, or pigments can lead to fading. There are no ASTM nor Federal test methods specifically designed for evaluating fade resistance, but the change in color on exposure can be measured in accordance with Test Method D 2244 (see 9.2).

10.3.6 *Stain Resistance*—There is neither an ASTM nor a Federal test method available for evaluating stain resistance of exterior latex paints. There are, of course, different kinds of stain such as the water-soluble extractives in wood substrates, the formation of lead or mercury sulfides, and rundown from metal surfaces of oxides that disfigure the surface. Different kinds of test methods are required for water-soluble and insoluble stains.

## 11. Coating Analysis

11.1 *Chemical Analysis*—If a specification requires certain raw materials or certain components in a given amount then analysis is needed to determine whether the specified components are present and in what amounts. Analysis is primarily a measure of uniformity and does not necessarily establish quality that can also be greatly affected by manufacturing techniques. No single schematic analysis is comprehensive enough to cover the wide variety of paint compositions. Most ASTM analytical methods apply to solvent-borne coatings. However, some of them can be adapted to analysis of the water-reducible type. Select test procedures from Practice D 215 and ASTM methods that are pertinent to the components of water-borne coatings.

11.2 *Volatile Content*—The percent of volatile matter is a measure of the amount of a liquid coating lost as it dries. This quantity is not necessarily indicative of the quality of the coating. It is useful, however, for determining the similarity of two batches. The referenced method covers the determination of the volatile content by weight of solvent- and water-reducible coatings. The quantity determined subtracted from 100 % gives the nonvolatile content of the coating. Determine the volatile content in accordance with Test Method D 2369.

11.3 *Volatile Organic Compound (VOC) Content*—Several local jurisdictions have adopted air pollution controls that severely limit the amount of VOC permitted in architectural coatings, including interior latex gloss and semigloss paints. Since these paints may contain solvent such as coalescent and co-solvent wet-edge aids, it is essential that these products not exceed the established VOC limits. Determine VOC content in accordance with Practice D 3960.

11.4 *Water Content*—The amount of water may be required in the calculation of the VOC of coatings. The referenced methods cover the determination of the total water content of water-borne coatings, one using gas-liquid chromatography and the other the Karl Fischer reaction. Determine water content in accordance with Test Methods D 3792 or D 4017.

11.5 *Pigment Content*—Pigment provides the hiding and color and influences many other properties of a coating. The referenced method describes the procedure for the low-temperature ashing of water-borne coatings. Some of these coatings may contain ingredients that lose water of hydration or decompose at the test temperature. Consequently, caution must be exercised in applying the method to materials containing unknown pigment compositions. If difficulties or disagreements are encountered with this procedure, the pigment should be separated from the binder using a centrifuge. Determine the pigment content in accordance with Test Method D 3723.

11.6 *Pigment Analysis*—The analysis of pigment may be required if the product is covered by a specification or upon agreement between the purchaser and seller. Analyze the pigment in accordance with selected test procedures from Practice D 215 and appropriate ASTM methods.

11.7 *Identification of Nonvolatile Vehicle*—The type of binder used in a coating has a great influence on its properties. The referenced method covers the qualitative characterization or identification of the extracted vehicle by infrared spectroscopy and pyrolysis of the paint followed by gas-liquid chromatography. It is useful in detecting batch to batch uniformity and the presence of major adulterants. Identify the nonvolatile vehicle in accordance with Practice D 3168.

## 12. Field Testing

12.1 Although many of the recommended test methods attempt to simulate conditions under which water-reducible coatings are applied and used, it is not possible to duplicate accurately all possible conditions. Testing materials under field conditions is recommended for the final evaluation of suitability.

## TABLE 2 Alphabetical List of Properties

| Property (or related test) | Section | ASTM Standard | Federal Test Method Standard 141 |
|---|---|---|---|
| Abrasion Resistance | 10.1.1 | D 658, D 968, D 4060 | 6192 |
| Adhesion | 10.1.2 | D 2197, D 3359 | |
| Adhesion to chalky surfaces | 10.3.1 | | 6301 |
| Analysis, chemical | 11.1 | D 215 | |
| Application properties | 8.1 | | 4541 |
| Blister resistance | 10.3.4 | D 4585 | |
| Block resistance | 10.2.1 | D 4946 | |
| Brush application | 8.1.1 | | 2141 |
| Brush drag | 8.1.1.1 | D 4958 | |
| Chalking | 10.3.5.2 | D 4214 | |
| Checking | 10.3.5.3 | D 660 | |
| Cleansability | 10.2.4.2 | D 3450, D 4828 | 6141[A] |
| Coarse particles and foreign matter | 7.2 | D 185 | |
| Colorant acceptance | 7.6 | | |
| Color differences by visual comparison | 9.1 | D 1729 | |
| Color differences using instrumental measurements | 9.2 | D 2244 | |
| Condition in container | 7.1 | | 3011 |
| Consistency (Low-shear viscosity) | 8.4.1 | D 562 | |
| Cracking | 10.3.5.4 | D 661 | |
| Density or weight per gal | 7.3 | D 1475 | |
| Dirt pick-up | 10.3.2 | D 3719 | |
| Drying properties | 8.5 | D 1640 | 4061 |
| Efflorescence from the film | 10.1.6 | D 1736 | |
| Efflorescence from the film substrate | 10.1.7 | | |
| Erosion | 10.3.5.5 | D 662 | |
| Exposure resistance | 10.3.5 | D 1006, D 1014 | |
| Fade resistance | 10.3.5.7 | D 2244 | |
| Film porosity | 10.2.3 | D 3258 | |
| Fineness of dispersion | 7.4 | D 1210 | |
| Flaking | 10.3.5.6 | D 772 | |
| Flexibility | 10.1.4 | D 522, D 2370 | 6221[B] |
| Freeze-thaw stability | 7.8.2 | D 2243 | |
| Fume resistance | 10.3.3 | 10.3.3.1 | |
| Gloss | 9.4 | | |
| Gloss, 60° | 9.4.1 | D 523 | |
| Heat stability | 7.8.1 | D 1849 | |
| Hiding power | 9.5 | D 344, D 2805 | |
| Levelling properties | 8.4.4 | D 4062 | |
| Low-temperature coalescence | 8.3 | D 3793 | |
| Microorganism resistance | 7.8.4 | D 2574 | |
| Nonvolatile vehicle identification | 11.7 | D 3168 | |
| Odor | 7.5 | D 1296 | 4401 |
| Package stability | 7.8 | | |
| pH | 7.7 | E 70 | |
| Pigment analysis | 11.6 | D 215 | 7261 |
| Pigment content | 11.5 | D 3723 | |
| Reflectance, directional | 9.3 | E 1347 | |
| Resistance to household chemicals | 10.1.5 | D 1308 | |
| Rheological properties of non-Newtonian materials | 8.4.2 | D 2196, D 4287 | |
| Roller application | 8.1.2 | | 2112 |
| Roller spatter | 8.1.2.1 | D 4707 | |
| Sag resistance | 8.4.3 | D 4400 | 4494 |
| Sampling | 6.2 | D 3925 | 1022 |
| Settling | 7.8.3 | D 869 | |
| Sheen (85° gloss) | 9.4.2 | D 523 | |
| Spray application | 8.1.3 | | 2131 |
| Stain resistance | 10.3.6 | | |
| Touch-up uniformity | 8.2 | D 3928 | |
| Volatile content | 11.2 | D 2369 | |
| Volatile organic content (VOC) | 11.3 | D 3960 | |
| Washability | 10.2.4.1 | D 2486, D 4213 | |
| Water content | 11.4 | D 3792, D 4017 | |
| Wet adhesion | 10.1.3 | | 6301 |

[A] Except for scrub medium.

[B] Equivalent only to Method B of D 522.

Designation: D 5326 – 92

# Standard Test Method for Color Development in Tinted Latex Paints[1]

This standard is issued under the fixed designation D 5326; 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 a method for measuring color development in tinted latex paints, for the purpose of determining the efficiency of colorants and the potential for poor color uniformity of applied paint films.

1.2 *This standard does not purport to address all of the safety problems, 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 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products[2]

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Coordinates[2]

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials[2]

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings[2]

E 284 Terminology of Appearance[2]

E 1164 Practice for Obtaining Spectrophotometric Data for Object-Color Evaluation[2]

E 1331 Test Method for Reflectance Factor and Color by Spectrophotometry Using Hemispherical Geometry[2]

E 1345 Practice for Reducing the Variability of Color Measurements by the Use of Multiple Measurements[2]

E 1347 Test Method for Color and Color-Difference Measurement of Object-Color Specimens by Tristimulus (Filter) Colorimetry[2]

E 1349 Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional Geometry[2]

## 3. Terminology

3.1 *Definitions:*

3.1.1 *color development*—the extent to which the colorant has achieved its full tinting potential, as evidenced by the color change or lack thereof, when the tinted paint is subjected to very strong shear stress.

3.2 See Terminology D 16, E 284, and the *Paint/Coatings Dictionary*[3] for definitions of other terms used in this test method.

## 4. Summary of Test Method

4.1 The test paint is applied by drawing it down on a striped black and white sealed chart, at a film thickness sufficient to obtain full hiding.

4.2 A portion of the drawdown is subjected to strong shearing forces in a prescribed manner.

4.3 The CIELAB color difference between the drawdown and sheared areas after drying, is reported as a measure of deficiency in the color development of the test paint.

## 5. Significance and Use

5.1 A colorant sometimes fails to disperse completely in a base paint due to poor compatibility, which can be the fault of the colorant, the paint, or both. This will result in poor color development, which is readily manifested by the common procedure of applying the paint with a doctor blade and subjecting the drawdown to high shear stress by finger-rubbing a small area of the partially dry film. This tends to disperse undeveloped colorant, if any, and produces a color variation between the unsheared and sheared areas of the paint film. The variation can be measured colorimetrically to give a numerical color difference value that is a measure of the color development of the original paint, the smaller the difference the better the color development and vice versa.

5.2 Poor color development can be a problem in the production of paints, and in their performance in the field. In production it causes a loss of colorant monetary value, and unpredictable tinting results. In field performance it results in color variations in the applied paint film due to the varying shear forces to which the paint is subjected at different stages or by different modes of application.

5.3 Although poor color development is primarily and most often related to the colorant portion of a tinted paint, the white pigment in the base paint can also be poorly developed due to flocculation or other causes. In the latter case, shear dispersion can make the paint film lighter and less colorful, rather than the reverse. Then too, the colorant and the white might both be poorly developed, and the color change due to shear stress would then be the combined effect of both.

5.4 In any case, color development is an important paint property, for the measurement of which this test method is intended to provide a generally accepted and reproducible test method.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

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

[3] Available from the Federation of Societies for Coatings and Technology, 494 Norristown Rd., Blue Bell, PA 19422.

## 6. Apparatus

6.1 *Film Applicator,* 6-in. (150-mm) wide with a clearance of 10 mils (250 µm).[4]

6.2 *Drawdown Plate*—A vacuum plate or thick plate glass are both satisfactory.

6.3 *Test Charts,* black and white striped, with a sealed surface, and overall size approximately 8½ by 11 in. (245 by 280 mm) having a test area of 0.538 ft$^2$ (500 cm$^2$).[5]

6.4 *Paint Brush,* 2-in. (50-mm) of good quality.[6]

6.5 *Syringe,* 20 mL, plastic disposable type.

6.6 *Force-Draft Oven,* maintained at 120 ± 2°F (49 ± 1°C).

6.7 *Reflectometer,* using CIE Illuminant C, specular reflection excluded, and capable of measuring CIELAB color coordinates as defined in Test Method D 2244 and Terminology E 284.[7]

## 7. Sampling and Conditioning

7.1 Sample the material in accordance with Practice D 3925.

7.2 Condition the samples in accordance with Specification D 3924.

## 8. Procedure

8.1 Stir the test paint thoroughly by hand, making sure that it is completely uniform with no trace of sediment or otherwise undispersed material. If necessary filter to remove persistent large agregates.

8.2 Clean the drawdown plate thoroughly and place the test chart on it with the applicator close to the top edge of the chart.

8.3 Fill the syringe to the 17-mL mark and from it distribute 10 mL of test coating evenly onto the chart in front of and close to the applicator, then draw down with a uniform motion.

8.4 Without delay cut an approximately 3-in. (75-mm) wide paint strip from the bottom end of the chart and set it aside.

8.5 Tape one edge of the remaining portion of the chart onto a flat support surface and distribute an additional 7 mL of test paint from the syringe evenly over the test area.

8.6 Dampen a clean paint brush, shake out the moisture vigorously, then dip the brush to ⅔ of its bristle length into the test paint in a ¾-filled pint container. Remove the brush, wipe the tip against the edge of the container to remove the surplus, then spread the paint evenly over the test area of the chart and allow to dry for 15 ± 1 min under ambient conditions.

8.7 During this drying period keep the wet paint brush wrapped in plastic wrap to prevent it from drying out.

8.8 After the specified drying period, brush over the painted area repeatedly for 2 min ± 10 s. Make sure that each brush stroke fully traverses the painted area and is at a uniform speed such that there will be 150 ± 5 strokes during the stated brushing period. Cover the entire area with six successive parallel strokes in alternate opposite directions followed by the same in the direction perpendicular to the preceding six, alternating in this way until the specified brushing time is completed. During each stroke press the brush down so that a third of the bristle length is pressed against the surface of the chart. Manipulate the brush so as to maintain a uniform spreading rate as much as possible during this operation. Complete the brushout by sweeping the tip of the brush over the painted area to smooth and eliminate brushmarks, and to completely obscure the black and white substrate pattern.

8.9 Place the completed brushout section along with the drawdown section of the chart in the force-draft oven for one h ± 2 min.

8.10 Remove the two sections from the oven and tape them together at the original cut.

8.11 Measure the CIELAB color difference $\Delta E^*_{ab}$ between the drawdown and the brushout. Avoid making measurements on show-through areas that might have occurred due to persistent deep brush marks or other deficiency in spreading rate uniformity. To obtain $\Delta E^*_{ab}$ determine CIELAB color coordinates $L^*a^*b^*$, and from them calculate $\Delta E^*_{ab}$ in accordance with Section 8 of Test Method D 2244. Modern color difference meters give $\Delta E^*_{ab}$ directly without need for calculations.

## 9. Report

9.1 Report the following information:

9.1.1 CIELAB Color difference $\Delta E^*_{ab}$, between drawdown and brushout,

9.1.2 A general description of the color and the type of paint being tested, and

9.1.3 Specific names and sources of commercial products being tested, paints or colorants or both.

## 10. Precision and Bias

10.1 *Precision—Experimental:* In an interlaboratory study[8] of this test method, one operator in each of six laboratories applied by brush and by bar applicator, six latex paints that varied widely in color and color development (Note 1). The interlaboratory pooled standard deviation, after discarding several outliers (see Note 2), was found to be 0.43 $\Delta E^*_{ab}$ with 21 df when five of the laboratories made their own color difference measurements. Based on this standard deviation the following criteria should be used for judging, at the 95 % confidence level the acceptability of results:

10.1.1 *Reproducibility*—Two single results obtained by

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[4] A Bird type film applicator, obtainable from most suppliers of paint test equipment, has been found satisfactory for this purpose. Other suitable applicators may be used.

[5] Leneta Form 8K-BW; obtainable from The Leneta Company; 15 Whitney Road; Mahwah, NJ 07430, was used in the round robins for this standard and found satisfactory. An equivalent may be used.

[6] The E-Z Paintr "ONE-V20-90 ONE COATER BRISTLENE" brush has been found satisfactory. Available from E-Z Paintr, 4051 S. Iowa Ave., Milwaukee, WI 53207. This is a chisel-tip polyester brush, 2¾ inch length-out, 9/16 inches thick. An equivalent may be used.

[7] Any reflectometer that measures the CIE tristimulus values of opaque surfaces will be found suitable. Types that provide a direct read-out of color difference $\Delta E^*_{ab}$ are available, and are particularly desirable for this test method. The following standards are applicable to this measurement, depending on instrumentation and other factors: Practice E 1164, Test Method E 1331, Practice E 1345, Test Methods E 1347, E 1349.

[8] Supporting data are available from ASTM Headquarters. Request RR: D01-1079.

operators in different laboratories should be considered suspect if they differ by more than 1.26 $\Delta E^*_{ab}$.

NOTE 1—In the interlaboratory study the test applications were air dried, but it was established that there is essentially perfect correlation between color difference measurements made on air-dried and force-dried panels.[8]

NOTE 2—Results from one laboratory for three of the paints and one result from another for one of the same paints were discarded because they differed significantly from other results for the same materials or because the laboratory mean differed significantly from means of all the other laboratories.

10.1.2 *Repeatability*—Insufficient data was obtained to directly establish the repeatability of this test, but it should be at least equal to the reproducibility stated in 10.2.

10.2 *Bias*—The concept of bias is inapplicable to this test method, because there are no independent authoritative values.

## 11. Keywords

11.1 color; color development; color uniformity; colorants; tinted paints

**Designation: D 5327 – 92**

# Standard Practice for Evaluating and Comparing Transfer Efficiency Under General Laboratory Conditions[1]

## 1. Scope

1.1 This practice covers the evaluation and comparison of the transfer efficiency of spray-applied coatings under general laboratory conditions. Transfer efficiency is the ratio of paint solids deposited to the total paint solids used during the application process, expressed as a percent. This practice can be used to study the effect on transfer efficiency of changing operating variables and paint formulations. Key variables that need to be controlled are listed in 8.13.

NOTE 1—It is important that all process or formulation parameters, except that which is intentionally being changed, be kept consistent from test to test. If not done, the results of the study are to be questioned.

1.2 The reproducibility of this practice is highly dependent on the degree of control of the parameters listed in Section 8 of the practice.

1.3 *Limitations*—This laboratory practice indicates only the direction of the effect of operating variables and liquid paint formulations on transfer efficiency under conditions of the laboratory test: the magnitude of the effect can be determined only with specific plant experience. In fact, the nature of the critical parameters that affect transfer efficiency make clear that it is not possible to extrapolate laboratory results.

NOTE 2—The laboratory practice outlined involves general laboratory spray equipment and procedures and is derived from Test Method D 5009. This practice and Test Method D 5009 are both derived from a study and report of transfer efficiency measurements conducted for the U.S. Environmental Protection Agency. For laboratories that have access to a conveyor and mass flow measurement equipment, a suitable, potentially more reproducible, tested method is defined in Test Method D 5009.

1.4 *This standard does not purport to address all of the safety problems, 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 7, Notes 8 and 9.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1200 Test Method for Viscosity by Ford Viscosity Cup[2]
D 2369 Test Method for Volatile Content of Coatings[2]
D 3925 Practice for Sampling Liquid Paints and Related Coatings[2]
D 5009 Test Method for Evaluating and Comparing Transfer Efficiency Under Laboratory Conditions[3]

2.2 *Other Standards:*

NFPA 33 Spray Application Using Flammable and Combustible Materials[4]
NFPA 86 Standard for Ovens and Furnaces[4]
*EPA 600/2-88-026a and EPA-600/2-88-026b, Development of Proposed Standard Test Method for Spray Painting Transfer Efficiency* Vols I and II[5]

## 3. Terminology

3.1 *Descriptions of Terms Specific to This Practice:*

3.1.1 *fluid mass flow rate*—the mass flow rate of paint in grams per minute during the test.

3.1.2 *mass of foil*—the weight of each target foil in grams before being painted.

3.1.3 *mass of foil plus paint solids*—the weight of each target foil in grams after being painted and baked.

3.1.4 *mass of paint solids*—the difference in the mass of the foil before painting and the mass of the foil after painting and baking. The sum of the mass of the foil plus paint solids less the sum of the mass of the foil.

3.1.5 *transfer efficiency*—the ratio of the mass of the paint solids deposited on the foil to the mass of the paint solids sprayed during the test, expressed as a percent.

3.1.6 *weight percent solids*—the solids content as percent of the total weight of a sample of the paint used during the test.

DISCUSSION—Weight percent solids determination as specified in 8.4.2.

## 4. Summary of Practice

4.1 Metal panels covered with preweighed aluminum foil are coated in a spray booth. The coated foils are baked to remove volatile matter. The transfer efficiency is calculated on a weight percent basis using the solids content, quantity of the paint sprayed, and the amount of solids on the coated aluminum foil.

## 5. Significance and Use

5.1 Subject to the limitations listed in 1.3, this practice can be used as a research tool to optimize spray equipment

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[1] This practice is under the jurisdiction of ASTM D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

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

[3] *Annual Book of ASTM Standards*, Vol 06.02.

[4] Available from National Fire Protection Association, Battery March Park, Quincy, MA 22269.

[5] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

and paint formulations as well as to study the relative effect on transfer efficiency of changing operating variables, spray application equipment, type of coatings, etc.

## 6. Apparatus

6.1 *Laboratory Scale*, accurate to ±0.001 g.

6.2 *Platform Scale*, accurate to ±0.01 g.

6.3 *Targets*, should consist of a minimum of three steel panels, two scavengers and a target panel. If more than one target panel is used, a scavenger panel is to be used at the start and end of the test panel set. The steel panels are 6 by 0.0625 in. wide (15.2 by 0.15875 cm) with 0.25 in. (0.635 cm) radius corners. The length of the panel should be sufficient that a minimum of 12 in. (30.4 cm) above and below the spray pattern is achieved.

NOTE 3—It is essential to effectively capture the entire height of the spray pattern.

NOTE 4—Other panel sizes similar to those in the end use can be used. Differences in the part shape will influence the transfer efficiency.

NOTE 5—Results of this test may not be extrapolated to different spray booths, part geometries, etc.

6.4 *Aluminum Foil*, medium temper or equivalent, 1.5-mil (0.0037-cm) thick. The aluminum foil should be preheated at the conditions specified in the cure schedule recommended by the paint manufacturer.

6.5 *Back-Draw Water-Wash Spray Booth*, or equivalent. The booth should be capable of developing and maintaining up to 120 ft/min (36.58 m/min) air velocity in the middle at the plane of the target. If a filter booth is used, filters should be changed as frequently as necessary to maintain uniform air velocity.

6.6 *Forced Draft Curing Oven*, if required, of sufficient size for curing targets, capable of achieving and maintaining the cure temperature specified by the paint supplier. All ovens should conform to NFPA 86.

6.7 *Curing Rack.*

6.8 *Stopwatch.*

6.9 *Air Velocity Measurement Equipment.*

6.10 *Humidity and Temperature Measurement Equipment.*

6.11 *Compressed Air Supply.*

6.12 *Spray Gun.*

NOTE 6—The spray gun can be attached to a spray machine or hand held. Needless to say, operator variation with a hand held spray gun can mask the effect of changes in the variable being studied.

## 7. Hazards

7.1 For specific hazard information and guidance, consult the supplier's Material Safety Data Sheet (MSDS) for the materials used.

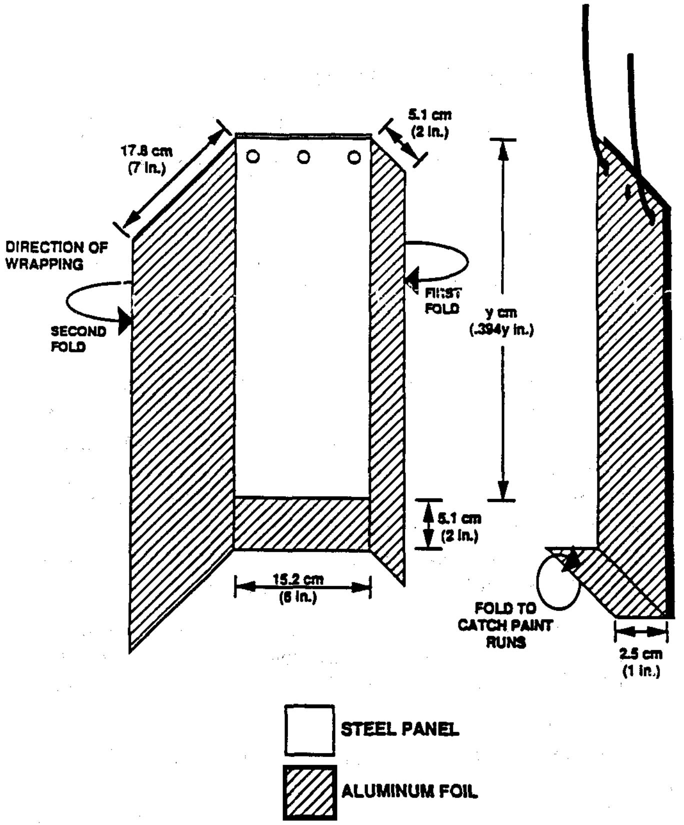
## 8. Procedure

8.1 Set up the paint supply equipment to the spray apparatus in accordance with the manufacturer's instructions.

8.1.1 Ground all electrically conductive objects in the spray area, except those objects required by the process to be at high voltage in accordance with Chapter 9.11 of NFPA 33.

8.2 Agitate the test paint in a closed container at least 30 min before paint samples are taken.

8.3 Using an airtight container, take a paint grab sample from the paint pot in accordance with Practice D 3925.



NOTE—Scale: Not To Scale. For *y* see 6.3 for dimensions.

**FIG. 1 Foil Attachment Technique**

8.4 Determine and record the following from the paint sample:

8.4.1 Paint viscosity in accordance with Test Method D 1200,

8.4.2 *Weight Percent Solids*—The preferred method is Test Method D 2369. If the baking temperature in Test Method D 2369 is considered inadequate for complete cure, use the manufacturer's recommended cure schedule. Make sure that the cure schedule used is agreed upon and recorded, and

8.4.3 Electrical resistivity for samples being applied electrostatically.

8.5 Cut the aluminum foil to dimensions of 15 by approximately 2 in. (58 by 5 cm) longer than the length of the target panel.

8.6 Number each precut foil strip, before weighing, using a permanent marking pen.

8.7 Weigh each test foil strip and record the uncoated weight and the foil number.

8.8 Attach the preweighed, labeled test foil to the targets using the technique shown in Fig. 1.

8.9 Mount the foil covered targets on a panel or target holder, with the foil seam on each target facing away from the spray gun. Set panel spacing as desired.

8.9.1 If electrostatic equipment is being used, the resistance shall be less than 1 by $10^6$ Ω between the target and earth ground in accordance with Chapter 9.8 of NFPA 33.

8.10 Adjust the following equipment operating parameters, as appropriate, to the values desired for testing:

8.10.1 Paint fluid pressure at spray gun, psi (kPa),

8.10.2 Atomizing air pressure at spray gun, psi (kPa),

8.10.3 Rotating atomizer head speed (revolutions per minute) with and without paint fluid flow,

8.10.4 Operating voltage if electrostatic equipment is used, kV,

8.10.5 Ambient air temperature (°C),

8.10.6 Paint fluid temperature (°C),

8.10.7 Booth air velocity, feet per minute (meters per minute),

8.10.8 Relative humidity (percent),

8.10.9 Spray machine variables or spray gun procedures when using a hand held spray gun,

NOTE 7—The spray gun can be attached to a spray machine or hand held. If hand held, variations in technique can effect reproducibility of the transfer efficiency study and may override comparisons made.

8.10.10 Set the following variables where applicable (see Note 8):

8.10.10.1 Spray gun to target distance, in. (cm),

8.10.10.2 Travel speed, inches per minute (centimetres per minute),

8.10.10.3 Number of coats,

8.10.10.4 Number of passes per coat, and

8.10.10.5 Flash between coats, seconds.

NOTE 8: **Precaution**—If electrostatic equipment is being used, the gun-to-target distance shall be at least twice the sparking distance in accordance with Chapter 9.7 NFPA 33, where applicable.

8.10.11 Set the cure time and temperature in accordance with the manufacturer's instructions.

8.11 For electrostatic spray equipment, measure the operating voltage and adjust it according to the manufacturer's instructions.

8.12 Turn on the spray booth. Turn on paint spray equipment. Maintain uniform paint flow during the test. Turn off spray equipment.

8.13 Weigh the paint before and after the test.

8.13.1 If the weight difference measurement approach is not appropriate, use the following technique: just after turning on the spray equipment, spray the unatomized paint into a preweighed plastic container for a minimum of 30 s.

NOTE 9: **Precaution**—Turn off all high voltage to electrostatic spray guns to prevent personal injury.

8.13.2 Immediately weigh the container with paint, calculate the flow rate, and record the result. Before turning off the spray machine, repeat this procedure. Average the two results to obtain the average flow rate for the test.

NOTE 10—The use of a gallon plastic bottle with the top cut off to conveniently fit around the front of the gun is recommended. A large plastic beaker covered with plastic wrap with a hole punched in the center of the plastic wrap may also be used. Turn off all air sources to the spray gun before using this procedure to prevent paint splatter out of the container.

8.13 Record the following data:

8.13.1 *Application Equipment:*

8.13.1.1 Paint fluid pressure at the spray gun, psi (kPa),

8.13.1.2 Atomizing air pressure at spray gun, psi (kPa),

8.13.1.3 Rotating atomizer head speed (revolutions per minute) with and without paint fluid flow,

8.13.1.4 Spray machine settings,

8.13.1.5 Operating voltage at the electrode tip if electrostatic equipment is used, kV,

NOTE 11—If a hand held spray gun is used, record the gun-to-panel distance, number of coats, and the flash time between coats.

8.13.1.6 Panel spacing, and

8.13.1.7 Spray time.

8.13.2 *Spray Booth Variables:*

8.13.2.1 Ambient temperature (°C),

8.13.2.2 Paint fluid temperature (°C),

8.13.2.3 Booth air velocity, feet per minute (meters per minute),

NOTE 12—Variations in booth air velocity can affect transfer efficiency results.

8.13.2.4 Relative humidity (percent), and

8.13.2.5 Mass of paint used for painting the targets, grams (pounds).

NOTE 13—The mass of paint used may be determined by measuring the difference in weight of the paint before and after the sprayout or by multiplying the paint flow rate determined in 8.13.2 by the length of time that the spray equipment is on.

8.13.3 Operator name and date.

8.14 After the paint flow is topped, carefully remove the painted targets from the panel holder to ensure that no paint is lost.

8.15 For products that are baked, hang the coated targets on oven racks so all painted surfaces are exposed for uniform drying. Flash per the manufacturer's recommendations, insert the racks into the oven and bake using the manufacturer's recommended cure schedule. For products that are not baked, follow the manufacturer's recommended cure schedule.

8.16 Where appropriate, remove the targets from the oven and let cool.

8.17 Remove the foil from each target, weigh and record the coated weight, the foil number, the dry film thickness at the center of the spray pattern, and the percent vertical film coverage. If the desired dry film thickness and the percent vertical film coverage is not achieved, repeat the procedure listed in 8.1 to 8.16.

8.18 The mass of the paint solids deposited is the difference between the weight of the foil after painting and baking and the total weight of the foil before painting.

## 9. Calculation

9.1 Calculate the transfer efficiency using the following equation:

$$T = 100(P / W \times A)$$

where:

$T$ = transfer efficiency, %

$P$ = mass of paint solids deposited, as specified in 8.18,

$W$ = weight percent solids expressed as a decimal as specified in 8.4.2, and

$A$ = mass of paint applied, as specified in 8.13, 8.13.1, or 8.13.2.

## 10. Report

10.1 Report the following information:

10.1.1 Transfer efficiency results,

10.1.2 Type of spray equipment,

10.1.3 Type of paint applied,

10.1.4 Paint application conditions as listed in 8.13, and

10.1.5 Conditions of test other than those specified in the procedure section of this practice.

## 11. Keywords

11.1 general laboratory method; laboratory practice; spray-applied coatings; transfer efficiency

# Standard Test Method for Volatile Organic Compound (VOC40) Content of Non-Heatset Paste Printing Ink Systems at 40°C[1]

## 1. Scope

1.1 This test method covers the determination of the volatile organic compound (VOC40) content of non-heatset printing ink systems of the paste type using a baking cycle of 1 h at 40°C.

1.2 This test method is applicable to paste printing inks and vehicles that dry primarily by absorption, polymerization, or related means without the application of heat. Such systems do not contain appreciable quantities of low-boiling solvents that evaporate readily at ordinary room temperatures.

NOTE 1—The 40°C baking temperature in this test method is the same as that specified in Bay Area Method 30. The temperature of heating is 110°C in several related test methods, for example, D 2369, D 4713 and EPA Reference Method 24. VOC determinations are reviewed in ASTM Manual Series MNL 4.[2]

1.3 *This standard does not purport to address all of the safety problems, 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 1475 Test Methods for Density of Paint, Varnish, Lacquer and Related Products[3]

D 2369 Test Methods for Volatile Content of Coatings[3]

D 3792 Test Method for Water Content of Water-Reducible Paints by Direct Injection into a Gas Chromatograph[3]

D 3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings[3]

D 4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method[3]

D 4713 Test Methods for Nonvolatile Content of Heatset and Liquid Printing Ink Systems[4]

E 1 Specification for ASTM Thermometers[5]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of Test Methods[6]

2.2 *Other Standards:*

EPA Federal Reference Method 24—Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids of Surface Coatings[7]

Method 30—Determination of Volatile Organic Compounds (VOC) in Solvent-Based Non-Heatset Inks[8]

## 3. Terminology

3.1 *Definitions*—Definitions pertaining to this test method are covered in Practice D 3960.

## 4. Summary of Test Method

4.1 The test material is spread in a weighing dish to a nominal thickness corresponding to about 115 g/m$^2$. The weight percent of total volatile compounds is determined after heating at 40°C for one h.

4.2 The typical paste ink does not contain water or exempt solvents; in such cases, the volatile organic content is the same as the total volatile content. If the test sample is based on an emulsion system or is suspected of containing more than 5 % water, the water content is determined by the Karl Fischer method or by gas chromatography.

4.3 The density of the test sample is measured, and the volatile organic content in weight percent at 40°C is converted to units of grams per litre or pounds per gallon. Alternatively, if the water content was found to be greater than 5 %, the volatile organic content is calculated in accordance with Note 3.

## 5. Significance and Use

5.1 VOC content data are required by various regulatory agencies. This test method determines the volatile organic content of non-heatset paste printing ink systems at 40°C in accordance with Bay Area Method 30. This temperature is considered more realistic in terms of end use than the 110°C used in several similar test methods. Due to the complex chemical composition and boiling characteristics of the oils used in these inks, temperature of 110°C may cause volatile loss that would not occur at ambient temperatures, even over extended periods of time.

5.2 The inverse value, nonvolatile, is used to determine the weight percent solids content.

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[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved Nov. 15, 1992. Published January 1993.

[2] Brezinski, J. J., *Manual on Determination of Volatile Organic Compounds in Paints, Inks, and Related Coatings, ASTM Manual Series MNL4*, ASTM, 1989.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.02.

[5] *Annual Book of ASTM Standards*, Vol 14.03.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[8] Available from Bay Area Quality Measurement District (BAAQMD), 939 Ellis Street, San Francisco, CA 94109.

5.3 The specimen film thickness must be controlled in order to obtain reproducible test results within the specified 1-h heating time.

## 6. Apparatus

6.1 *Balance*, accurate to 1 mg.

6.2 *Oven*, forced-ventilation type conforming to Type IIB in Specification E 145, properly leveled and maintained at 40° ± 1°C (104 ± 2°F). If air flow is adjustable, set control dampers, at 50 %.

6.3 *Thermometer*, bulb-type, capable of reading to ±1°C, such as Thermometer 88C prescribed in Specification E 1.

6.4 *Weighing Dishes*, such as aluminum foil dishes 57 or 70-mm wide, the lid of 1-lb ink can 94-mm wide, or other flat-bottomed containers. The bottom of the containers must not have a trough or depression into which the test material might collect. Dishes should be rinsed with toluene or other appropriate solvent, preconditioned for 30 min in an oven at 110 ± 5°C, and stored in a desiccator prior to use.

6.5 *Spatula*, or small ink knife.

6.6 *Spreading Device*, one per weighing dish, of heat-stable material, such as a glass stirring rod or thick L-shaped wire.

6.7 *Forceps.*

6.8 *Desiccator.*

6.9 *Water Content Measuring Apparatus*, such as a Karl Fischer apparatus conforming to Test Method D 4017 or a gas chromatograph conforming to Test Method D 3792.

6.10 *Weight-Per-Gallon Cup*, 20 to 100-mL capacity calibrated in accordance with Test Methods D 1475.

## 7. Reagents

7.1 *Toluene*, technical grade, or other appropriate solvent.

## 8. Preparations and Sample

8.1 Wear disposable gloves prior to handling weighing dish, spreading device, or weight-per-gallon cup in order to minimize contamination by moisture from hands.

8.2 Measure diameter of bottom of weighing dish in millimetres. The bottom diameter must be used in the calculations of weight per unit area in 13.1.

8.3 Prior to removing a specimen, carefully remove surface skin, if any, and thoroughly mix the sample in its container to ensure uniformity. Close can and reseal when finished.

## 9. Procedure for Volatile Content

9.1 Set the oven for 40° ± 1°C.

9.2 For each sample, tare to the nearest milligram two preconditioned weighing dishes each with a spreading device. Retain spreading device in the dish throughout the test.

9.3 Transfer a representative portion (see 8.3) of the sample to the tip of a spatula and dab an appropriate quantity around the bottom of the dish as follows:

| | |
|---|---|
| 57-mm dish | 0.20–0.25 g |
| 70-mm dish | 0.40–0.50 g |
| 94-mm dish | 0.70–0.90 g |

Quickly reweigh and calculate the weight per area following 12.1. *If not between 100 and 130* g/m², adjust the quantity of the specimen.

9.4 With the spreader, smooth out the specimen into a reasonably uniform film covering the entire bottom of the dish. If necessary, add a few drops of toluene or other appropriate solvent to aid in spreading out the film.

9.5 Place the dishes in the forced-draft oven at 40°C for exactly 1 h. Remove dishes from oven, cool in desiccator, and reweigh.

## 10. Procedure for Water Content and Exempt Solvents

10.1 Paste inks are not water-reducible. If the sample is an emulsion or is believed to contain more than 5 % water, measure the water content in one of the following ways:

10.1.1 *Test Method D 4017*—Dilute the specimen with pyridine, add a catalyst (1-ethylpiperidine), titrate to the end point in the Karl Fischer apparatus, and compute the water content.

10.1.2 *Test Method D 3792*—Dilute the specimen with dimethyl formamide, add an internal standard (2-propanol), inject an aliquot directly into the gas chromatograph, and compute the water content from appropriate area calculations on the chromatogram.

10.2 Exempt solvents are generally too volatile to be used in paste ink systems. Therefore, there is no need to test for their presence. (A list of exempt solvents is covered in Practice D 3960.)

## 11. Procedure for Density

11.1 Determine the density of the sample at 25°C using a calibrated weight-per-gallon cup in accordance with Test Method D 1475.

11.2 Transfer the thoroughly mixed sample to the weighed cup in a manner so as to avoid occluding air bubbles. Fill to the top and cap, leaving the overflow orifice open. Immediately remove excess sample using an absorbent material. Quickly reweigh the filled cup.

11.3 Clean the cup and repeat 11.2.

## 12. Calculation

12.1 Calculate initial weight/area $S/A$ in grams per square metre of each specimen as follows:

$$S/A = (S \times 10^6)/3.14\ R^2 \qquad (1)$$

where:

$S$ = initial specimen weight, g,

$R$ = radius of the dish bottom, = diameter/2, mm.

Record to the nearest whole number.

NOTE 2—Weight per area equals 510 $S$ for a 57-mm dish with a 50-mm diameter bottom, 260 $S$ for a dish with a 70-mm diameter bottom, and 145 $S$ for a dish with a 94-mm diameter bottom.

12.2 Calculate the weight percent nonvolatile content at 40°C $NVC40$ as follows:

$$NVC40 = (W_1/S) \times 100 \qquad (2)$$

where:

$W_1$ = weight of the specimen after heating at 40°C, g and

$S$ = initial specimen weight, g.

Record to the nearest 0.1 %.

12.3 Calculate the weight percent volatile organic content $VOC40$ as follows:

$$VOC40 = 100 - NVC40 \qquad (3)$$

12.4 Compute the density of the sample, $D_m$ in grams per millilitre as follows:

$$D_m = W_2/V \tag{4}$$

where:

$W_2$ = specimen weight in the weight-per-gallon cup, g, and

$V$ = volume of the cup, mL.

Record to three decimal places.

12.5 Compute the *VOC*40 content in grams per litre or pounds per gallon as follows:

12.5.1 Grams per litre:

$$VOC40 = D_m\ VOC40 \times 10 \tag{5}$$

12.5.2 Pounds per gallon:

$$VOC40 = 8.3454\ D_m\ VOC40 \times 10^{-2} \tag{6}$$

where:

8.3454 = factor converting g/mL to lb/gal (see Test Method D 1475).

Record to two decimal places.

NOTE 3—If the sample contains more than 5 % water, compute the $VOC40_{nw}$ in grams per litre as follows:

$$VOC40_{nw} = \frac{D_m\,(VOC40 - W_w)}{100 - (W_w)(D_m/D_w)} \times 1000 \tag{7}$$

where:

$VOC40_{nw}$ = *VOC*40, ink minus water, g/L,

$VOC40$ = (100 − *NVC*), weight %,

$W_w$ = water content, weight %, and

$D_w$ = density of water, at 25°C (0.997), g/L.

Multiply $VOC40_{nw}$ in grams per litre by 8.3454 to obtain $VOC40_{nw}$ in pounds per gallon.

## 13. Report

13.1 Report the following:

13.1.1 The weight/area of the sample,

13.1.2 The temperature and time of heating,

13.1.3 The total nonvolatile content at 40°C (*NVC*40) in weight percent as the mean of two determinations,

13.1.4 The volatile organic content at 40°C (*VOC*40) in weight percent,

13.1.5 The density of the sample in g/mL as the mean of two determinations, and

13.1.6 The *VOC*40 content in g/L or lb/gal.

NOTE 4—For those samples that contain more than 5 % water, report the water content in weight percent as the mean of two determinations and the volatile organic content at 40°C in accordance with Eq 5 of Practice D 3960.

## 14. Precision and Bias

14.1 *Precision of VOC40 in Weight Percent*[9]—An interlaboratory study of this test method was conducted in which operators in 12 laboratories tested the weight percent volatile content in duplicate on each of two days of four non-heatset wet-offset black inks whose *VOC*40 ranged from 0.5 to 8 % by weight (92 to 99.5 % expressed as nonvolatile content). Since the inks did not contain water, the volatile organic content was the same as the total volatile content. The test results were analyzed in accordance with Practice E 691. Two laboratories did not conform to the prescribed film thickness range and were deleted from the analysis, as was another laboratory because of a faulty oven. The ink with the 0.5 % by weight *VOC*40 showed more variability (less when expressed as *NVC*40) than the other three inks and was also deleted from the analysis. For the inks with a *VOC*40 content between 1 and 8 % by weight, the within-laboratory pooled standard deviation was 0.53, and the between-laboratories pooled standard deviation was 0.90. Based on statistical analysis of the results, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1 *Repeatability*—Two *VOC*40 weight percent results, each the mean of two determinations obtained by the same operator on different days, should be considered suspect if they differ by more than 36 % relative (1.4 % relative if expressed as *NVC*40).

14.1.2 *Reproducibility*—Two *VOC*40 weight percent results, each the mean of determinations obtained on different days by operators in different laboratories, should be considered suspect if they differ by more than 60 % relative (2.3 % relative if expressed as *NVC*40).

14.2 *Precision of VOC40 in g/L or lb/gal*—A study has been initiated to determine the precision of weight-per-gallon density measurements on paste inks. If less than that given in Test Method D 1475, a new round robin will be conducted to determine the precision of *VOC*40 in g/L or lb/gal.

14.3 *Bias*—Bias cannot be determined as there are no standard materials.

## 15. Keywords

15.1 non-heatset paste printing inks; non-heatset paste printing ink vehicles; nonvolatile content; printing inks; vehicles; volatile organic compound (VOC40) content at 40°C

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[9] Supporting data are available from ASTM Headquarters. Request RR:D01-1078.

Designation: G 6 – 88 (Reapproved 1992)$^{\epsilon 1}$

# Standard Test Method for Abrasion Resistance of Pipeline Coatings[1]

$^{\epsilon 1}$ NOTE—Section 13 was added editorially in September 1992.

## 1. Scope

1.1 This test method of accelerated test is a procedure for determining the relative resistance of steel pipeline coatings to abrasion by a slurry of coarse abrasive and water. The method is intended to apply to the testing of all types of electrical insulating pipeline coatings and tapes, including thermoplastics, thermoset, and bituminous materials.

1.2 Pipeline coatings are not normally subjected to the type of abrasion herein specified.

1.3 Metallic protective coatings such as zinc may be compared visually, but do not meet the electrical requirements of this test method.

1.4 The values stated in SI units to three significant decimals are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, 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:*

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

G 62 Test Methods for Holiday Detection in Pipeline Coatings[2]

## 3. Terminology

3.1 *Definition:*

3.1.1 *holidays*—small faults or pinholes which permit current drainage through protective coatings on steel pipe.

## 4. Summary of Test Method

4.1 Apparatus and materials are described whereby coatings on steel pipe are worn away by an abrasive slurry contained in a horizontally revolving drum. Specimens extend through both ends of the drum and are electrically insulated from contact with the test apparatus.

4.2 Periodic measurement of the electrical resistance between the specimen and the drum indicates the degree of abrasion resistance of the coating.

## 5. Significance and Use

5.1 This procedure defines a test method for comparing the relative resistance of pipeline coatings to abrasion.

5.2 Abrasion resistance may be used to specify optimum coating thickness of candidate materials both in development and research work to study new coating systems or methods and in quality control.

## 6. Apparatus

6.1 *Drum*—A cylindrical steel container suitable for testing nine specimens simultaneously. Figures 1 to 3 show construction details which may be altered at the cap assembly if desired. A valve shall be provided to release any pressure built up during test.

6.2 *Apparatus for Revolving the Drum*—Any suitable apparatus used to revolve the drum at 30.48 m (100 linear ft)/min. A modified jar rolling mill[3] has been found satisfactory for revolving the drum.

6.3 *Thickness Gage,* to be used in accordance with Test Method G 12.

6.4 *Holiday Detectors,* low-voltage wet-sponge type for thin film coatings (coatings thinner than 20 mils), and high-voltage type for thick film coatings (coatings thicker than 20 mils).

NOTE—Low voltage detectors are used to locate pinholes, voids, or thin spots in pipeline coatings. The potential used with wet-sponge detectors can be up to 100 V DC. Procedures for using these detectors are found in Method A of G 62. For use of high voltage detectors operating in the 900 to 20 000 Vdc range, see Method B of G 62.

6.5 *Volt-ohm-meter*—Any electrical test instrument for measuring electrical resistance of the circuit.

## 7. Reagents and Materials

7.1 The test slurry shall consist of the following:

7.1.1 *Aluminum Oxide Grit,*[4] 13.6 kg (30 lb).

7.1.2 *Tap Water,* 5.68 L (1½ gal).

## 8. Test Specimen

8.1 A 19.1 mm test specimen 406 mm (16 in.) long shall be prepared with its surface preparation and coating proce-

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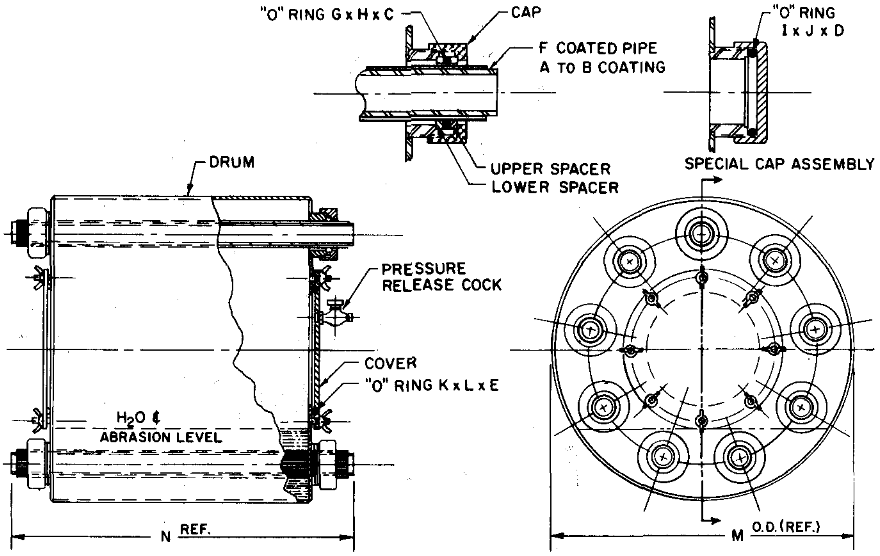
[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 27, 1988. Published July 1988. Originally published as G 6 – 69 T. Last previous edition G 6 – 83.

[2] *Annual Book of ASTM Standards,* Vol 06.02.

[3] Figure 801-V Jar Mill (U.S. Stoneware, Inc., Akron, OH) provides a suitable means for revolving the drum.

[4] Exolon aluminum oxide abrasive, No. 3 mesh, Type TA, untumbled, available from Exolon Co., Tonawanda, NY 14150, has been found satisfactory for this purpose.



Dimensions for Fig. 1:

| Symbol | mm | in. |
|---|---|---|
| A | 0.15 | 0.006 |
| B | 2.28 | 0.090 |
| C | 3.17 | 0.125 |
| D | 4.76 | 0.188 |
| E | 6.35 | 0.250 |
| F | 19.05 | 0.750 |
| G | 25.40 | 1.000 |
| H | 31.75 | 1.125 |
| I | 38.10 | 1.500 |
| J | 47.63 | 1.875 |
| K | 136.53 | 5.375 |
| L | 149.23 | 5.875 |
| M | 355.6 | 14.0 |
| N | 406.4 | 16.0 |

NOTE—Estimated weight includes water and abrasive 39.5 kg (87 lb)

**FIG. 1 Abrasion-Tester Assembly**

dures equivalent to that of production coated pipe. Only holiday-free specimens, in duplicate, shall be used in this test.

8.2 Control specimens shall be included in each test, the selection of which is optional with the user.

## 9. Conditioning

9.1 The specimen shall be exposed to a room temperature of 21° to 25°C (70° to 77°F) for a period of 24 h before beginning the test.

## 10. Procedure

10.1 Perform the test at a room temperature of 21° to 25°C (70° to 77°F).

10.2 Measure and record the coating thickness of each specimen in accordance with Test Method G 12.

10.3 Check all specimens or holidays using an appropriate detector.

10.4 Position the specimens in the drum taking care not to damage the coating.

10.5 Load the apparatus with 13.6 kg (30 lb) of unused abrasive and 5.68 L (1½ gal) of water.

10.6 Test each specimen for electrical resistance after sealing the drum by using the volt-ohm-meter. Immerse the specimen in the slurry at the time the electrical resistance is measured. Reject and replace any specimen showing less than infinite resistance.

10.7 Revolve the drum at the rate of 30.5 m (100 linear ft)/min.

10.8 Determine the electrical resistance at 25-h intervals for a test period of 200 h.

10.9 Relieve the pressure built up during the test by opening the release valve each time the electrical resistance is measured.

## 11. Report

11.1 The report shall include the following:

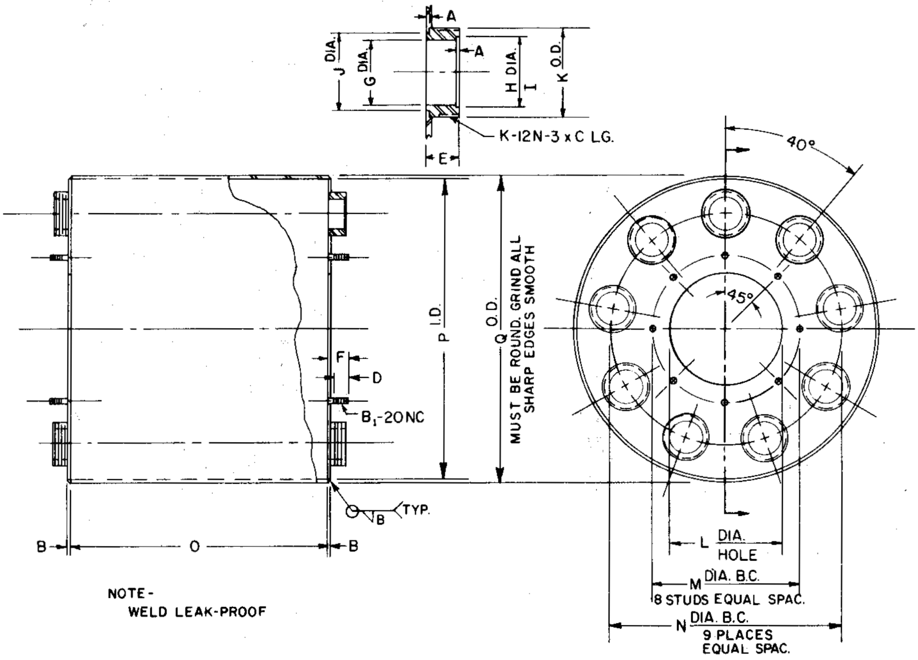
11.1.1 Complete identification of test specimens, including name and code number of coating, size of the pipe, production date, production run number, and any other information that may be pertinent to identification,

11.1.2 Date of starting and of terminating the test,

11.1.3 Coating thickness of the test specimen before testing and minimum coating thickness after testing,

11.1.4 Volt-ohm-meter readings at 25-h intervals, and

11.1.5 Description of coating appearance after testing.



Dimensions for Fig. 2:

| Symbol | mm | in. |
|---|---|---|
| A | 1.59 | 0.063 |
| B | 3.18 | 0.125 |
| $B_1$ | 6.35 | 0.250 |
| C | 12.70 | 0.500 |
| D | 19.05 | 0.750 |
| E | 22.23 | 0.875 |
| F | 25.40 | 1.000 |
| G | 38.10 | 1.500 |
| H | 41.25 | 1.624 |
| I | 41.28 | 1.625 |
| J | 44.45 | 1.750 |
| K | 50.8 | 2.0 |
| L | 127.0 | 5.0 |
| M | 165.1 | 6.5 |
| N | 266.7 | 10.5 |
| O | 304.8 | 12.0 |
| P | 349.25 | 13.75 |
| Q | 355.6 | 14.0 |

FIG. 2 Drum

## 12. Precision

12.1 Precision data are limited to two adjacent specimens taken from the same production-coated pipe, assuming that the production process was uniform with respect to pipe surface condition and coating material. Specimens that were not adjacent in the as-produced condition, or were taken from different lengths of pipe may represent differing process conditions.

12.2 *Repeatability*—When more than one test of the same coating material is involved, the standard deviation of the results should not be greater than 1.0 times the hourly time interval between inspections for the specific coating material. The standard deviation[5] of a set of numbers is the square root of the average of the squares of the deviations of the numbers from their average. The standard deviation of two or more specimen results may be found by the following procedure:

$$\text{Standard deviation, } \sigma = 1/n[(n\Sigma X^2 - (\Sigma X)^2)^{1/2}]$$

where:

$\Sigma X$ = sum of all values of $X$, from $X_1$ to $X_n$ inclusive,
$n$ = number of observed values (observations), and
$\sigma$ = standard deviation, the root-mean-square (rms) deviation of the observed values from their average.

12.3 *Reproducibility*—Agreement between laboratories should be such that the standard deviation of the results for a specific coating should not be greater than 1.5 times the inspection time interval.

## 13. Keywords

13.1 abrasion; coatings; pipeline; revolving; slurry

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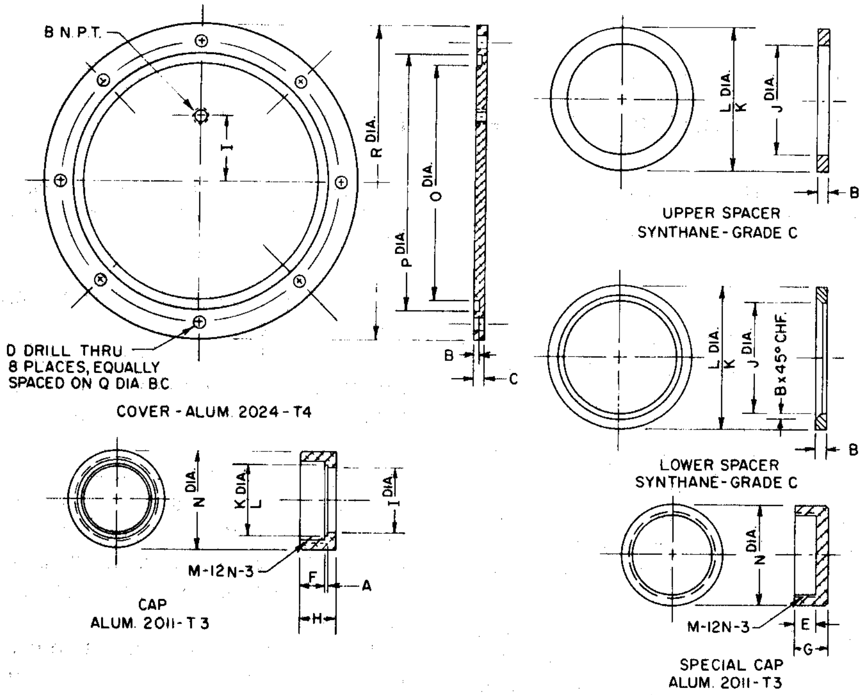
[5] For more information see the *ASTM Manual on Presentation of Data and Control Chart Analysis, STP 15D,* October 1976.



Dimensions for Fig. 3:

| Symbol | mm | in. |
|---|---|---|
| A | 1.59 | 0.063 |
| B | 3.18 | 0.125 |
| C | 6.35 | 0.250 |
| D | 7.94 | 0.313 |
| E | 12.70 | 0.500 |
| F | 15.88 | 0.625 |
| G | 19.05 | 0.750 |
| H | 22.23 | 0.875 |
| I | 38.10 | 1.500 |
| J | 25.68 | 1.281 |
| K | 41.25 | 1.624 |
| L | 41.28 | 1.625 |
| M | 50.80 | 2.0 |
| N | 51.05 | 2.250 |
| O | 136.53 | 5.375 |
| P | 149.23 | 5.875 |
| Q | 165.10 | 6.500 |
| R | 184.15 | 7.250 |

**FIG. 3 Details**

Designation: G 8 – 90

# Standard Test Methods for Cathodic Disbonding of Pipeline Coatings[1]

This standard is issued under the fixed designation G 8; 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 ($\epsilon$) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover accelerated procedures for simultaneously determining comparative characteristics of insulating coating systems applied to steel pipe exterior for the purpose of preventing or mitigating corrosion that may occur in underground service where the pipe will be in contact with inland soils and may or may not receive cathodic protection. They are intended for use with samples of coated pipe taken from commercial production and are applicable to such samples when the coating is characterized by function as an electrical barrier.

1.2 This test method is intended for testing coatings submerged or immersed in the test solution at room temperature. When it is impractical to submerge or immerse the test specimen, Test Method G 95 may be considered where the test cell is cemented to the surface of the coated pipe specimen. If higher temperatures are required, see Test Method G 42. If a specific test method is required with no options, see Test Method G 80.

1.3 The values stated in SI units to 3 significant decimals are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

G 42 Method for Cathodic Disbonding of Pipeline Coatings Subjected to Elevated Temperatures[2]

G 80 Test Method for Specific Cathodic Disbonding of Pipeline Coatings[2]

G 95 Test Method for Specific Cathodic Disbondment Test of Pipeline Coatings (Attached Cell Method)[2]

## 3. Summary of Test Methods

3.1 Both of the two test methods described subject the coating on the test specimen to electrical stress in a highly conductive, alkaline electrolyte. Electrical stress is obtained either by means of a sacrificial magnesium anode or from an impressed current system. The coating is perforated before starting the test.

3.1.1 In Method A, a magnesium anode is used with no electrical monitoring during the test period. The results are determined by physical examination after the test period is concluded.

3.1.2 In Method B, either a magnesium anode or an impressed current system may be used. Electrical instrumentation is provided for measuring the current in the cell circuit. The electrical potential is also measured, and upon conclusion of the test period, the test specimen is physically examined.

3.1.3 In both test methods physical examination is conducted by comparing the extent of loosened or disbonded coating at the perforations in the immersed area with extent of loosened or disbonded coating at a new test hole in the coating made in an area that was not immersed.

## 4. Significance and Use

4.1 Breaks or holidays in pipe coatings may expose the pipe to possible corrosion, since after a pipe has been installed underground, the surrounding earth will be more or less moisture-bearing and it constitutes an effective electrolyte. Damage to pipe coating is almost unavoidable during transportation and construction. Normal soil potentials as well as applied cathodic protection potentials may cause loosening of the coating, beginning at holiday edges, in some cases increasing the apparent size of the holiday. Holidays may also be caused by such potentials. While apparently loosened coating and cathodic holidays may not result in corrosion, this test provides accelerated conditions for loosening to occur and therefore gives a measure of resistance of coatings to this type of action.

4.2 The effects of the test may be evaluated by either physical examination or monitoring the current drawn by the test specimen or both of these two. Usually there is no correlation between the two methods of evaluation but both methods are significant. Traditional physical examination consists of assessing the effective contact of the coating with the metal surface in terms of observed differences in the relative adhesive bond. It is usually found that the electrically stressed area propagates from the holiday to a boundary where the loosened coating leaves off for the more effective contact or bond attributed to an original condition throughout the specimen before electrical stressing was applied. Assumptions associated with test results include the following:

4.2.1 Attempting to loosen or disbond the coating at a new test hole made in the coating in an area that was not immersed represents maximum adhesion or bond as measured by the lifting technique used, and that the same lifting

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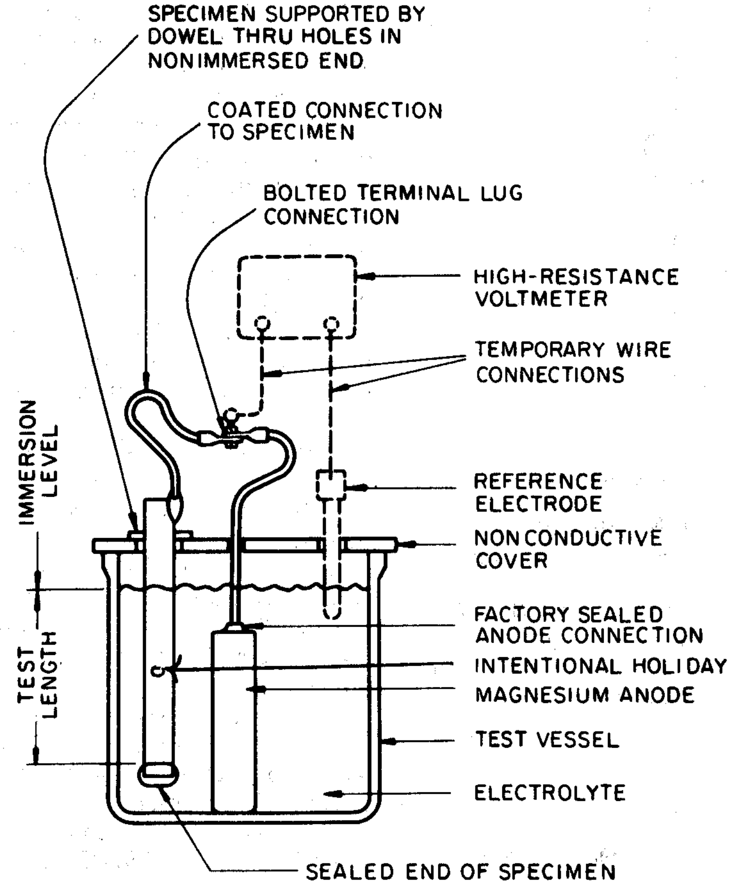
[1] These test methods are under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and are the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 25, 1990. Published July 1990. Originally published as G 8 – 69 T. Last previous edition G 8 – 85.

[2] *Annual Book of ASTM Standards*, Vol 06.02.



NOTE—Test hole made in non-immersed area after testing not shown (see Fig. 5).

FIG. 1 Test Assembly for Method A Using a Magnesium Anode

technique can be used at a test hole that was immersed thereby providing a means of comparing relative resistance to lifting.

4.2.2 Any relatively lesser bonded area at the immersed test holes in the coating was caused by electrical stressing and was not attributable to an anomaly in the application process. Ability to resist disbondment is a desired quality on a comparative basis, but disbondment per se in this test is not necessarily an adverse indication. The virtue of this test is that all dielectric type coatings now in common use will disbond to some degree thus providing a means of comparing one coating with another. Bond strength is more important for proper functioning of some coatings than others and the same measured disbondment for two different coating systems may not represent equivalent loss of corrosion protection.

4.2.3 The amount of current in the test cell is a relative indicator of the extent of areas requiring protection against corrosion; however, the current density appearing in this test is much greater than that usually required for cathodic protection in natural, inland soil environments.

## 5. Apparatus

5.1 *Apparatus for Both Methods:*

5.1.1 *Test Vessel*—A nonconducting material shall be used for the vessel or as a lining in a metallic vessel. Dimensions of the vessel shall permit the following requirements:

5.1.1.1 Test specimens shall be suspended vertically in the vessel with at least 25.4-mm (1-in.) clearance from the bottom.

5.1.1.2 Each test specimen shall be separated from the other specimens, from the anodes and from the walls of the test vessel by at least 38.1 mm (1.500 in.).

5.1.1.3 Depth of electrolyte shall permit the test length of the specimen to be immersed as required in 7.4.

5.1.1.4 If electrical monitoring is to be performed as required in Method B, the reference electrode may be placed anywhere in the vessel, provided it is separated from the specimen and from the anode by not less than 38.1 mm (1.500 in.).

5.1.2 *Magnesium Anode*—The anode shall be made of a magnesium alloy having a solution potential of −1.45 to −1.55 V with respect to a $CuCuSO_4$ reference electrode in the electrolyte given in 6.1. It shall have a surface area not less than one third that of the total specimen area exposed to electrolyte (outside area exposed only). The anode shall be provided with a factory-sealed, 4107-cmil (14-gage Awg), minimum, insulated copper wire. Anodes without a factory seal may be used if the magnesium extends above the cover.

5.1.3 *Connectors*—Wiring from anode to test specimen shall be 4107-cmil (14-gage Awg), minimum, insulated copper. Attachment to the test specimen shall be by sol-

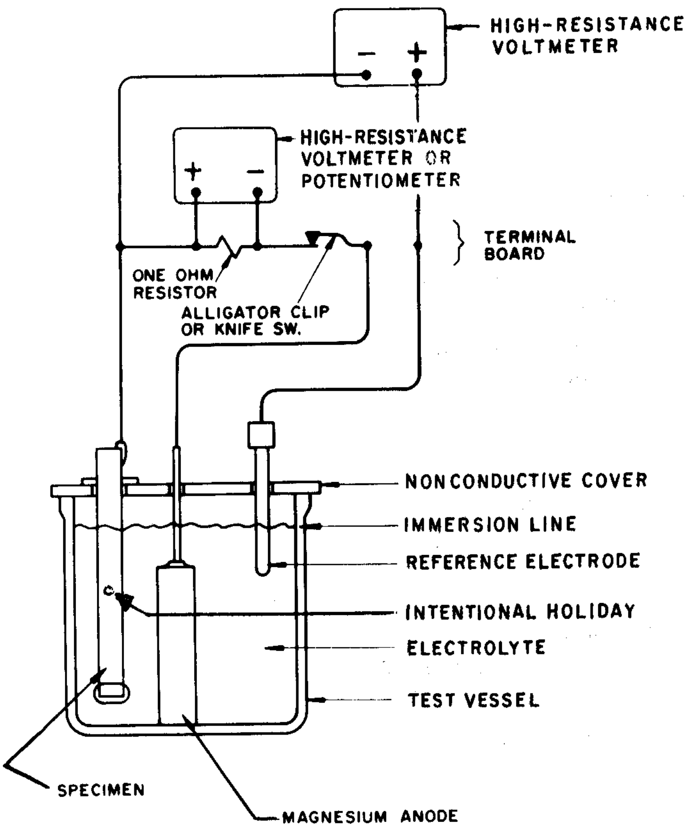


**FIG. 2 Test Assembly for Method B Using a Magnesium Anode**

dering, brazing, or bolting to the nonimmersed end, and the place of attachment shall be coated with an insulating material. A junction in the connecting wire is permitted, provided that it is made by means of a bolted pair of terminal lugs soldered or mechanically crimped to clean wire ends.

5.1.4 *Holiday Tools*—Holidays shall be made with conventional drills of the required diameter. For use in preparing small-diameter pipe specimens such as 19.05 mm (0.750 in.) nominal diameter pipe, the use of a drill modified by substantially grinding away the sharp cone point has been found effective in preventing perforation of the metal wall of the pipe. A sharp-pointed knife with a safe handle is required for use in making physical examinations.

5.1.5 *High-Resistance Voltmeter,* for direct current, having an internal resistance of not less than 10 MΩ and having a range from 0.01 to 5 V for measuring potential to the reference electrode.

5.1.6 *Reference Electrode,* saturated $CuCuSO_4$ of conventional glass or plastic tube with porous plug construction, preferably not over 19.05 mm (0.750 in.) in diameter, having a potential of −0.316 V with respect to the standard hydrogen electrode. A calomel electrode may be used, but measurements made with it shall be converted to the $CuCuSO_4$ reference for reporting by adding −0.072 V to the observed reading.

5.1.7 *Thickness Gage,* for measuring coating thickness in accordance with Test Method G 12.

5.1.8 *Thermometer,* for measuring electrolyte temperature, general lab type, 1° subdivisions, 76.2 mm (3 in.) immersion.

5.2 *Additional Apparatus for Method B:*

5.2.1 *High-Resistance Voltmeter,* for direct current, having an internal resistance of not less than 10 MΩ and capable of measuring as low as 10 μV potential drop across a shunt in the test cell circuit.

5.2.2 *Precision Wire-Wound Resistor,* 1-Ω ± 1 %, 1-W (minimum), to be used in the test cell circuit as a shunt for current.

5.2.3 *Volt-Ohm-Meter,* for initial testing of apparent coating resistance.

5.2.4 *Metallic Electrode,* used temporarily with the volt-ohm-meter to determine apparent initial holiday status of the test specimen.

5.2.5 *Additional Connecting Wires,* 4107-cmil (14-gage

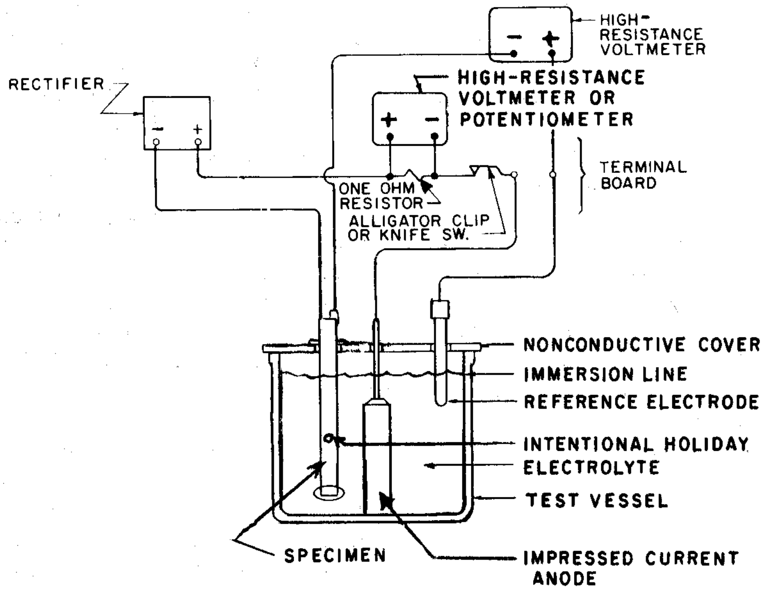
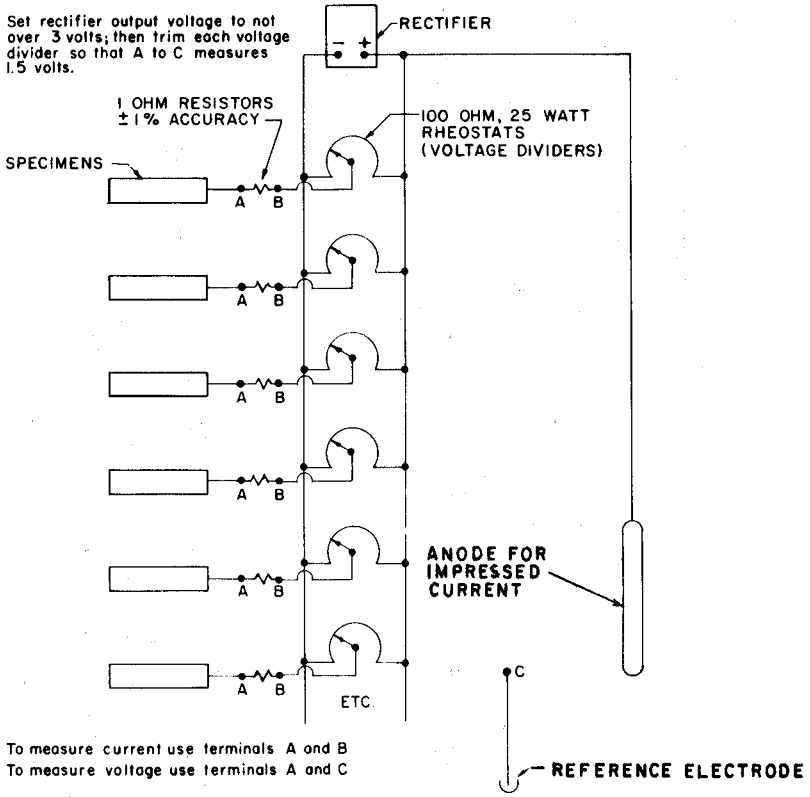


**FIG. 3 Test Assembly for Method B Using an Impressed Current with One Specimen**



Set rectifier output voltage to not over 3 volts; then trim each voltage divider so that A to C measures 1.5 volts.

To measure current use terminals A and B
To measure voltage use terminals A and C

FIG. 4 Modification of Method B (Fig. 3) Using Impressed Current to Test More than One Specimen

Awg), minimum, insulated copper.

5.2.6 *Brass Studs,* used at a terminal board, together with alligator clips or knife switches, for making and breaking circuits. Alligator clips shall not be used to connect to electrodes or specimens at the top location of test cells.

5.2.7 *Zero-Resistance Ammeter,* capable of measuring direct current as low as 10 μA may be used in the alternative method given in 9.1.3 and substituted for the apparatus described in 5.2.1 and 5.2.2.

5.2.8 *Direct-Current Rectifier,* capable of supplying constant voltage at a voltage of 1.50 ± 0.01 V, as measured between the specimen and reference electrode.

5.2.9 *Impressed Current Anode,* shall be of the nonconsumable type provided with a factory sealed, insulated copper wire.[4]

5.2.10 *Voltage Divider,* 100-Ω, 25-W rheostat, to be used if more than one specimen is to be tested as shown in Fig. 4.

## 6. Reagent and Materials

6.1 The electrolyte shall consist of potable tap water with the addition of 1 mass % of each of the following technical-grade salts, calculated on an anhydrous basis: sodium chloride, sodium sulfate, and sodium carbonate. Use freshly prepared solution for each test.

6.2 Materials for sealing the ends of coated pipe specimens may consist of bituminous products, wax, epoxy, or other materials, including molded elastomeric or plastic end caps.

6.3 Plywood or plastic material has been found suitable for the construction of test vessel covers and for the support through apertures of test specimens and electrodes. Wood dowels introduced through holes in the top ends of test specimens have been found suitable for suspending test specimens from the vessel cover.

## 7. Test Specimen

7.1 The test specimen shall be a representative piece of production-coated pipe. One end shall be plugged or capped, and sealed.

7.2 One or three holidays shall be made in each specimen. Three holidays are recommended. Recommended dimensions are given in Fig. 5. A specimen with one holiday shall have it drilled in the middle of the immersed length. If three holidays are used, they shall be drilled 120° apart with one in the center and the other two at locations one fourth the distance from top and bottom of the immersed test length. Each holiday shall be drilled so that the angular cone point of the drill will fully enter the steel where the cylindrical portion of the drill meets the steel surface. The drill diameter shall be not less than three times the coating thickness, but it shall never be smaller than 6.35 mm (0.250 in.) in diameter. The steel wall of the pipe shall not be perforated. With small-diameter pipes, where there is danger of perforating the pipe, the holiday shall be started with a standard 60° cone point and finished with a drill that has had a substantial portion of the cone point ground away.

NOTE 1—Before making the holiday, see 8.1.

7.3 The end of the pipe which will protrude above the

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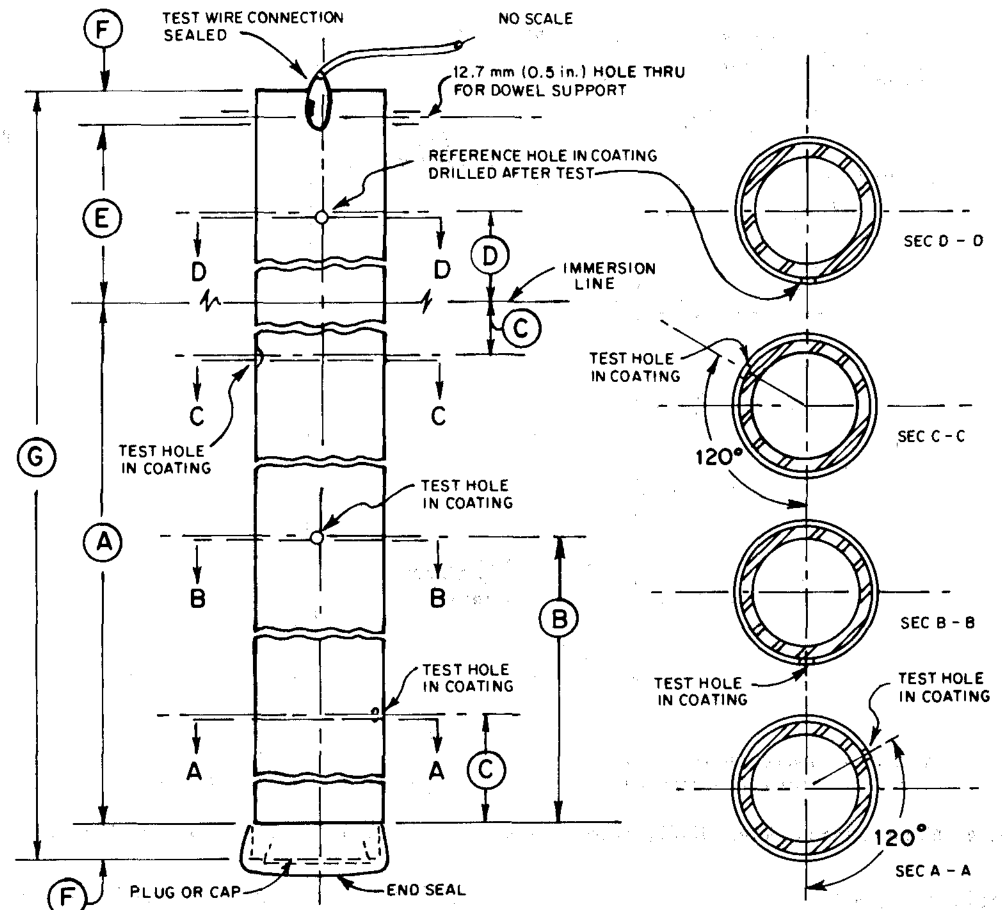
[4] Durion, a material found suitable for this purpose is available from Durion Co., Inc., Dayton OH.



| Dimension | mm | (in.) |
|---|---|---|
| A | 490.22 ± 12.7 | (19.300 ± 0.500) |
| B | 245.11 ± 12.7 | ( 9.650 ± 0.500) |
| C | 120.65 ± 6.35 | ( 4.750 ± 0.250) |
| D | 114.3 ± 6.35 | ( 4.500 ± 0.250) |
| E | 233.363, min | ( 9.1875, min) |
| F | 19.05, min | ( 0.750, min) |
| G | 762, min | (30, min) |

FIG. 5 Recommended Dimensions for Specimen

immersion line shall be provided with suitable supporting means and a separate wire connection for electrical purposes, soldered, brazed, or bolted to the pipe. The protruding end, including hanger and wire connections, shall be protected and sealed with an insulating coating material.

7.4 The specimen test area shall consist of the area between the edge of the bottom end seal and the immersion line. The bottom end seal area shall not be considered part of the area tested. Any suitable diameter and specimen length of pipe may be used, but the immersed area shall be not less than 23 227 mm$^2$ (36 in.$^2$). An area of 92 900 mm$^2$ (1 ft$^2$) has been found preferable when convenient.

## 8. Specimen Preparation

8.1 Before making artificial holidays, verify the continuity of the coating and the effectiveness of the end-cap seal as follows:

8.1.1 Immerse the test specimen and a metallic electrode in the electrolyte. Connect one terminal of the multimeter to the test specimen and the other terminal to the metallic electrode. Measure the apparent resistance in ohms, making two determinations: one with the specimen connected to the positive terminal of the multimeter; and one with the specimen connected to the negative terminal.

8.1.2 Disconnect the specimen from the multimeter but leave it immersed for 15 min. Then, measure the resistance again as in 8.1.1.

8.1.3 A significant decrease in either resistance reading after 15 min will indicate a flaw in the coating or end-cap seal. Reject the specimen if the flaw is identified in the coating. If the flaw is in the end-cap seal, it may be repaired and the resistance remeasured as in 8.1.1 and 8.1.2.

8.1.4 The lowest resistance after 15 min of immersion shall be not less than 1000 MΩ but a stable reading below 1000 MΩ may not indicate a flaw and the specimen may be used for test. All resistance measurements shall be reported in the results.

8.2 Record initial holiday diameter(s).

8.3 Measure and record the minimum and maximum coating thickness in accordance with Method G 12, and the

**Data Sheet and Report, Part I, for Method A and Method B Cathodic Disbonding of Pipeline Coatings**

1. Specimen No. ________ Report No. ________ Initials ________ Date ________
2. Pipe:

________ mm (in.) O.D. ________ mm (in.) Wall ________ mm (in.) Length

Mfgr. ________ API ________

3. Coating:

Name, No. ________
Mfgr. ________
Application method ________
Applicator ________
Thickness, mm (in.)

Max. ________ Min. ________ Av. ________ At holidays: Top ________ Middle ________ Bottom ________

4. Test:

Date Started ________ Date finished ________
Test area ________ mm$^2$ (in.$^2$)

| | Top | Middle | Bottom | Average |
|---|---|---|---|---|
| Initial holiday dia. mm (in.) | | | | |
| Final unsealed area mm$^2$ (in.$^2$) | | | | |
| (—) Initial holiday area mm$^2$ (in.$^2$) | | | | |
| = Net disbonded area mm$^2$ (in.$^2$) | | | | |
| Disbonded Equivalent Circle Diameter mm$^2$ (in.$^2$) | | | | |

5. Preliminary verification

| Group | Largest Disbonded Equivalent Circle Diameter (ECD) does not exceed | | Spontaneous Holidays |
|---|---|---|---|
| | mm | in. | |
| A | 12.7 | 0.50 | None |
| B | 25.4 | 1.00 | None |
| C | 38.1 | 1.50 | None |
| D | 50.8 | 2.00 | None |
| E | More than 50.8 | More than 2.00 | Any |

| Verification of coating continuity before starting test per Sec. 7.6 | | | |
|---|---|---|---|
| Trial | Polarity | Megohms | |
| | | Initial | After 15 min. |
| Initial | Plus | | |
| | Minus | | |
| Final | Plus | | |
| | Minus | | |

6. Rectifier Current:

If rectifier current was not continuous indicate interrupted time (min., hrs.): ________

**FIG. 6 Suggested Form, Part I, for Use in Presenting Data for One Specimen Method A and Bethod B**

thickness where each holiday is made.

## 9. Procedure for Method A

9.1 Immerse the test specimen in the electrolyte and connect it to the anode as shown in Fig. 1. Position the middle or single holiday so that it faces away from the anode. Space the anode with respect to test specimens as described in 5.1.1. Mark the correct immersion level of the test specimen with a grease pencil and maintain by daily additions of potable water as required. Perform the test at electrolyte temperature of 21 to 25°C (70 to 77°F).

9.1.1 In order to ascertain that the test cell is functioning, measure the potential between test specimen and a reference electrode immediately after starting the test and immediately before terminating it. Use temporary connections and instrumentation, as shown in Fig. 1. The potential measured shall be −1.45 V to −1.55 V with respect to a $CuCuSO_4$ reference electrode. Use the instrument described in 5.1.5.

9.2 Duration of the test period shall be 30 days. Optionally, other test periods such as 60 or 90 days may be used.

9.3 An examination shall be performed immediately upon termination of the test period as follows:

9.3.1 At the end of the test period, disassemble the cell and rinse the test area with warm tap water. Immediately wipe the sample dry and visually examine the entire test area for any evidence of unintentional holidays and loosening of coating at the edge of all holidays, including the intentional holiday, and record coating condition, for example, color, blisters, cracking, crazing, adhering deposits, etc.

9.3.2 Drill a new reference holiday in the coating in an area that was not immersed. Follow the same drilling procedure as described in 7.2.

**Data Sheet and Report, Part II, for Method B Cathodic Disbonding of Pipeline Coatings**

Electrical Monitoring, Method B

Initial Ohms + ________ − ________

| Elapsed days of Test, $T_0$ | Date and Time | Potential to Ref., V | | Actual $I_1$, µA | mV $E_2 - E_1 = \Delta E$ | Average Values on Target Dates | | |
|---|---|---|---|---|---|---|---|---|
| | | $E_1$ | $E_2$ | | | $\Delta E$, V | $I_1$, µA | (−) log $I_1$ |
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| | | | | | | | | |
| Change, Start to Termination: | | For the specimen: | | | | | | |
| | | per Initial Holiday: Av | | | | | | |

FIG. 7 Suggested Form, Part II, for Use in Presenting Data for One Specimen, Method B

9.3.3 Make radial 45° cuts through the coating intersecting at the center of both the intentional holiday and the reference holiday with a sharp, thin-bladed knife. Take care to ensure that coating is cut completely through to the steel substrate.

9.3.4 Attempt to lift the coating at both the reference holiday and the intentional holiday with the point of a sharp, thin-bladed knife. Use the bond at the reference holiday as a reference for judging the quality of the bond at the intentional holiday. Measure and record the total area of disbonded coating at the intentional holiday.

NOTE 2—The use of a transparent film having a grid laid out in small squares such as 2.54 mm (0.1 in.) on a side has been found useful. The film is placed against the unsealed area and the boundary of the unsealed area traced on the grid. The area is then obtained by counting the squares within the bonded area.

## 10. Procedure for Method B

10.1 In addition to the procedure given in Section 9, monitor the progress of the test electrically in accordance with the schedule given in 10.2 as follows:

10.1.1 If a magnesium anode is to be used, install the test assembly shown in Fig. 2. If impressed current for a single specimen is to be used, install the test assembly shown in Fig. 3; if more than one specimen is to be tested, use the modification shown in Fig. 4.

10.1.2 Measure $E_2$, the stress potential in volts between test specimen and reference electrode, with a multimeter without disconnecting the anode from the test specimen. Use the instrument described in 5.1.5. If a $CuCuSO_4$ electrode is used, immerse only temporarily.

10.1.3 Measure $I_1$, the current demand in amperes, by determining the potential drop across the 1-Ω resistor permanently installed in the test cell circuit with the multimeter described in 5.2.1. The voltage reading will be numerically equal to amperes.

NOTE 3—An alternative method of measuring current demand may be used by means of the instrument described in 5.2.4. In this method, the wire connection between test specimen and anode is temporarily broken and a zero-resistance ammeter temporarily interposed between the specimen and the anode. Reconnect the specimen to the anode with the connector wire as soon as this measurement is completed.

10.1.4 Measure $E_1$, the polarized potential, in volts. Do this with the multimeter described in 5.1.5 connected between the test specimen and the reference electrode as follows:

10.1.4.1 Disconnect the anode from the test specimen while closely observing the multimeter. As the instrument pointer falls, it will dwell significantly at the polarized value before receding further. The dwell point is $E_1$. If a $CuCuSO_4$ electrode is used, immerse only temporarily.

10.2 *Electrical Monitoring Schedule:*

10.2.1 Electrical measurements at the start of the test are defined as the average of measurements taken on the second and third days after immersion. Measurements may be taken on the day of immersion in order to ascertain functioning of the test cell, but such measurements are not to be used in calculating the change in characteristics from start to target dates in the conduct of the test.

10.2.2 Make electrical measurements at the start of a test and on a target date after 30 days. The test may be continued for 60 or 90-day targets with intermediate and corresponding electrical measurements.

10.2.3 Take electrical measurements for intermediate target dates and for the terminal date on 2 successive days prior to and including the target date. The average of readings taken on the 2 days is defined as the target date measurement.

10.2.4 Rectifier current shall be continuous. Any interruptions must be reported.

## 11. Report (see Figs. 6 and 7):

11.1 The report for Method A shall include the following:

11.1.1 Complete identification of the test specimen, including:

11.1.1.1 Name and code number of the coating,

11.1.1.2 Size and wall thickness of pipe,

11.1.1.3 Source, production date, and production run number,

11.1.1.4 Minimum-maximum coating thickness, average thickness and the thickness at the holiday,

11.1.1.5 Immersed area,

11.1.1.6 Size and number of initial holidays, and

11.1.1.7 Resistance measurements verifying continuity of the coating and effectiveness of the end cap seal as required in 8.1.

11.1.2 Dates of starting and terminating test.

11.1.3 Tally of areas that have been found unsealed on the terminal date. Areas may be reported in square millimetres (square inches) or millimetres (inches) of equivalent circle diameter of the area, or both. If more than one holiday was used, the area per holiday may be reported as an average.

NOTE 4—Equivalent Circle Diameter (*ECD*) is obtained from the formula:

$$ECD = (A/0.785)^{1/2}$$

where:

$A$ = area of holiday, mm$^2$ (in.$^2$)

11.1.4 Other information that may be pertinent.

11.2 The report for Method B shall include the following:

11.2.1 The data required in the report for Method A,

11.2.2 The relative resistances of the test specimen in ohms before the artificial holiday was made as described in 8.1.4, and

11.2.3 The results of starting, intermediate, and terminal electrical measurements. Report the following measurements:

11.2.3.1 Current demand in microamperes, or negative characteristic of the logarithm of the current in amperes, or both,

11.2.3.2 The value of $\Delta E = E_2 - E_1$ in volts, and

11.2.3.3 Change from start to termination for values 11.2.3.1 and 11.2.3.2. If more than one holiday was used the average change per holiday may be reported for 11.2.3.1.

11.2.4 Any interrupted time of the rectifier current.

## 12. Precision and Bias

12.1 Precision data are limited to two adjacent specimens taken from the same production-coated pipe and assume that the production process was uniform with respect to pipe surface condition and coating material. Specimens that were not adjacent in the as-produced condition or were taken from different lengths of pipe may represent differing process conditions. The following data should be used for judging the acceptability of results: (These precision data are approximations based on limited data, but they provide a reasonable basis for judging the significance of results.)

12.2 *Method A:*

12.2.1 *Repeatability*—Duplicate results by the same worker should not be considered suspect unless they differ by more than 12.7 mm (0.5 in.) in value *ECD* in accordance with the following equation:

$$ECD = (A/0.785)^{1/2}$$

where:

$A$ = unsealed area developed from 1 artificial holiday, mm$^2$ (in.$^2$).

12.2.2 *Reproducibility*—The results reported by one laboratory should not be considered suspect unless they differ from those of another laboratory by more than 25 mm (1 in.) for value *ECD* in the equation given in 12.2.1.

12.3 *Method B:*

12.3.1 *Repeatability*—Duplicate results by the same worker should not be considered suspect unless they differ by more than unity in the negative characteristic of the logarithm of the current demand in amperes.

12.3.2 *Reproducibility*—The results reported by one laboratory should not be considered suspect unless they differ from those of another laboratory by more than unity in the negative characteristic of the logarithm of the current demand in amperes.

# Standard Test Method for Water Penetration into Pipeline Coatings[1]

This standard is issued under the fixed designation G 9; 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.

$^{\epsilon 1}$ NOTE—Section 12 was added editorially in September 1992.

## 1. Scope

1.1 This method covers the determination of the apparent rate of depth of water penetration into insulating coatings applied to pipe.

1.2 The values stated in inch-pound units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety problems, 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 Document

2.1 *ASTM Standard:*

G12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

## 3. Summary of Test Method

3.1 The method consists of an immersion-type test where pipe specimens are suspended in an aqueous electrolyte for the duration of the test period. Electrical measurements of coating capacitance and dissipation factor are used to follow the water absorption rate of the test materials.

## 4. Significance and Use

4.1 The deterioration of an insulating coating film is intimately related to its moisture content. The water penetration test provides a means for monitoring the passage of moisture through a coating material by means of changes in its dielectric constant. When expressed in relation to time, the test data will reflect a rate of deterioration which is a characteristic of the coating material and will bear a relation to its expected useful life as an insulating coating. The test for water penetration will also provide information that is useful in establishing the optimum coating thickness for a given material.

## 5. Apparatus

5.1 *Immersion Cell*—Any suitable nonmetallic vessel to contain the test specimens. Dimensions of the vessel shall permit the following requirements:

5.1.1 Test specimens shall be suspended vertically with at least 25 mm (1.0 in.) clearance from the sides and bottom.

5.1.2 Test specimens shall be separated by not less than 25 to 40 mm (1 to 1.5 in.) and a vertically suspended anode shall be placed at an equal distance from each specimen not less than the separation of distance.

5.1.3 The test vessel shall be deep enough to allow for immersion of the samples in the electrolyte to the level specified in 8.1.

NOTE 1—Commercially available, glass battery jars in 2-dm$^3$ (0.55-gal) and 10-dm$^3$ (2.7-gal) sizes can be conveniently used with 19-mm (0.75-in.) and 51-mm (2.0-in. nominal) diameter specimens, respectively.

5.1.4 A suitable sample support plate fabricated from a material having a low dielectric constant shall be used to suspend the samples and anode above the immersion cell. The support plate shall contain an access hole for the reference electrode. A typical test cell is illustrated in Fig. 1.

5.2 *Electrolyte,* consisting of tap water with the addition of 1 weight % of each of the following technical-grade anhydrous salts: sodium chloride, sodium sulfate, and sodium carbonate.

NOTE 2—Add 30 g (1.05 oz) of sodium chloride for each dm$^3$ (0.26 gal) of water.

5.2.1 The electrolyte in the immersion cell shall be maintained at the proper level by regular additions of tap water. The electrolyte shall not be reused after completion of the test.

5.3 *Voltage Source*—A direct current power supply, capable of supplying low ripple voltage shall be used to maintain a potential difference of 6.0 ± 0.1 V dc between each of the test specimens and a common electrode.

5.4 *Connectors*—Wiring connections from the anode to the specimen shall be of No. 18 AWG insulated copper. Attachment to the anode shall be sealed and kept above the level of the electrolyte. Attachment to the specimen shall be made by a method that will allow disconnection from the anode when the measuring bridge is in use. A convenient means for accomplishing this is through the use of insulated pin-type jacks.

5.5 *Capacitance Bridge*[3]—Measurements of equivalent specimen capacitance and coating dissipation factor shall be made with a low-voltage a-c, resistive-ratio-arm type measuring bridge having the following characteristics:

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 29, 1987. Published July 1987. Originally published as G 9 – 69 T. Last previous edition G 9 – 82.

[2] *Annual Book of ASTM Standards*, Vol 06.02.

[3] A bridge found to meet the requirements specified is the General Model 1656 Impedance Bridge, GenRad, Concord, MA 01742.

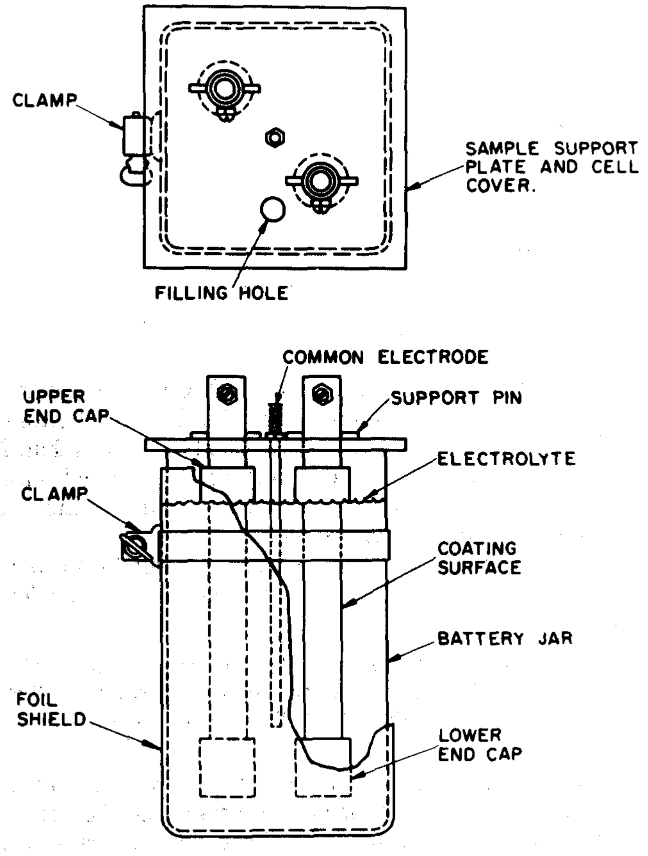


FIG. 1 Typical Test Cell

5.5.1 Oscillator frequency, 1 kHz ± 2 %,
5.5.2 Series capacitance range, 1 to 1100 pF ± 1 %,
5.5.3 Series capacitance sensitivity, 0.5 pF,
5.5.4 Dissipation factor range, 0.001 to 1.0 at 1 kHz, and
5.5.5 Dissipation factor sensitivity, 0.001 at 1 kHz.

5.6 *Measuring Circuit*—Measurements of specimen capacitance and coating dissipation factor shall be made using a circuit that places the sample unknown in series with the comparison circuit of the measuring bridge. Connection of the unknown to the measuring bridge shall be made in such a manner as to eliminate the introduction of stray capacitance into the measuring circuit. A diagram for connecting the test cell to the bridge is shown in Fig. 2. In this arrangement, both the test leads are shielded and the chassis of the bridge is grounded. The immersion cell shall also be shielded to avoid capacitance effects from surrounding objects.

NOTE 3—A shield for the test cell can conveniently be fabricated from most commercially-available tin or aluminum foils of approximately 0.0382-mm (0.0015-in.) thickness and formed around the container.

5.7 *Thickness Gage*—Measurements of coating thickness will be required for this test. Any instrument suitable for use with Test Method G 12 can be used.

5.8 *Anode,* fabricated from 4.76-mm (0.1875-in.) diameter AISI Type 303 stainless-steel rod, and shall be 178 mm (7.00 in.) long, with the upper 50 mm (2.00 in.) threaded to accept a locking nut.

## 6. Test Specimen

6.1 The test specimen shall be a representative piece of production-coated pipe and shall be free of obvious coating flaws or defects (see Fig. 3). Any suitable diameter and specimen length can be used. Physical limitations of the immersion cells suggested in 5.1.3, Note 1, make it necessary to restrict the over-all sample length to approximately 300 mm (12.0 in.) for both the 26.7-mm (0.75-in. nominal) and 60.3-mm (2.0-in. nominal) diameter coated pipe specimens.

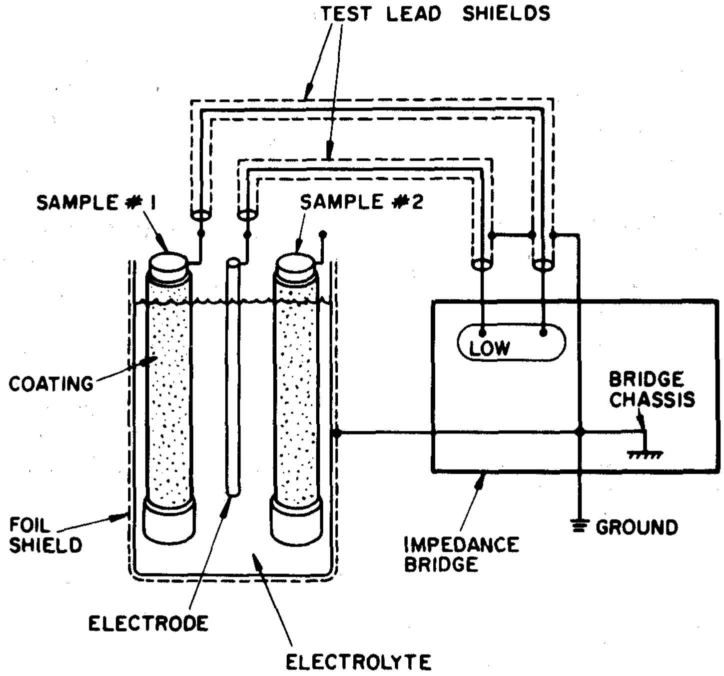
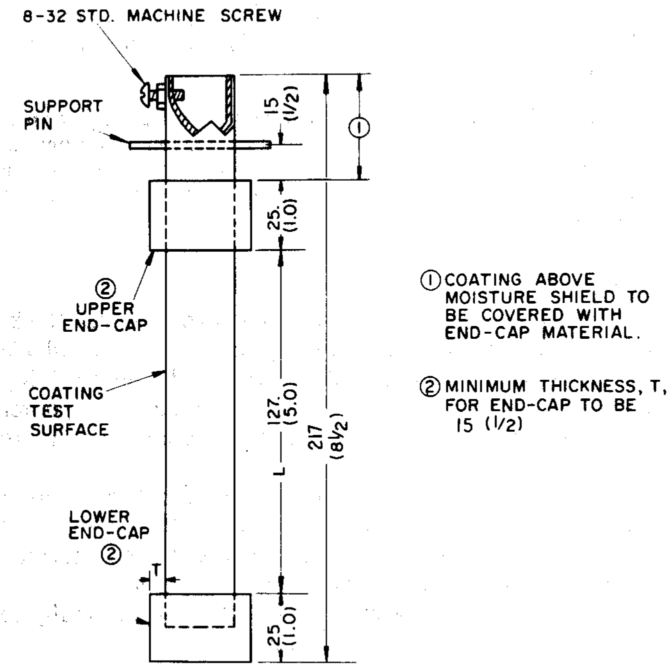


FIG. 2 Connecting the Test Cell to the Bridge



NOTE—Dimensions are in millimetres with inches in parentheses.

**FIG. 3 Detail Drawing of Pipe Specimen**

6.2 The upper and lower ends of the test specimen shall be plugged and sealed with nonconductive caps of sufficient bulk to minimize effectively capacitive end effects in the measuring circuit. For this purpose, an end-cap thickness of from 13 mm (0.5 in.) to 19 mm (0.75 in.) shall be maintained.

6.2.1 The end-cap material shall have a dielectric constant in the range from 2 to 6, bond well to the coating surface, and exhibit a low water-absorption rate. Several commercially available poly(vinyl chloride)-paraffin compounds,[4] are well suited for this purpose. They have a melting point in the 150 to 200°C (300 to 390°F) range, can be poured into molds around the pipe sample, and appear as resilient, durable solids at room temperature.

NOTE 4—Using these materials, the end-caps can be applied to the required thickness by repeated dipping of the sample ends into a molten-wax bath, or through the use of light-weight, disposable molds of aluminum foil or paper formed around the pipe sample to allow the casting of the caps directly to the surface of the coated pipe sample.

6.3 The end of the specimen which will protrude above the immersion line shall be provided with a suitable means of support and a separate wire connection for electrical purposes. The protruding end of the sample shall be waterproofed with a thin coating of end-cap material (see Fig. 1).

## 7. Preliminary Test Measurements

7.1 *Coating Thickness*—Measure and record the coating thickness by referring to Test Method G 12.

7.2 *Specimen Length*—Measure and record the length of exposed coating surface, between the end caps.

7.3 *End-Cap Capacitance*—Vertically suspend the test specimens and anode in the immersion cell, observing the clearances specified in 5.1.1 through 5.1.3. Fill the container with the electrolyte until it just covers the lower end cap. Energize the impedance bridge and measure the series capacitance, $C_c$ of the lower end cap.

7.4 *Initial Coating Capacitance*—Add additional electrolyte to the immersion cell until its level reaches the lower edge of the upper end cap. Immediately measure and record the initial series capacitance, $C_0$, and dissipation factor, *DF*, of the specimen.

## 8. Procedure

8.1 Energize each specimen by connecting it to the negative side of the voltage source. Keep each sample energized and immersed to the lower edge of the upper end cap for the duration of the test period. Maintain the specified electrolyte level through regular additions of tap water.

8.2 Throughout the test make periodic measurements of the series capacitance and dissipation factor of the immersed specimens in the following manner:

8.2.1 Temporarily disconnect the test specimen from the voltage source. Verify that the electrolyte is at the proper level within the immersion cell. Connect the measuring bridge between the test specimen and stainless-steel anode. Energize the bridge and measure the series capacitance, *C*, and dissipation faction, *DF*, of the test specimen.

8.2.2 Using the observed value of series capacitance, $C_s$, calculate the apparent depth of water penetration by the method described in 9.2.

8.2.3 Repeat the measurements of series capacitance and specimen dissipation factor at periodic intervals throughout the duration of the test. The frequency of measurement will depend upon the rate of deterioration of the coating sample. Where the water penetration process is relatively rapid, daily readings of sample capacitance and dissipation factor will be required. Normally, readings made at weekly intervals will adequately define the penetration rate.

NOTE 5—Some coatings exhibit an initial rise in capacitance and dissipation factor, but reach a state of equilibrium in 6 to 9 months. An increase in capacitance and dissipation factor following this period of equilibrium, (or failure to reach equilibrium) indicates impending failure. A sample can be considered to have failed when the dissipation factor reaches a value of 1.0.

## 9. Calculations

9.1 *Dielectric Constant*—Calculate the dielectric constant, $K_0$, for the coating film as follows:

$$K_0 = \frac{(C_0 - C_c) \ln [(2t_0 + d)/d]}{NL}$$

where:

$K_0$ = dielectric constant,
$C_0$ = initial coating capacitance, pF,
$C_c$ = end-cap capacitance, pF,
$d$ = outside pipe diameter, mm (in.),
$t_0$ = initial coating thickness, mm (in.),
$L$ = exposed coating length, mm (in.), and
$N$ = 0.0556 when $d$, $t_0$, $L$, are in mm (1.413 when $d$, $t_0$, $L$, are in in.).

9.2 *Apparent Depth of Penetration*—Calculate the depth

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[4] A material found suitable for this purpose is Boler Petroleum No. L-480-86 Wax Blend, Boler Petroleum Co. 85 Old Eagle School Rd., Wayne, PA 19087.

of water penetration by applying the calculated value of $K_0$ from 9.1 and the measured value of equivalent series capacitance, $C$, to the following equations:

$$t_p = t_0 - t$$

$$t = (d/2)[(e^M) - 1]$$

where:

$M = NK_0L/(C - C_c)$,
$t$ = unpenetrated coating thickness, mm (in.),
$C$ = series capacitance, pF, and
$t_p$ = depth of penetration, mm (in.).

## 10. Report

10.1 The report shall include the following:

10.1.1 Complete identification of specimen, including:

10.1.1.1 Name and code number of the coating,

10.1.1.2 Size of pipe,

10.1.1.3 Source, production date, and production-run number,

10.1.1.4 Minimum, maximum, and average coating thickness,

10.1.1.5 Dates of starting and terminating test, and

10.1.1.6 Other information that may be pertinent,

10.1.2 Magnitude and polarity of d-c voltage applied to sample during the test period,

10.1.3 Length of the test period in days,

10.1.4 Apparent depth of water penetration for the test period indicated,

10.1.5 Initial value of coating dissipation factor, and

10.1.6 Value of coating dissipation factor at the end of the test period.

NOTE 6—For the purpose of monitoring coating performance, plotted graphs of apparent depth of water penetration versus time in rectangular coordinates and coating dissipation factor versus time in semilogarithmic coordinates will render useful information over the duration of the test period.

## 11. Precision

11.1 Due to the range of coating formulations, thicknesses, densities, etc., found among commercially available coated pipe samples, the overall accuracy and reproducibility of the test results by these methods will tend to be poorer than those expected on insulating material of a more uniform nature.

11.2 The precision (reproducibility) of the dissipation factor and depth of penetration by these methods in general is considered to be such that when two tests are performed consecutively on the same specimen under identical conditions, the difference between the two results may normally be expected nor to exceed ± 5 % of their mean.

## 12. Keywords

12.1 capacitance; coating; dielectric constant; dissipation factor; immersion; pipeline; water absorption; water penetration

Designation: G 10 – 83 (Reapproved 1988)

# Standard Test Method for Specific Bendability of Pipeline Coatings[1]

This standard is issued under the fixed designation G 10; 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 method covers the specific determination of the effect of short-radius bends on coatings applied to 33.4-mm (1-in. nominal) diameter pipe.

1.2 *This standard may involve hazardous operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this practice to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The values stated in SI units to three significant decimals are to be regarded as the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

G 6 Test Method for Abrasion Resistance of Pipeline Coatings[2]

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

## 3. Summary of Method

3.1 The method consists of bending a 33.4-mm (1-in. nominal) diameter specimen of coated pipe around a mandrel to produce a range of short-radius bends. Coating failure in the form of cracking or loss of adhesion is detected through visual and electrical inspection of the bent specimen.

## 4. Significance and Use

4.1 This test will provide information on the ability of coatings applied to pipe to resist cracking, disbonding, or other mechanical damage as a result of bending. Because the test is applied to coated pipe from commercial production, the results can be directly used in the selection of similar materials for service. The test also has application as a quality control method when variations in coating application or material formulation will affect bending performance.

## 5. Apparatus

5.1 The bending apparatus shall be essentially as shown in Figs. 1 to 3 and shall include the following:

5.1.1 *Variable-Radius Mandrel,* constructed from four 19-mm (0.75-in.) thick pieces of plywood, bolted together and conforming to the geometric shape shown in Fig. 1. The geometric construction is accomplished by laying out along the outer edge of the mandrel a series of seven consecutive arcs at decreasing radii of 610, 530, 460, 380, 300, and 230 mm (24, 21, 18, 15, 12, and 9 in.). The first five arcs shall be carried through a 45-deg angle to the next point of tangency. A 45-deg V-notch shall be cut into the edge of the mandrel for seating the pipe specimen. Holes shall be drilled at appropriate locations in the mandrel face for positioning the lever arm and fastening pipe clamps.

5.1.2 *Lever Arm-Roller Assembly*—A 1.83-m (72-in.) lever arm with nylon roller supplies the mechanical advantage necessary to bend the pipe specimen. The lever arm shall contain a series of holes which are used to maintain proper clearance between the roller and pipe sample during the bending operation.

5.2 *Thickness Gage*—Measurements of coating thickness will be required for this test. This shall be done in accordance with Method G 12.

5.3 *Holiday Detector*—A low-voltage d-c holiday detector of the wet-sponge type, specified in Method G 6, shall be used to locate breaks in the coating film.

## 6. Test Specimens

6.1 The test specimen should be 2.5 m (100 in.) in length. It should be representative of production-coated pipe and be free of obvious coating flaws or defects. Coating specimens shall be applied to 33.4-mm (1-in. nominal) diameter pipe.

## 7. Conditioning

7.1 The specimen shall be exposed to room temperature for a sufficient time to ensure thermal equilibrium in the pipe and coating. A temperature in the range from 20 to 30°C (68 to 85°F) shall be considered room temperature.

## 8. Procedure

8.1 Perform the test at a room temperature of 21 to 25°C (70 to 77°F).

8.2 Measure the applied coating thickness of each specimen in accordance with Test Method G 12.

8.3 Place the conditioned test specimen into the V-notch. Secure the pipe in place with clamp No. 1 and tighten the thumb screw. Remove the threaded handle from clamp No. 1 to allow for clearance of the lever arm. With the steel pivot pin at hole A of the lever arm (see Fig. 3), insert the pin into the socket on the bed of the bending jig. Place the nylon roller at position C on the lever arm. Apply the roller to the pipe specimen, and with a constant, even force with the lever handle, bend the pipe aound the mandrel until the 460-mm (18-in.) radius bend has been made. Insert and secure pipe clamp No. 2. Continue bending the pipe until the 300-mm (12-in.) radius has been reached. Insert and secure pipe

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[1] This method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

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[2] *Annual Book of ASTM Standards,* Vol 06.02.

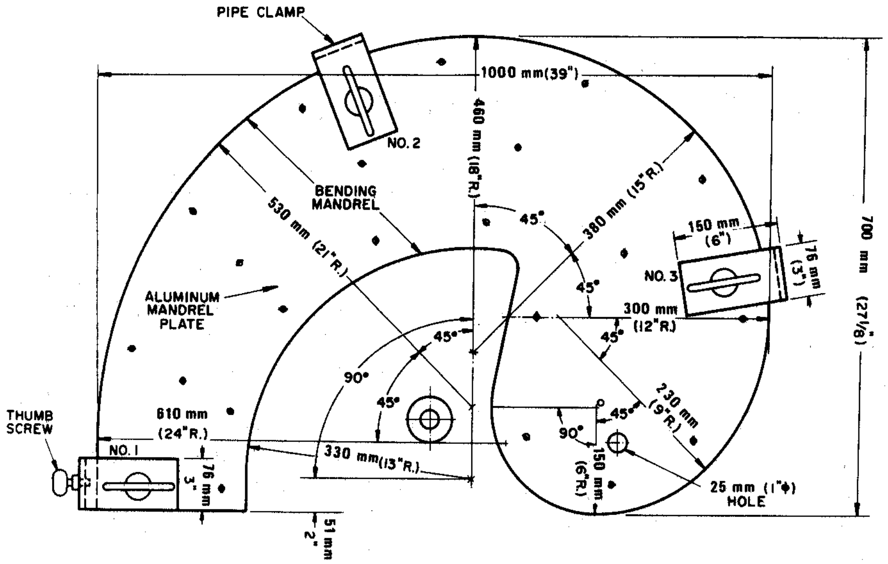

FIG. 1 Variable-Radius Mandrel

clamp No. 3. Reposition the lever arm by moving the pivot pin to the 25-mm (1-in.) diameter hole in the mandrel surface. Move the nylon roller to position B on the lever arm. Apply the roller to the pipe and complete the bending operation by forming the specimen through the 230-mm (9-in.) radius.

8.4 Examine the bent specimen wth a wet-sponge holiday detector to determine at what point, if any, cracking of the coating film occurred. Note the number, size, location, and type of cracks present. Examine the pipe specimen for any loss of coating bond caused by the bending operation.

NOTE 1—The possibility of some coatings developing stress-induced cracks in the period following the bending operation should not be overlooked. If this is a consideration, the bent specimen should be retained for a 24-h post-bent inspection.

## 9. Report

9.1 The initial test report shall include the following:

9.1.1 Complete identification of the coated pipe tested, including: name and type of coating, average coating thickness, minimum coating thickness, maximum coating thickness, manufacturer's lot number, and date of manufacture.

9.1.2 Temperature of the pipe specimen as tested,

9.1.3 Radius at which cracking first occurred,

9.1.4 Description of the type of cracking,

9.1.5 Location and extent of any loss in pipe-to-coating bond,

9.1.6 Number of specimens tested,

9.1.7 Any peculiar characteristics of the specimen noted during the test or after immediate removal from the test apparatus, and

9.1.8 Post-bend retention time.

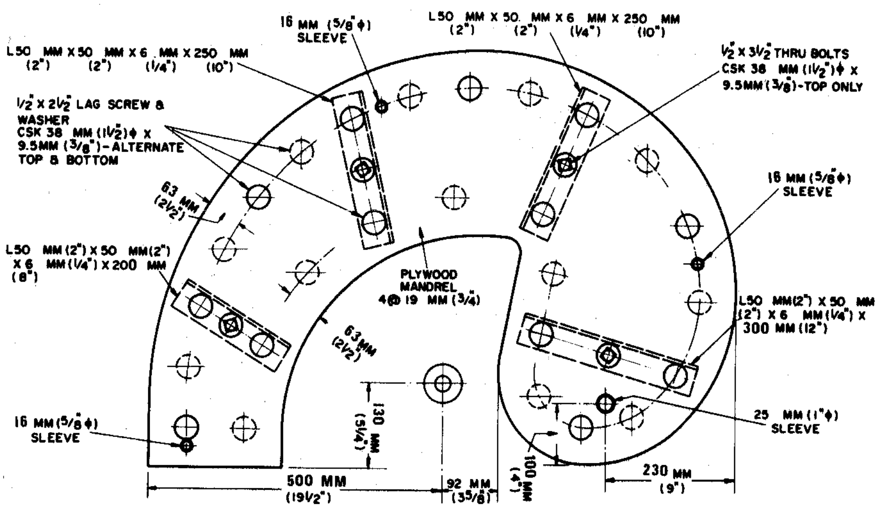

FIG. 2 Details of Mandrel Assembly and Support Brackets

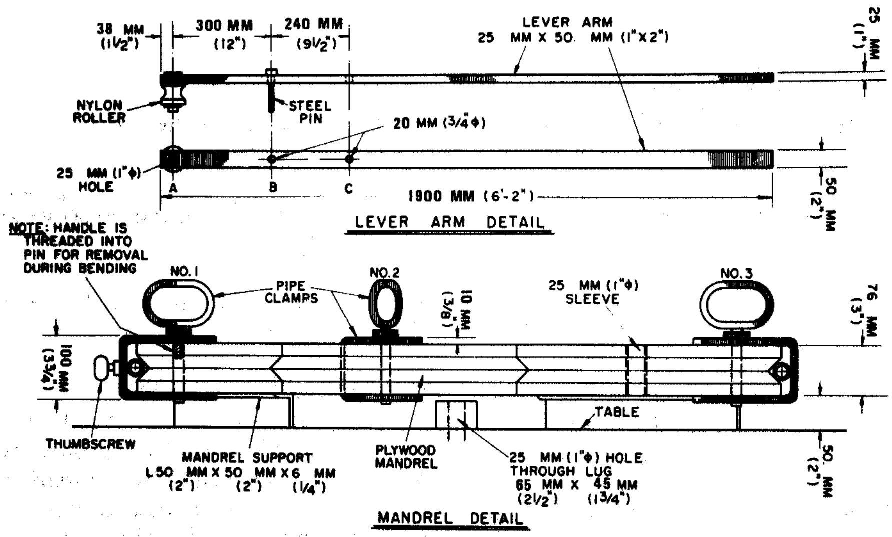

**FIG. 3 Details of Lever Arm and Pipe Clamps**

# Standard Test Method for Effects of Outdoor Weathering on Pipeline Coatings[1]

This standard is issued under the fixed designation G 11; 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 is intended to define conditions for the exposure of coated pipe to weather.

1.2 This test method specifies qualifications for the samples, procedure to be followed in exposure to weather and procedure for evaluating effects of exposure including visual examination and other tests.

1.3 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 The values stated in SI units to three significant decimals are to be regarded as the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

G 8 Test Methods for Cathodic Disbonding of Pipeline Coatings[2]

G 10 Test Method for Specific Bendability of Pipeline Coatings[2]

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

G 13 Test Method for Impact Resistance of Pipeline Coatings (Limestone Drop Test)[2]

G 62 Test Methods for Holiday Detection in Pipeline Coatings[2]

G 70 Test Method for Ring Bendability of Pipeline Coatings (Squeeze Test)[2]

## 3. Significance and Use

3.1 Since coated pipe may be stored outdoors for long periods before burial, weathering tests of the type described in this test method are needed to evaluate the stability of these coatings stored outdoors. The results obtained should be treated only as indicating the general effect of weathering. Exposure conditions vary greatly from year to year, from one part of a year to another, and from locality to locality. The results of short-term exposure tests in the north are more meaningful if exposure is started in the summer followed by a winter season. In southern areas where climatic conditions are more uniform throughout the year, the time of year when short-term exposure is started is less critical. In all localities, the longer the exposure period, the more reliable are the results obtained.

## 4. Summary of Test Method

4.1 The effects of outdoor weathering on pipeline coatings after 6, 12, and 24 months' exposure are determined visually and by electrical means by comparing exposed samples of coated pipe with unexposed samples of coated pipe before and after impact and bending tests. At the beginning of the test the starting samples are qualified by a cathodic disbonding test.

## 5. Location of Test Sites

5.1 Weathering racks shall be located in cleared areas representative of local outdoor weather conditions.

## 6. Apparatus

6.1 *Racks*—The horizontal racks shall be any suitable pipe storage racks of sufficient height to prevent any undesirable effects of vegetation growth during the period of exposure. Racks may be constructed from a variety of materials, but pipe specimens must rest on a nonconductive surface. An example of a suitable rack is illustrated in Fig. 1.

## 7. Sampling

7.1 Each starting sample shall consist of coated 26.7-mm (¾-in. nominal) diameter steel pipe from a production lot having a minimum length of 4.4 m (14.4 ft). The sample shall be from a lot produced under conditions capable of being duplicated on a production scale.

NOTE 2—Pipe having a nominal diameter of 33.4-mm (1-in. nominal) can be used and is the largest diameter that can be evaluated in accordance with Test Method G 10. Larger diameter pipes can be evaluated in accordance with Test Method G 70.

7.2 Each starting sample shall have a piece 600-mm (24-in.) long cut from one end by sawing for use in qualification testing (Section 8) and designated the "qualification sample." The remainder of each starting sample shall be designated the "sample."

7.3 The total number of samples for each coating in test shall be four, divided as follows:

7.3.1 One to be removed after 6 months exposure,

7.3.2 One to be removed after 12 months exposure,

7.3.3 One to be removed after 24 months exposure, and

7.3.4 One to be used for determining original control values, which should be obtained as soon as possible on receipt of the pipe.

## 8. Procedure for Qualification Testing

8.1 Test each qualification sample in accordance with Test Methods G 8, Method B.

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

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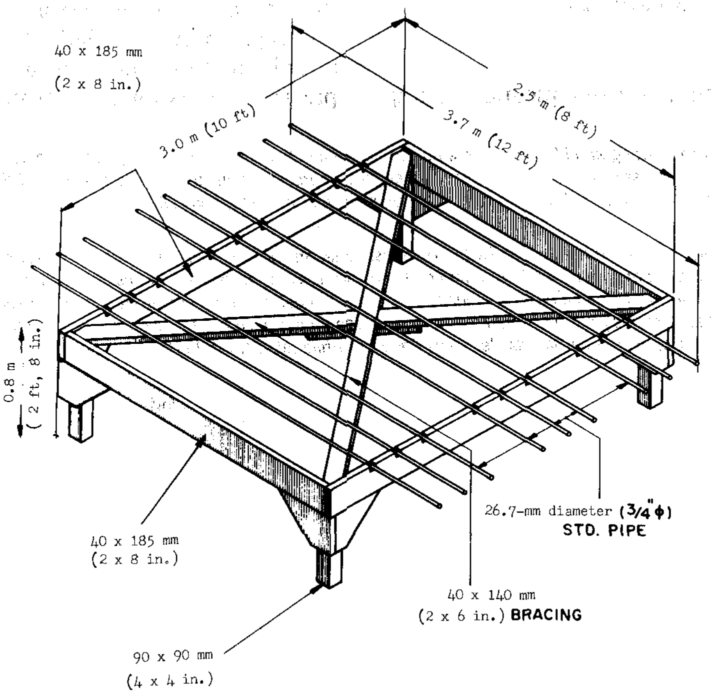
[2] *Annual Book of ASTM Standards*, Vol 06.02.



NOTE—All lumber is to be pretreated and painted.

**FIG. 1 Construction Details of Outdoor Weathering Rack**

8.2 Begin qualification testing at the same approximate time as sample testing.

8.3 If the results of the qualification testing are within the precision limits given in Test Methods G 8, consider the samples to be reasonably equivalent and proceed with sample testing.

8.4 If the results of the qualification testing are not within the precision limits given in Test Methods G 8, consider the samples to be unequivalent and discontinue exposure testing.

## 9. Procedure for Sample Testing

9.1 Identify the test samples with a letter, number, or symbol for ready identification after exposure. The marking shall not interfere with either the exposure or the testing.

9.2 Note and record the appearance of the sample.

9.3 Measure and record coating thickness in accordance with Test Method G 12.

9.4 Check coating for holidays with the appropriate detector, as described in Test Methods G 62.

9.5 Make two 80-mm (3-in.) long scribes through the coating to bare metal. Begin one scribe at a point approximately 230-mm (9-in.) from one end of the pipe and continue it for 80-mm along the length of the pipe; begin the other scribe at a point approximately 230-mm from the opposite end of the pipe and continue it for 80-mm along the length of the pipe. One scribe should be on the top of the pipe, the other on the bottom.

9.6 Mount the samples on the rack as shown in Fig. 1 with one scribe facing skyward and one facing toward the ground. Group the three samples together so that like samples are lying alongside one another.

9.7 After exposure, again note and record the appearance of the sample and check for holidays in the manner followed prior to exposure. Next examine the sample visually for cracking, checking, blistering, corrosion, undercutting from the intentional scribe, and any other form of impairment of the coating.

9.8 Finally, cut a 300-mm (12-in.) specimen from one end of the sample, where the scribe is facing downward, and subject to an impact test in accordance with Test Method G 13. In the impact test, position the specimen so that the scribe faces downward.

9.9 Subject the remaining 2.5 m (8.2 ft) to a bend test in accordance with Test Method G 10. In the bend test, position the skyward face of the sample as the outside of the bend.

9.10 Follow the procedure in 9.7, 9.8, and 9.9 on samples removed after each of the three exposure periods and compare results with those obtained on the control sample in 7.3.

## 10. Report

10.1 The report shall include the following:

10.1.1 Results of qualification tests,

10.1.2 Sample number and description of coating system including: surface preparation, method of application, and coating thickness,

10.1.3 Appearance of sample and presence or absence of holidays prior to exposure,

10.1.4 Appearance of sample, presence, or absence of holidays after exposure, and duration of exposure,

10.1.5 Visual evidence of coating failure as noted by 9.7,

10.1.6 Results of impact and bend tests after exposure as compared with those for the control sample, and

10.1.7 Type of environment and exposure.

## 11. Precision

11.1 The precision requirements for qualification testing in Section 8.3 of this test method and the precision requirements given in Test Method G 12 shall apply. Variations in samples and qualification samples shall be within respective precision limits for this test method to be valid.

# Standard Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[1]

$^{\epsilon 1}$NOTE—Section 9 was added editorially in October 1992.

## 1. Scope

1.1 This test method describes the nondestructive measurement of the thickness of a dry, nonmagnetic coating applied to the external surface of steel pipe. The method is recommended for coating thicknesses up to 6 mm (0.240 in.) and for any diameter pipe, but not smaller than 10 mm (0.5 in.). It does not apply to excessively soft films.

1.2 The values stated in SI units to three significant decimals are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety problems, 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. Summary of Test Method

2.1 The coating thickness is determined by an instrument that measures thickness by nondestructive techniques, based on variations in magnetic flux or magnetic attraction between its detection unit and the magnetic base material.

## 3. Significance and Use

3.1 Measurements of film thickness are an essential part of most ASTM test methods related to coatings on steel pipe. Adequate thickness is important for a coating to fulfill its function of preventing or mitigating corrosion of steel pipelines.

3.2 The accuracy of the thickness measurements may be influenced by the deformability of the coating. This test method is not applicable to coatings that would be readily deformable under the force exerted by the probe of the measuring instrument.

## 4. Apparatus

4.1 The apparatus shall be a nondestructive-type thickness gage capable of being standardized over its range of intended use. It shall be designed so that variations in magnetic flux or magnetic attraction between its detection unit and the steel base can be calibrated to indicate the thickness of the coating material.

4.2 It shall be suitable for measuring thicknesses of dry, nonmagnetic coatings on either a flat or a circular base.

## 5. Standardization of Instruments

5.1 The instrument shall be standardized in accordance with the manufacturer's instructions before use by employing suitable thickness standards. The standardization shall be checked at frequent intervals during use.

5.2 Nonmagnetic standards of uniform thickness are available in either of two types, foil or coated substrate, as supplied or recommended by the manufacturer of the instrument.

5.2.1 *Foils (Shims)*—The thickness of the foil should be as close as possible to the expected thickness of the coating to be measured. The foil should be placed on a smooth, clean, low-carbon, steel plate, making certain that there is intimate contact between the foil and the substrate. It is recommended that single foils of proper thickness be used and that foils be replaced frequently, since they are subject to indentation. Foils are advantageous for standardizing on curved surfaces and are often more readily available than coated standards.

NOTE 1—For some instruments there is an effective depth of penetration of the field created by the instrument probe. This is the critical depth of thickness at which the instrument will no longer be affected by increases of substrate thickness. Since it depends on the instrument and substrate, it should be determined experimentally.

5.2.2 *Coated Substrate Standards*[2]—These standards consist of nonmagnetic coatings of known thickness permanently bonded to a low-carbon steel (1010 grade) substrate, 8 mm (0.30 in.) thick. The thickness standard should be as close as possible to the expected thickness of the coating to be measured.

NOTE 2—If the curvature of the coating to be measured is sufficient to preclude standardization on a flat surface, the standardization should be done with a standard foil on a substrate with the same curvature as the coating to be measured.

## 6. Procedure

6.1 Operate each instrument in accordance with the manufacturer's instructions.

6.2 Take a minimum of twelve readings per piece of pipe as follows: Take three equally spaced readings along the top surface; rotate the pipe 90° three times, taking three equally

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G3.06 on Durability of Pipeline Coatings and Linings.

Current edition approved June 24, 1983. Published August 1983. Originally published as G 12 – 69 T. Last previous edition G 12 – 77.

[2] Coated substrate standards may be obtained from the U. S. Department of Commerce, National Bureau of Standards, Standard Materials Unit, Washington, DC 20234.

spaced readings after each rotation. Record the average, minimum, and maximum of the twelve readings as the coating thickness.

NOTE 3—Instruments with two contact poles shall be positioned on the coating surface with the poles in the same plane.

NOTE 4—The anchor pattern of the steel pipe substrate must be considered for its effect on the accuracy of the measurements if the anchor pattern is greater than 25 % of the coating thickness.

NOTE 5—Foreign materials such as dirt and grease shall be removed by suitable cleaning without removing any coating materials.

NOTE 6—Measuring instruments and methods may depend on the technique of the operator. For example, the pressure applied to a probe and the rate of applying a balancing force to a magnet will vary from one individual to another. Often such differences can be reduced or eliminated either by having the gage calibrated by the same operator who will make the measurement or by using constant-pressure probes. Probes must be positioned perpendicular to the surface of the specimen at the point of measurement. Some magnetic instruments of the magnetic-attraction type require the pole piece to be in a vertical position. If a magnetic instrument is to be used in a horizontal or upside-down position, its calibration for that position should be verified.

NOTE 7—Instruments using magnetic attraction are sensitive to the speed with which the magnet is removed from the specimen. The magnet must be removed so that thickness measurements are not affected by speed of removal.

## 7. Report

7.1 The report shall include the following:

7.1.1 Complete identification of the specimen including: name and code number of the coating; pipe diameter; source; production data, including surface preparation of the substrate and depth of anchor pattern, if known; and production run number.

7.1.2 Name and type of instrument used, and method of standardization, and

7.1.3 Number of thickness measurements taken, and the average, minimum, and maximum value of the measurements.

## 8. Precision

8.1 The repeatability and reproducibility of thickness measurements will depend on the instrument design, the operator, and the particular measuring application.

8.1.1 *Repeatability*—When the same instrument is used by the same operator, duplicate measurements on the same sample should agree with each other within ±5 %.

8.1.2 *Reproducibility*—Separate operators using different instruments on a known thickness standard should obtain average results agreeing with each other within 10 % of the thickness standard.

## 9. Keywords

9.1 coatings; film; nondestructive; nonmagnetic; pipeline; thickness

**Designation: G 13 – 89**

# Standard Test Method for Impact Resistance of Pipeline Coatings (Limestone Drop Test)[1]

This standard is issued under the fixed designation G 13; 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 relative resistance of pipeline coatings to impact by observing the effects of falling stones on coated pipe specimens.

1.2 *This standard may involve hazardous operations and equipment. This standard does not purport to address all of the safety problems 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.*

1.3 The values stated in SI units are to be regarded as the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

G 62 Test Methods for Holiday Detection in Pipeline Coatings[2]

2.2 *AASHTO Standard:*

M80-51 (No. 67) Specification for Coarse Aggregate for Portland Cement Concrete

## 3. Summary of Test Method

3.1 The impact resistance of pipeline coatings is determined by dropping weighed amounts of a specified type of limestone through a chute onto a coated pipe specimen. Results are reported as the number of drops required to pierce through the coating to bare metal, as determined visually or electrically.

## 4. Significance and Use

4.1 This test is intended to simulate the effects of backfilling after pipe has been placed in the trench. The backfill is often rocky soil and, if it is unscreened and the coated pipe is unshielded by sand or other protective padding, the falling rocks may seriously damage the coating.

## 5. Apparatus

5.1 The impact apparatus shall be essentially as shown in Fig. 1 and shall include the following:

5.1.1 *Box with Chute,* providing a means of dropping stones from a height of 1830 mm (6.0 ft) measured to the top of a piece of coated pipe under test. Construction details are shown in Fig. 2.

NOTE 1—The box and chute described in Fig. 2 are designed for testing coatings on 100-mm (4-in.) and 150-mm (6-in.) pipe specimens. Smaller diameter pipe may be used by inserting wood V-blocks in the trough in the box below the chute.

5.1.2 *Stones,* hard, coarse, limestone aggregate, conforming to AASHTO Designation: M80-51 (No. 67) which is taken from American Association of State Highway and Transportation Officials "Standard Specifications for Coarse Aggregate for Portland Cement Concrete."

NOTE 2—These specifications cover the quality and size of coarse aggregate. No. 67 designates a 19-mm (¾-in.) to No. 4 size stone with the following sieve analysis:

| Square Opening, mm (in.) | Weight % Passing |
|---|---|
| 25 (1) | 100 |
| 19 (¼) | 95 to 100 |
| 10 (⅛) | 20 to 55 |
| No. 4 | 0 to 10 |

5.1.3 *Bucket*—Any suitable bucket that will hold 16 kg (35 lb) of stones.

5.1.4 *Holiday Detectors*—Two types are needed as described in Test Method G 62.

NOTE 3—A holiday is defined as small faults or pinholes that permit current drainage through protective coatings on steel pipe.

5.1.5 *Thickness Gages*—Any instruments suitable for use with Test Method G 12.

## 6. Test Specimens

6.1 Test specimens shall be 600 mm (24 in.) long and shall be cut from a representative piece of coated pipe. Only holiday-free specimens shall be used in the test.

## 7. Procedure

7.1 Perform the test at a room temperature of 21 to 25°C (70 to 77°F).

7.2 The procedure consists of dropping up to 10 buckets of stones on coated specimens. The number of buckets of stones required represents the resistance of the coating to this type of impact.

7.3 Place the test specimen in the trough below the chute. Insert the retaining board in the top part of the chute and hold it in place across the chute opening with a suitable metal or wood clip. Weigh 16 ± 0.2 kg (35 ± 0.5 lb) of stones into the bucket. Lift the bucket to the top of the chute. Empty the stones into the trough formed by the hopper walls and the retaining board so that the stones are distributed evenly in the trough. Detach the holding clip from the

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

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[2] *Annual Book of ASTM Standards,* Vol 06.02.

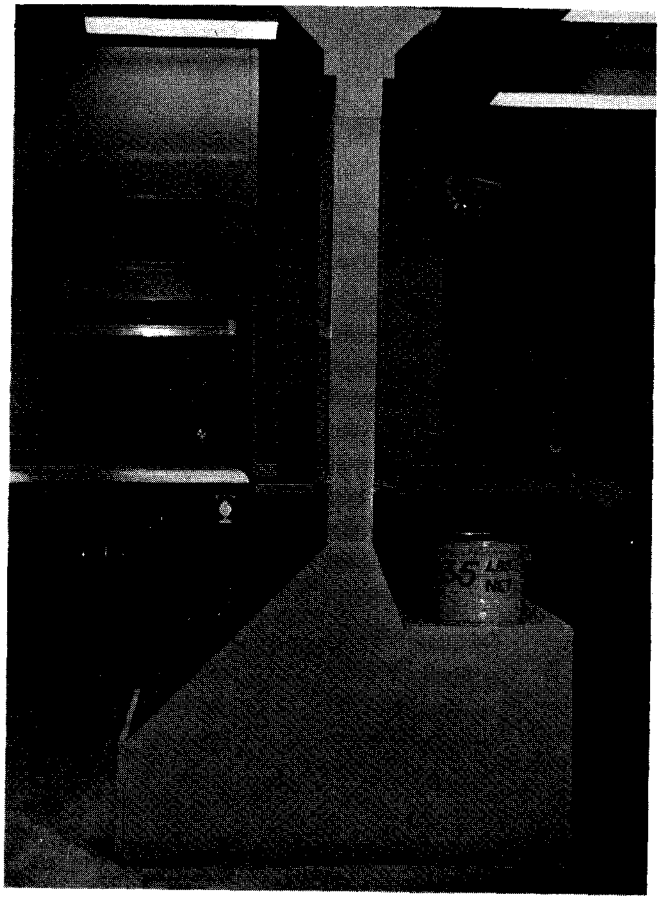
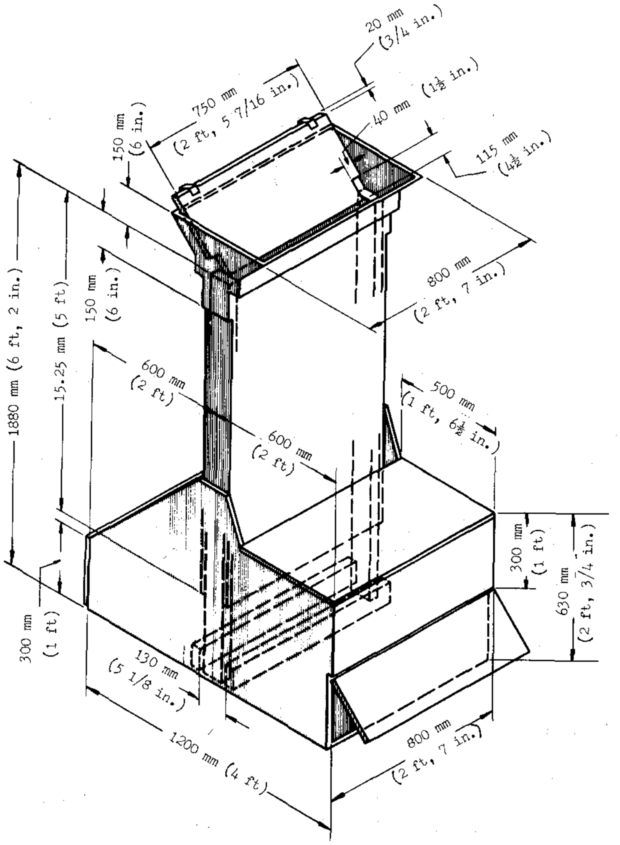


FIG. 1 Box, Chute, and Bucket



NOTE—Framing lumber 40 by 90 mm (2 by 4 in.); all other material 20 mm (¾ in.).

FIG. 2 Construction Details of Box and Chute

retaining board and tilt the board forward to release the stones. Experience with this procedure has indicated that the stones are so constricted within the narrow confines of the chute that they fall in a uniform manner.

7.4 After each drop, remove the specimen. Examine the specimen visually for breaks in the coating. If there are no visual breaks, check with the holiday detector appropriate for the coating thickness for holidays in the coating. If there is no visual or electrical evidence of holidays, repeat the test until the first holiday is detected and record the number of drops and the number of holidays detected. In repeating the test, the specimen should be replaced in the trough so that the same portion of the coated surface is always subjected to the falling stones. After the same stones have been used five times, they should be replaced.

7.5 If no failure has occurred after 10 buckets, terminate the test.

NOTE 4—As indicated in 7.4, the same charge of stones may be used five times. Experiments in which the same charges of stones were dropped more than five times have shown relatively little attrition of the stones.

## 8. Report

8.1 The report shall include the following:

8.1.1 Complete identification of the specimen including: name and code number of the coating, pipe diameter, coating thickness, source, production data, and production-run number,

8.1.2 Approximate pipe temperature at the beginning and at the end of the test, and

8.1.3 Number of buckets of stone to produce the first holiday and the number of holidays produced during this drop.

NOTE 5—If there is a large number of holidays after a given number of drops, the report should state this by the term "too numerous to count."

**Designation: G 14 - 88**

# Standard Test Method for Impact Resistance of Pipeline Coatings (Falling Weight Test)[1]

This standard is issued under the fixed designation G 14; 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 energy required to rupture coatings applied to pipe under specified conditions of impact from a falling weight.

1.2 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The values stated in SI units to three significant decimals are to be regarded as the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

G 62 Test Methods for Holiday Detection in Pipeline Coatings[2]

2.2 *SAE Standard:*

Grade 52100 Steel[3]

## 3. Summary of Test Method

3.1 This test method uses a falling fixed weight having a specified diameter impact surface, *tup*, which is restrained vertically and dropped from varying heights to produce impact energies over the required range. Electrical inspection is used to detect resultant breaks in the coating. Impact resistance is determined as the amount of energy required to cause penetration of the coating film.

## 4. Significance and Use

4.1 The ability of a pipe coating to resist mechanical damage during shipping, handling, and installation will depend upon its impact resistance. This test method provides a systematic means for screening coating materials with regard to this property.

## 5. Apparatus

5.1 This test method can be successfully used with impact apparatus conforming to the following specifications:

5.1.1 *Tup*—The tup shall be made up from a tup body and a tup nose having a combined, fixed weight of 1.361 kg (3.00 lb) and shall be used over a drop range of 0.61 to 1.22 m (2 to 4 ft). With most coatings, a 1.361-kg (3.00-lb) tup dropped through a distance of 914 mm (3 ft) yields suitable results. The tup nose shall have a 15.875-mm (⅝-in.) hemispherical head.

NOTE 1—Frequent replacement of the tup nose can be avoided if it is cut from steel capable of being hardened to a hardness of Rockwell C/45 while retaining an impact toughness of at least 15 ft·lb (20.34 J). Ball bearings conforming to SAE Grade 52100 have also been found suitable for this purpose.

5.1.2 *Drop Tube*—A tube 1.52 m (5 ft) long shall be used to contain the tup and guide it during free fall. The drop tube shall be constructed of steel, aluminum, or any other suitably rigid material and internally sized to provide a minimum of friction to the falling tup. A scale shall be attached for measuring the height of drop to the nearest 2.54 mm (0.10 in.).

5.1.3 *Specimen Holder*—The base plate of the apparatus shall include a device for positioning and holding the pipe specimen on line with the axis of the vertical drop tube.

NOTE 2—An arrangement using a V-notch vise made of metal with spring clamp is recommended for this purpose. Glancing blows, caused by an out-of-plumb condition between drop tube and pipe sample, will cause erratic test results.

5.1.4 *Apparatus Support*—Both the apparatus and sample shall be firmly supported and secured to a rigid base to optimize energy transfer from the tup to the specimen.

5.2 A design for the test apparatus appears in Figs. X1.1 through X1.3 of Appendix X1.

5.3 *Thickness Gage*—Measurements of coating thickness will be required for this test, and shall be done in accordance with Method G 12.

5.4 *Holiday Detector*—A suitable detector as specified in Test Methods G 62 shall be used to locate breaks in the coating film.

## 6. Test Specimen

6.1 The test specimen shall be a 406.4 mm (16 in.) long piece of Schedule 40, 60.325 mm (2.375 in.) outside diameter coated pipe prepared with its surface preparation and coating procedures equivalent to that of production coated pipe.

6.2 Seven specimens shall be required for the test.

## 7. Conditioning

7.1 The specimen shall be exposed to a room temperature of 21 to 25°C (70 to 77°F) for a period of 24 h before beginning the test.

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

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[2] *Annual Book of ASTM Standards*, Vol 06.02.

[3] Available from Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.

## 8. Preliminary Measurements

8.1 Measure the applied coating thickness of each specimen in accordance with Test Method G 12.

8.2 Place test specimen in sample holder and lightly place tup on surface of the coating. Adjust either the drop tube or the attached scale so that the wing bolt (lifting pin) is at the zero mark of the scale.

8.3 Make a preliminary set of impact readings to determine the approximate starting point for the test. This shall be done by striking the first specimen from a height sufficient to cause failure of the coating film. Consider any penetration a failure if it is detectable with a suitable Holiday Detector as specified in Test Methods G 62.

8.3.1 Reduce the height by 50 % and make a second exploratory drop at a fresh area on the pipe surface. Continue testing in this manner, with the corresponding reduction in height between drops, until the coating fails to break.

NOTE 3—Choose test locations at the specimen surface in a random manner and keep at least a 76.2-mm (3-in.) distance between adjacent points of impact and within 38 mm (1½ in.) from the ends. Choosing test points in any regular pattern will bias the experiment and introduce error into the test results.

8.3.2 Repeat the test at the height immediately preceding the occurrence of the nonfailure to determine if an approximate level for the mean impact strength has been bracketed. Two successive reversals of coating performance between failure and nonfailure will give sufficient indication that the point has been reached.

## 9. Procedure

9.1 Perform the test at a room temperature of 21 to 25°C (70 to 77°F).

9.2 Begin testing from the approximate height determined in 8.2 and corresponding to the point at which the first nonfailure was registered. Maintain a fixed increment between adjacent testing heights.

9.3 Use a suitable detector, as specified in Test Method G 6, to determine penetration or lack thereof of the coating after each individual impact.

9.4 If the coating film is penetrated on the initial drop, make the next test at the next lower height increment. If the first specimen does not fail, make the second test at the next higher increment.

9.5 In a similar manner, determine the height of fall by the performance of the coating on each preceding drop. Maintain a constant height increment between readings. Continue to apply this "up-and-down" method[4] until 20 successive impact readings have been made.

## 10. Calculation

10.1 Calculate the mean value of impact strength, *m*, in g/cm (or in./lb) as follows:

$$m = \left[h_0 + d\left(\frac{A}{N} \pm \frac{1}{2}\right)\right] \times W$$

where:

$h_0$ = minimum height at which the less frequent event occurs, cm (or in.),

$d$ = increment in height of drop, cm (or in.),

$A$ = sum of the frequency of occurrence at each height increment times the number of increments above the $h_0$ value for each observation in the $N$ total,

$N$ = total number of the *less* frequent event (coating failures or nonfailures), and

$W$ = tup weight, g (or lb).

NOTE 4—The minus sign is used when the calculation of the mean is based on the total number of coating failures and the plus sign when it is based on the nonfailures.

10.2 Calculate the sample standard deviation, *s*, in gram-centimetres (or inch-pounds) as follows:

$$S = 1.620\, dW\left(\frac{(NB - A^2)}{N^2} + C\right)$$

where:

$C$ = 0.737 when $d$ is in cm, or 0.029 when $d$ is in in.,

$d$, $N$, $A$, and $W$ are as defined in 10.1, and

$B$ = sum of the frequency of occurrence at each height increment times the square of the number of increments above the $h_0$ value for each observation in the $N$ total.

NOTE 5—Adequate definition of coating impact strength will result only if the height increment, *d*, has been properly chosen. When testing polymeric films in the range from 0.254 to 1.016 mm (0.010 to 0.040 in.), height increments of from 5.1 to 12.7 mm (0.2 to 0.5 in.) have been found suitable with the 1.361-kg (3.0-lb) tup. Larger increments may be necessary for thicker materials. If after making the calculations of 10.1 and 10.2, the ratio of the height increment to the standard deviation ($d/s$) is less than 0.20, the test should be repeated using a larger value for the increment *d*. This procedure will result in an improved estimate of the coating impact strength.

10.3 An illustration of the use of these equations appears in Appendix X2.

## 11. Report

11.1 The report shall include the following:

11.1.1 Complete identification of the specimen including:

11.1.1.1 Name and code number of the coating,

11.1.1.2 Size of pipe,

11.1.1.3 Source, production date, and production-run number,

11.1.1.4 Minimum, maximum, and average coating thickness,

11.1.1.5 Date of test, and

11.1.1.6 Other information that may be pertinent,

11.1.2 Average impact strength in g/cm (or in./lb),

11.1.3 Sample standard deviation in g/cm (or in./lb).

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[4] This system of testing, nomenclature, and calculation is described by Dixon, W. J. and Mood, A. M., "A Method for Obtaining and Analyzing Sensitivity Data," *Journal Am. Statistical Assn.*, JSTNA, Vol 43, March, 1948, p. 109.

# APPENDIXES

## (Nonmandatory Information)

## X1. SUGGESTED DESIGN FOR TEST APPARATUS

X1.1 A design for the impact test apparatus is shown in Figs. X1.1 to X1.3.

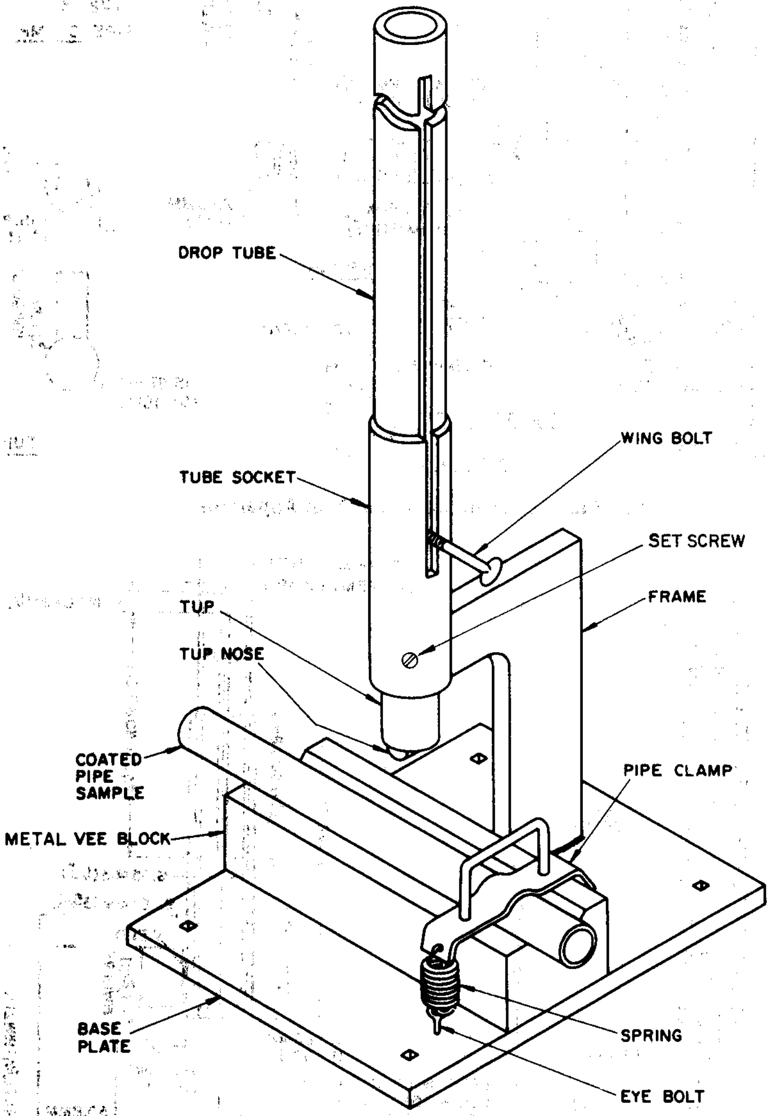

FIG. X1.1 Test Apparatus Assembly

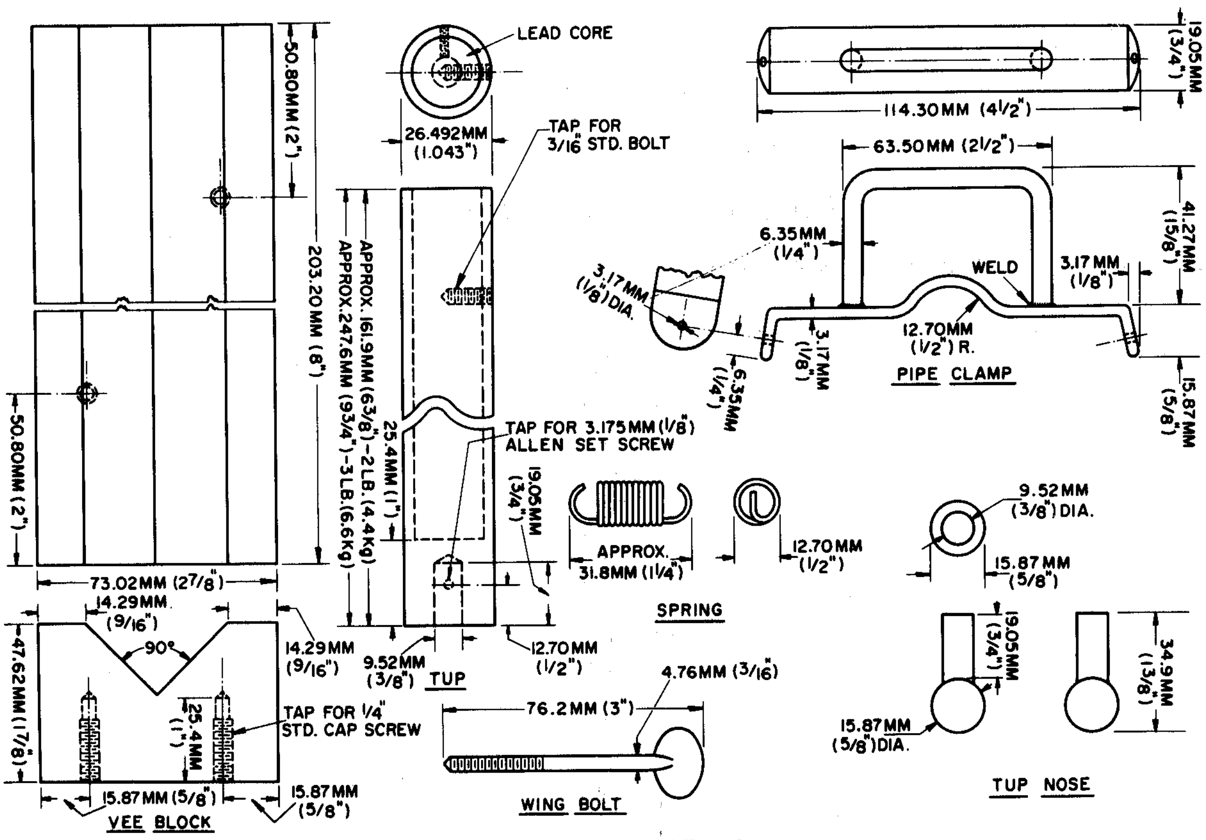

FIG. X1.2 Detailed Design of Test Apparatus

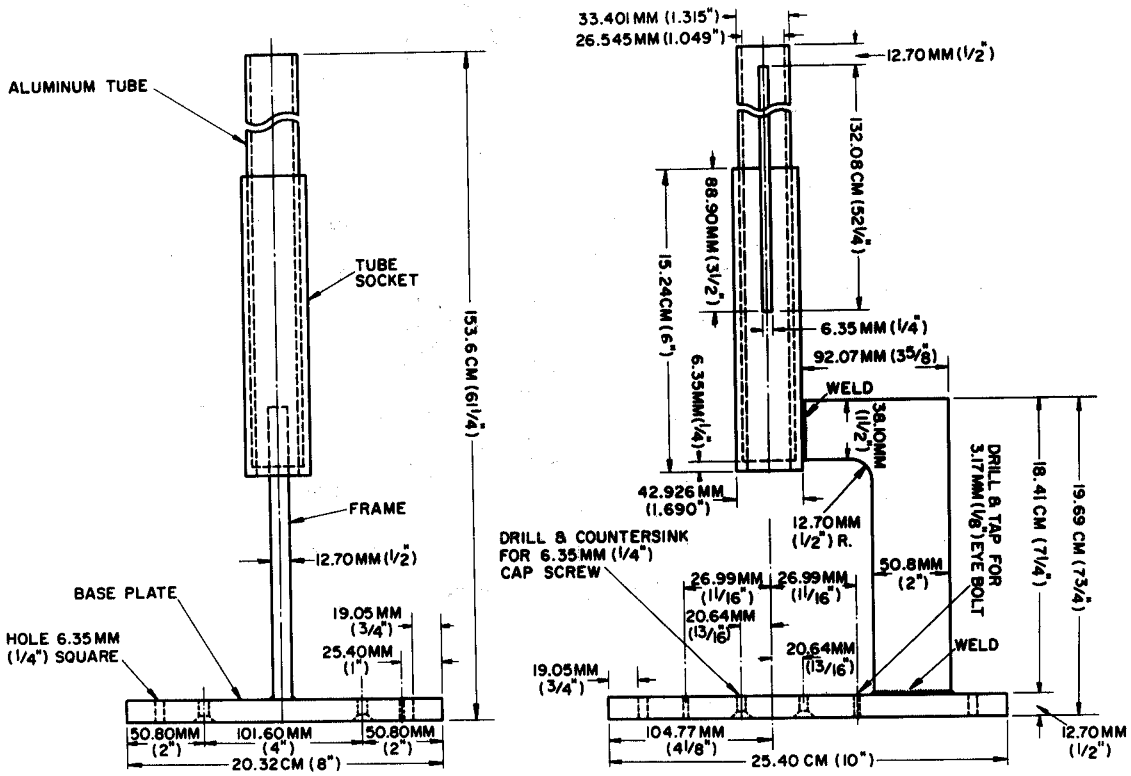

FIG. X1.3 Detailed Design of Test Apparatus

## X2. SAMPLE CALCULATIONS

X2.1 Test results for 20 drops with a 1.361-kg (3-lb) tup are given in Table X2.1.

**TABLE X2.1 Test Results**

| Test No. | Height of Drop, in. | Failed | Test No. | Height of Drop, in. | Failed |
|---|---|---|---|---|---|
| 1 | 14.1 | yes | 11 | 13.5 | no |
| 2 | 13.8 | no | 12 | 13.8 | no |
| 3 | 14.1 | no | 13 | 14.1 | yes |
| 4 | 14.4 | yes | 14 | 13.8 | yes |
| 5 | 14.1 | yes | 15 | 13.5 | no |
| 6 | 13.8 | no | 16 | 13.8 | yes |
| 7 | 14.1 | no | 17 | 13.5 | yes |
| 8 | 14.4 | yes | 18 | 13.2 | no |
| 9 | 14.1 | yes | 19 | 13.5 | no |
| 10 | 13.8 | yes | 20 | 13.8 | yes |

Height increment = 0.3 in.
Failures = 11
Nonfailures = 9
Nonfailures at 13.2 in. ($h_0$) = 1; at 13.5 in. = 3; at 13.8 in. = 3; at 14.1 in. = 2
$A = (0 \times 1) + (1 \times 3) + (2 \times 3) + (3 \times 2) = 15$
$B = ((0)^2 \times 1) + ((1)^2 \times 3) + ((2)^2 \times 3) + ((3)^2 \times 2) = 33$

$$m = \left[13.2 + 0.3\left(\frac{15}{9} + \frac{1}{2}\right)\right] 3.0 = 41.55 \text{ in.} \cdot \text{lb}$$

mean impact resistance = 41.55 in.·lb

$$S = 1.620 \times 0.29 \times 3.0 \left(\frac{9 \times 33 - (15)^2}{(9)^2}\right) = 1.338 \text{ in.} \cdot \text{lbs.}$$

Sample standard deviation = 1.383 in.·lb

Designation: G 17 – 88 (Reapproved 1992)$^{\epsilon 1}$

# Standard Test Method for Penetration Resistance of Pipeline Coatings (Blunt Rod)[1]

This standard is issued under the fixed designation G 17; 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 ($\epsilon$) indicates an editorial change since the last revision or reapproval.

$^{\epsilon 1}$NOTE—Section 11 was added editorially September 1992.

## 1. Scope

1.1 This accelerated test method is used to determine the relative resistance of steel pipeline coatings to penetration or deformation by a blunt rod under a specified load. The test method is intended to apply to the testing of all types of nonmetallic pipeline coatings subjected to various temperatures.

1.2 *This standard does not purport to address all of the safety problems, 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.*

1.3 The values stated in SI units to three significant decimals are to be regarded as the standard.

## 2. Summary of Test Method

2.1 The depth or rate of penetration or deformation that is caused by a weighted blunt rod to a coating system applied to steel pipe is measured over a period of time with a micrometer depth gage. Three consecutive identical readings taken at specified intervals conclude the test.

## 3. Significance and Use

3.1 Since pipeline coatings are subjected to concentrated pressures exerted by the weight of the pipe and the backfill, this test is intended primarily for determining the resistance of the coating to penetration or deformation under controlled conditions.

3.1.1 Deformation of a compressible coating does not signify a potential weakness in underground burial service, if the coating is not penetrated. Deformation cannot be compared to penetration into and through a coating.

## 4. Apparatus

4.1 Test apparatus shall consist of a dead weight tester that can press the flat tip of a rod against the coated pipe. The flat-tipped end of the rod contacting the coating shall have a diameter of 6.350 mm ± 0.0254 mm (0.250 in. ± 0.001 in.) and together with supplementary weight and any other weight-contributing parts shall have a total weight of 4.453 kg (9.817 lb), resulting in a unit pressure of 14.060 kg/cm$^2$ (200 psi) against the coating. Detailed design of the apparatus is shown in Fig. 1 through 6.

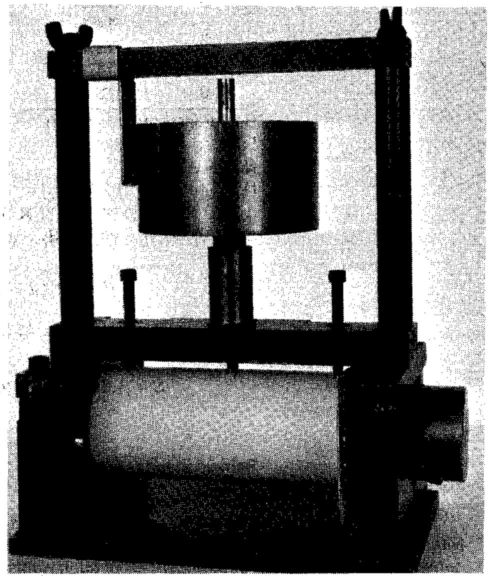

FIG. 1 Blunt Rod Penetration Tester

4.2 *Depth Gage*, a device capable of measuring the penetration movement of the rod accurately to 0.0254 mm (0.001 in.).

4.3 *Temperature Chamber*, a thermostatically controlled enclosure to provide heating and cooling of the specimen and test apparatus (excluding depth gage) to within ±2°C (3.6°F) of the desired test temperature. This equipment is optional when the test can be run within ±2°C (3.6°F) of the desired test temperature without its use.

4.4 *Temperature Controlled Box*, to fully enclose the test apparatus except the indicating means of the depth gage, as optional equipment in cases where a test is to be run at a temperature higher than room temperature. The enclosure shall be thermostatically controlled, capable of maintaining the temperature to within ±2°C (3.6°F) of the test temperature, which normally will be in the range of 23 to 76.6°C (73.4 to 170°F).

4.5 *Thermometer*, a temperature measurement device accurate to ±0.5°C (±1°F).

## 5. Test Specimens

5.1 The test specimens shall be 150 mm (6 in.) long and prepared with its surface preparation and coating procedures

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on the Durability of Pipeline Coatings and Linings.

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**TABLE 1 Dimensions for Fig. 3**

| Symbol | mm | in. |
|---|---|---|
| A | 1.59 | 0.063 |
| B | 3.18 | 0.125 |
| C | 7.94 | 0.313 |
| D | 8.73 | 0.343 |
| E | 9.53 | 0.375 |
| F | 10.32 | 0.406 |
| G | 12.70 | 0.500 |
| H | 15.86 | 0.625 |
| I | 19.05 | 0.750 |
| J | 20.64 | 0.813 |
| K | 25.40 | 1.000 |
| L | 38.10 | 1.500 |
| M | 55.56 | 2.188 |
| N | 63.50 | 2.500 |
| O | 76.20 | 3.000 |
| P | 111.13 | 4.375 |
| Q | 127.00 | 5.000 |
| R | 184.15 | 7.250 |
| S | 215.90 | 8.500 |
| T | 50.80 | 2.000 |
| U | 88.90 | 3.500 |

equivalent to that of production coated pipe. Duplicate tests shall be run.

## 6. Conditioning

6.1 The specimen shall be exposed to the test temperature for a period of 24 h before beginning the test. If the test temperature is the same as room temperature, it shall be 21° to 25°C (70° to 77°F).

**TABLE 2 Dimensions for Fig. 4**

| Symbol | mm | in. |
|---|---|---|
| A | 0.79 | 0.031 |
| B | 1.59 | 0.063 |
| C | 3.99 | 0.157 |
| D | 11.11 | 0.438 |
| E | 12.70 | 0.500 |
| F | 12.70 | 0.500 |
| G | 12.71 | 0.500 |
| H | 12.72 | 0.501 |
| I | 13.49 | 0.531 |
| J | 14.29 | 0.563 |
| K | 15.88 | 0.625 |
| L | 17.46 | 0.688 |
| M | 19.05 | 0.750 |
| N | 19.06 | 0.751 |
| O | 25.40 | 1.000 |
| P | 38.10 | 1.500 |
| Q | 60.33 | 2.375 |
| R | 92.08 | 3.625 |
| S | 101.60 | 4.000 |
| T | 120.65 | 4.750 |
| U | 141.29 | 5.563 |
| V | 184.15 | 7.250 |
| W | 203.20 | 8.000 |

## 7. Procedure

7.1 Perform the test at room temperature or at the selected temperature.

7.2 Accurately measure the thickness coating in the area to be tested. Assemble the sample and end plates. Position the sample in the test unit so that the known film thickness

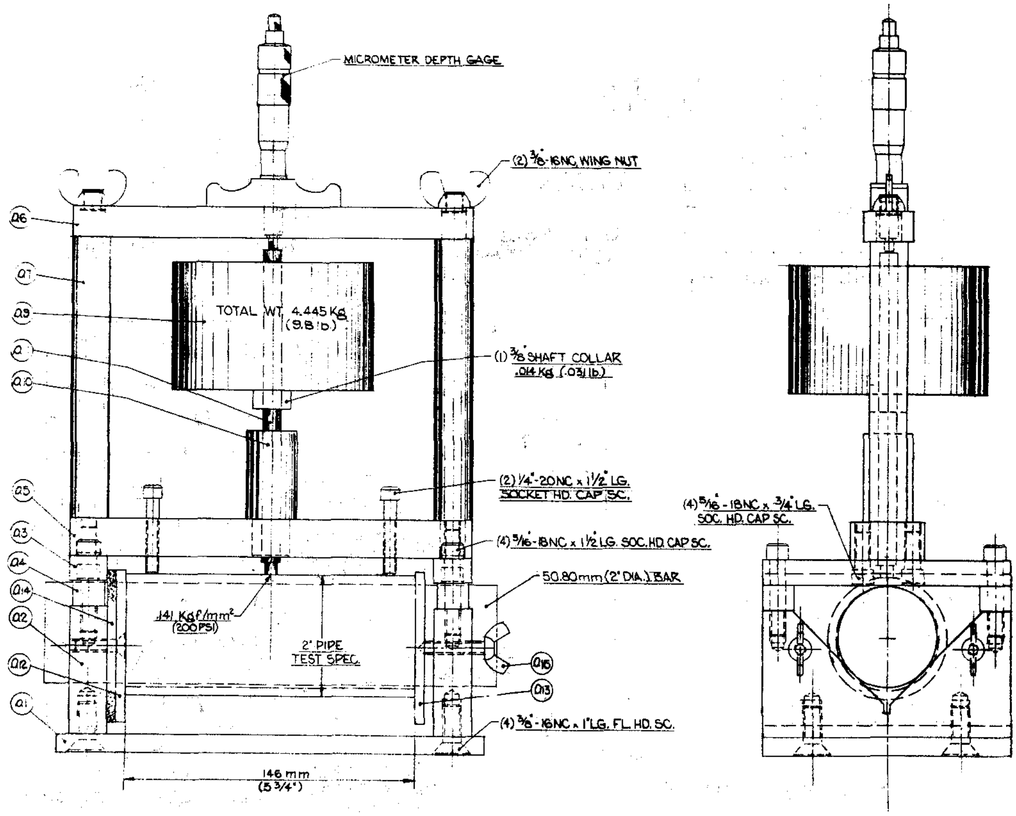

FIG. 2 Blunt Rod Penetration Tester, Assembly

**TABLE 3 Dimensions for Fig. 5**

| Symbol | mm | in. |
|---|---|---|
| A | 1.59 | 0.063 |
| B | 6.35 | 0.250 |
| C | 6.35 | 0.250 |
| D | 9.50 | 0.374 |
| E | 9.53 | 0.375 |
| F | 9.55 | 0.376 |
| G | 10.32 | 0.406 |
| H | 19.05 | 0.750 |
| I | 19.06 | 0.750 |
| J | 19.07 | 0.751 |
| K | 25.40 | 1.000 |
| L | 64.29 | 2.531 |
| M | 63.50 | 2.500 |
| N | 101.60 | 4.000 |
| O | 165.10 | 6.500 |

area is directly under the weighted rod and hold the sample in position by use of the cap screws.

7.3 Loosen the shaft collar and allow the weight to rest on the shaft guide. Using the depth gage take a measurement without the load applied. Raise the weight and tighten the set screw in the collar so that the weight rests on the shaft.

7.4 Take measurements every 24 h.

7.5 Continue the test until the maximum penetration has been reached as evidenced by three consecutive unchanged measurements.

## 8. Calculation

8.1 Calculate the net change and the percent change as follows:

**TABLE 4 Dimensions for Fig. 6**

| Symbol | mm | in. |
|---|---|---|
| A | 4.76 | 0.188 |
| B | 6.35 | 0.250 |
| C | 7.14 | 0.281 |
| D | 13.49 | 0.531 |
| E | 38.10 | 1.500 |
| F | 44.45 | 1.750 |
| G | 52.38 | 2.062 |
| H | 63.50 | 2.500 |
| I | 76.20 | 3.000 |
| J | 88.90 | 3.500 |
| K | 127.00 | 5.000 |
| L | | |

$$C_N = M_F - M_I$$
$$C_P = (C_N/T) \times 100$$

where:

$C_N$ = net change,
$M_F$ = final measurement,
$M_I$ = initial measurement,
$C_P$ = percent change, and
$T$ = film thickness.

## 9. Report

9.1 The report shall include the following:

9.1.1 Complete identification of specimens, including name and code number of coatings, size of the pipe, source, production date, production run number, and any other information that may be pertinent to identification,

9.1.2 Film thickness of the specimen at the test location in millimetres (inches),

9.1.3 Temperature at which the test was conducted,

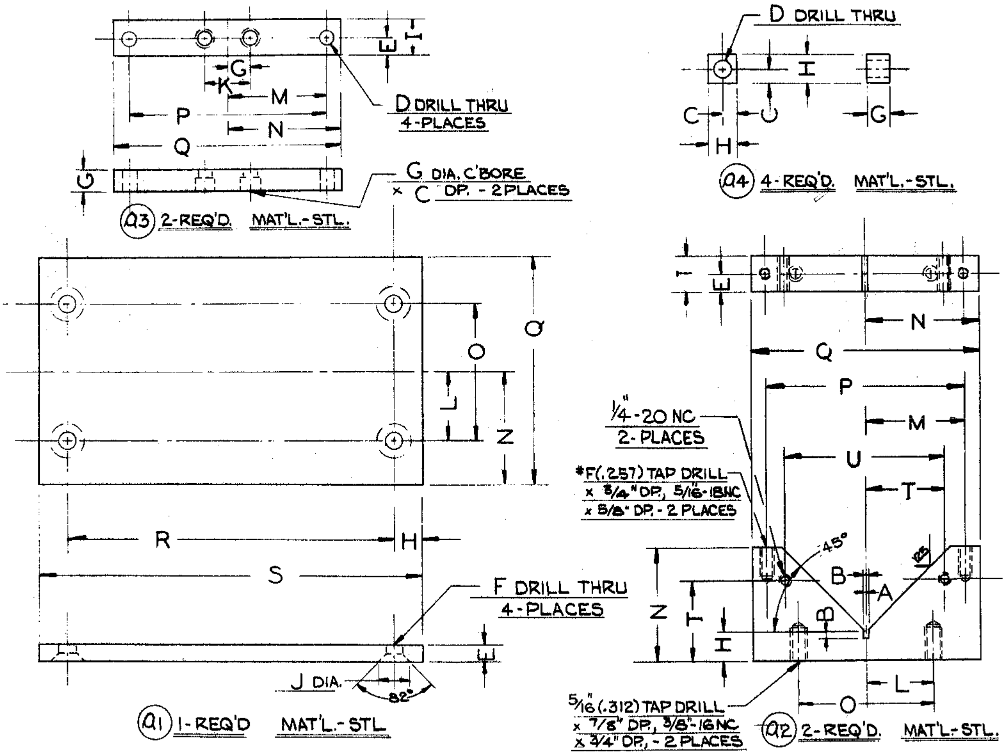

FIG. 3

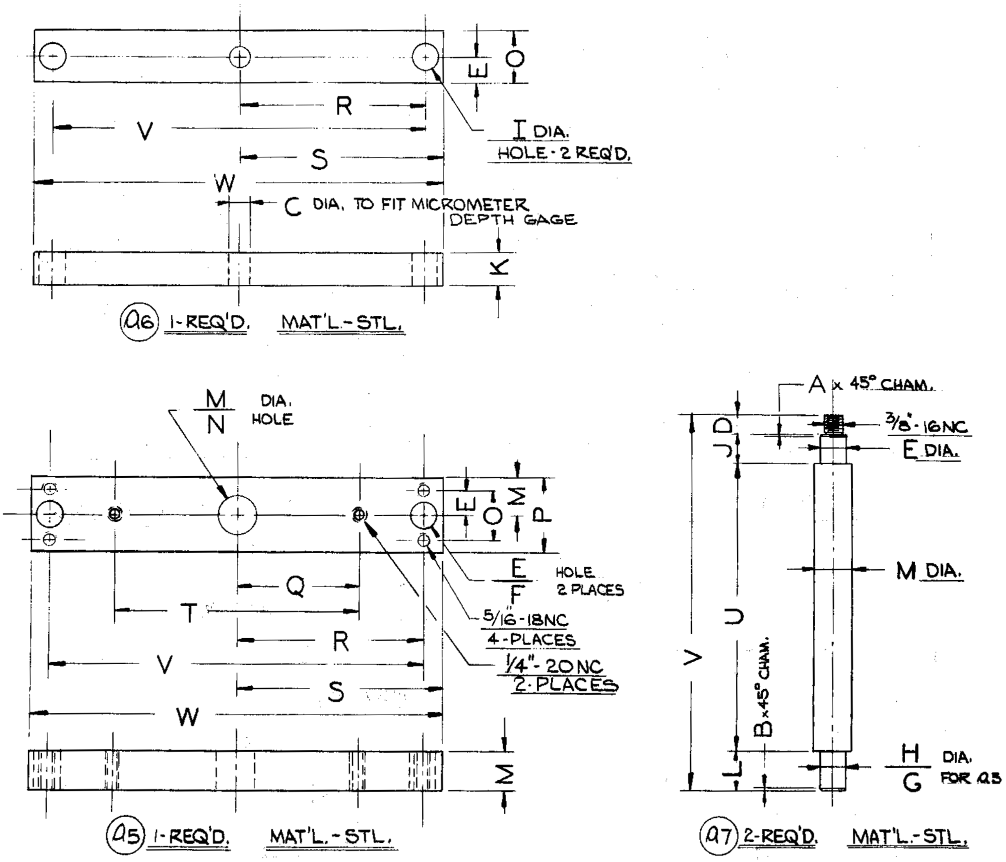

FIG. 4

9.1.4 Final and initial measurements in millimetres (inches),

9.1.5 Whether the weighted rod has caused penetration or deformation,

9.1.6 Net penetration or deformation in millimetres (inches) and the percent penetration or deformation, and

9.1.7 Number of hours or days to net penetration or deformation.

## 10. Precision

10.1 *Precision Data* are limited to two adjacent specimens taken from the same source coated pipe and assume that the coating process was uniform with respect to pipe surface condition and coating material. Specimens that were not

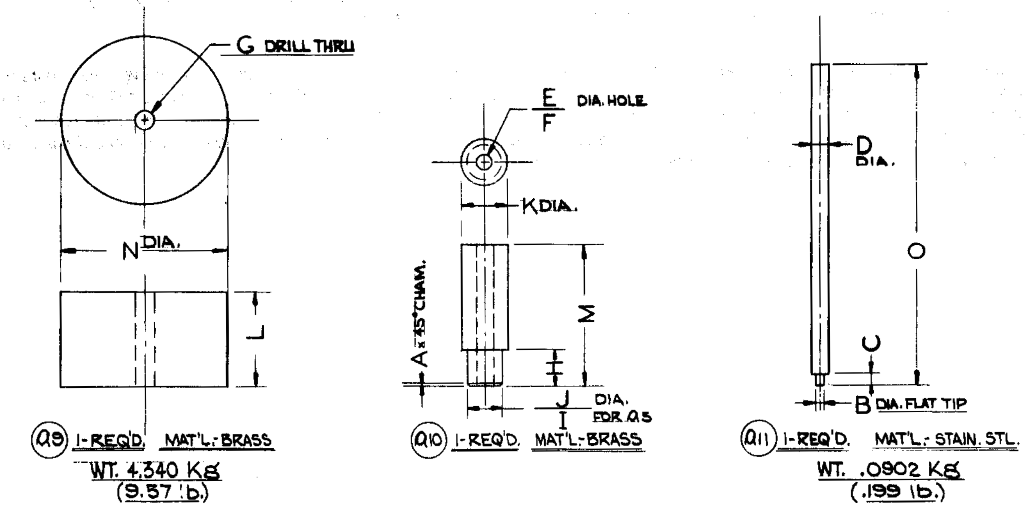

FIG. 5

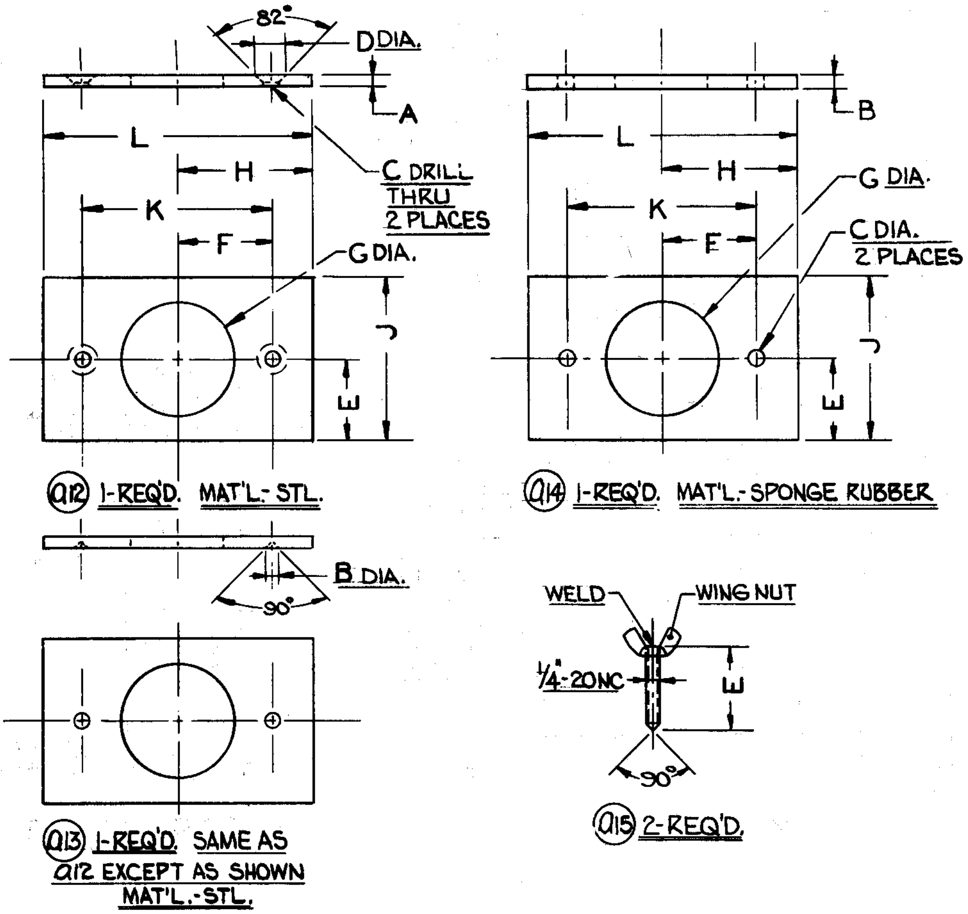

FIG. 6

adjacent in the as-produced condition or were taken from different lengths of pipe may represent differing process conditions.

10.2 *Repeatability*—Initial and final measurements should be ±0.0127 mm (0.0005 in.) or 5 %.

10.3 *Reproducibility*—The results reported by one laboratory should not differ from those of another laboratory by more than ±5 % of the net penetration.

## 11. Keywords

11.1 blunt rod; coating; compression; deformation; load; penetration; pipeline; resistance

# Standard Test Method for Joints, Fittings, and Patches in Coated Pipelines[1]

This standard is issued under the fixed designation G 18; 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.

$^{\epsilon 1}$ NOTE—Section 15 was added editorially in September 1992.

## 1. Scope

1.1 This test method describes determination of the comparative corrosion preventative characteristics of materials used for applications to joints, couplings, irregular fittings, and patched areas in coated pipelines. The test method is applicable to materials whose principal function is to act as barriers between the pipe surface and surrounding soil environment.

1.2 The test method described employs measurements of leakage current, capacitance, and dissipation factor to indicate changes in the insulating effectiveness of joint and patching materials.

1.3 *This standard does not purport to address all of the safety problems, 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 The values stated in SI units to three significant decimals are to be regarded as the standard.

## 2. Referenced Document

2.1 *ASTM Standard:*

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

## 3. Summary of Test Method

3.1 The test method consists of an immersion test where coated pipe specimens, each containing a simulated joint, tee, or patched area, are suspended in an electrolyte and placed under cathodic protection by connecting the specimens to the negative (−) terminal of a 6-V d-c power supply (see Fig. 1). An anode, also immersed in the electrolyte and connected to the positive (+) terminal of the power supply, completes the test circuit. Joint or patch performance is followed through periodic determinations of leakage current measured as voltage drops across a calibrated resistor in the anode-to-cathode circuit.

3.2 Capacitance and dissipation factor measurements are used to supplement the periodic leakage current determinations.

## 4. Significance and Use

4.1 The exposed metal surfaces at joints, fittings, and damaged areas in an otherwise coated pipeline will be subjected to corrosion if allowed to come in contact with the soil environment. The performance of joint and patching materials designed to function as protective coverings will depend upon such factors as the ability of the material to bond to both the pipe coating and exposed metal surfaces, the integrity of the moisture seal at lapped joints, and the water absorption characteristics of the joint material.

4.2 The existence of substantial leakage current through the coating joint, patch, or fitting is reliable evidence that the material has suffered a significant decrease in its performance as a protective barrier. In a similar manner, measured changes in joint capacitance and dissipation factor are useful because they are related to the water absorption rate of the joint material. Water permeating an insulating barrier increases its capacitance and its progress can be measured through the use of a suitable impedance bridge.

## 5. Apparatus

5.1 *Test Vessel*, nonconducting, shall be used to contain the test specimens. Dimensions of the vessel shall permit the following requirements:

5.1.1 The test vessel shall be large enough to allow for suspension of the specimens in a vertical position and equidistant from a centrally located anode. The specimens shall not touch either each other, the walls, or bottom of the test vessel.

5.1.2 The test vessel shall be deep enough to allow for immersion of the specimens in the electrolyte to the lower edge of the upper moisture shield (see Fig. 2).

NOTE 1—A commercially available, 42 L (11-gal) waste container of high-density polyethylene can be conveniently used as a test vessel and will accommodate up to six test specimens of a size indicated in 7.2.

5.2 *Support Plate*, fabricated from a nonconductive material, to suspend the specimens in the test vessel. The support plate shall contain an access hole for the reference electrode. A typical test cell is illustrated in Fig. 3.

5.3 *Anode*, fabricated from 9.525 mm (0.375-in.) diameter 300 series stainless steel rod, 609.6 mm (24.00 in.) long. Other inert anodes such as carbon or platinum may be used.

5.4 *D-C Voltmeter*, to serve the dual purpose of (*1*) measuring leakage current as a potential drop across a 1,000-Ω shunt in the measuring circuit and (*2*) measuring the potential of the test specimen with reference to a Cu-CuSO$_4$ half cell. The instrument characteristics for these functions shall be:

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved Sept. 30, 1988. Published November 1988. Originally published as G 18 – 71 T. Last previous edition G 18 – 83.

[2] *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

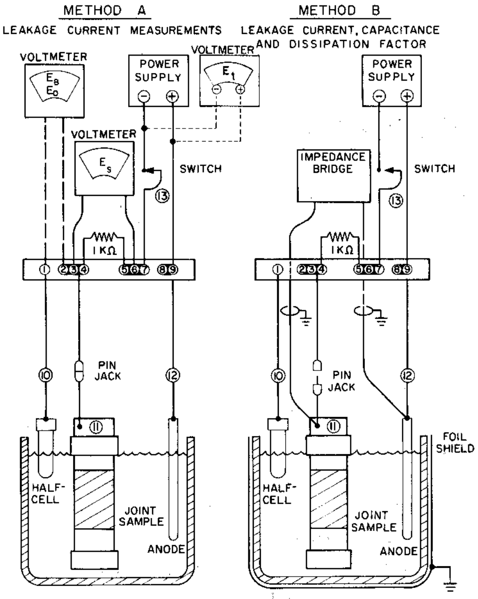

**FIG. 1 Test Circuits**

5.4.1 *Voltage Range*—50 µV full scale to 10 V full scale in overlapping 1× and 3× ranges.

5.4.2 *Accuracy*—±3 percent of full scale on all ranges.

5.4.3 *Input Resistance*—Greater than 10 MΩ on all ranges.

5.5 *Thickness Gage*—Measurements of coating thickness will be required for this test. Any instrument suitable for use with Test Method G 12[2] can be used. However, the choice of measuring gage shall be compatible with the joint coating thickness that will be encountered in the test.

5.6 *Ohmmeter*—Measurements for end-cap integrity shall be made with a suitable ohmmeter capable of reading resistance to an upper limit of 1000 MΩ ± 5 %.

5.7 *Reference Electrode*—A $Cu-CuSO_4$ half cell of conventional glass or plastic tube with porous plug construction, but preferably not over 19.05 mm (¾ in.) in diameter, having a potential of −0.316 V with respect to the standard hydrogen electrode.

NOTE 2—A saturated calomel half cell may be used, but measurements made with it shall be converted to the $Cu-CuSO_4$ reference for reporting by adding −0.072 V to the observed reading.

5.8 *Voltage Source*—A battery or rectifier-type power supply shall be used to maintain a potential difference of 6.0 ± 0.1 V dc between each of the test specimens and the $Cu-CuSO_4$ half cell. Where multiple specimens are tested, a suitable voltage-dividing circuit will be required for individual control of the voltage applied to each specimen.

5.9 *Circuit Wiring* from the anode to specimen shall be of No. 18 Awg insulated copper. A switch for disconnecting each specimen from its voltage source shall be included in the circuit. A 1000 Ω ± 1 percent, 1-W (minimum) precision resistor shall be placed in the anode-to-cathode circuit as a shunt for current. A diagram illustrating the test cell wiring appears as Fig. 1.

5.10 *Capacitance Bridge*—Measurements of specimen capacitance and dissipation factor shall be made with a low-voltage a-c, resistive ratio arm-type bridge[3] having the following characteristics:

5.10.1 *Oscillator Frequency*, 1 kHz ± 2 % tolerance.

5.10.2 *Series Capacitance Range*, 100 pF to 1100 pF accuracy ± 1 % ± 1 pF, whichever is larger.

5.10.3 *Dissipation Factor Range*, 0.002 to 1.0 at 1 kHz accuracy ± 5 % or ± 0.001 dissipation, whichever is larger.

5.11 *Connectors*—Miniature, pin-type, insulated jacks shall be used at the point of connection to each test specimen. The jacks serve two important functions: (*1*) they permit the disconnection of the specimen from the voltage source when the impedance bridge is in use, and (*2*) disconnection of the specimen from the test circuit also removes the effect of stray capacitance due to excessive lead length that may introduce error into impedance bridge measurements.

5.12 *Foil Shield*—As an additional safeguard against stray capacitance effects, wrap the entire test vessel in heavy-gage aluminum foil as shown in Fig. 3 and ground the shield.

## 6. Materials

6.1 *Electrolyte*—The electrolyte shall consist of potable tap water with the addition of 3 weight percent of technical-grade sodium chloride.

6.2 Materials for sealing the ends of the specimens may consist of waxes, epoxies, or other suitable materials. However, they should have a dielectric constant in the range from 2 to 6, and exhibit a low water-absorption rate. It is also important that the end-cap material maintains good adhesion to any coated pipe surfaces throughout the test period.

## 7. Test Specimen

7.1 The joint specimen shall be prepared from a representative piece of 60.325 mm (2.375 in.) outside diameter, Schedule 40, production-coated pipe.

7.2 Each piece of coated pipe shall be 381 mm (15.0 in.) long and serve as a carrier for the particular joint material to be tested.

7.3 The simulated coating joint shall be made by cleaning away a 101.6 mm (4.0-in.) band of pipe coating starting from a point 203.2 mm (8.0 in.) below the upper end of the pipe. The coating may be removed by power brushing or any other suitable device that will remove all of the coating in the area indicated and leave a clean metal surface behind.

7.4 The lower end of the test specimen shall be plugged flush with a stopper and sealed or capped with a material meeting the requirements of 6.2.

7.5 When used, the primer shall be applied to the test specimen in sufficient quantity to coat the test specimen from a point 88.9 mm (3.50 in.) below its upper end and ending at a point 317.5 mm (12.5 in.) from the upper end. This will allow for a 12.7-mm (0.5-in.) band of excess primer beyond each tape margin.

7.6 The joint material to be tested shall be applied to the prepared pipe specimen (using a spiral wrap for tapes) and starting from a point 101.6 mm (4.0 in.) below the upper end of the pipe section and ending at a point 304.8 mm (12.0 in.)

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[3] Instruments found to meet these requirements are the General Radio Type 1650-B Impedance Bridge and the Heath Co. Model IB-28 Impedance Bridge (Kit).

from the upper end. The 203.2 mm (8.0 in.) of joint material thus applied should overlap the bared section of pipe by 50.8 mm (2.0 in.) at each end. This distance represents the typical cut-back distance encountered in the field joining, through welding, of coated pipe in 60.325 mm (2.375 in.) outside diameter size. A diagram of the joint specimen appears as Fig. 2.

7.7 The manner of applying the joint material shall be done in accordance with the manufacturer's specifications. The supplier of the joint material should specify the desired time interval between the application of the material and the start of the test.

NOTE 3—Materials that are intended for use as a field-applied patch over damaged areas on coated pipelines can be tested using the same procedures, with the patching compound applied, instead, to the bared area of the simulated joint.

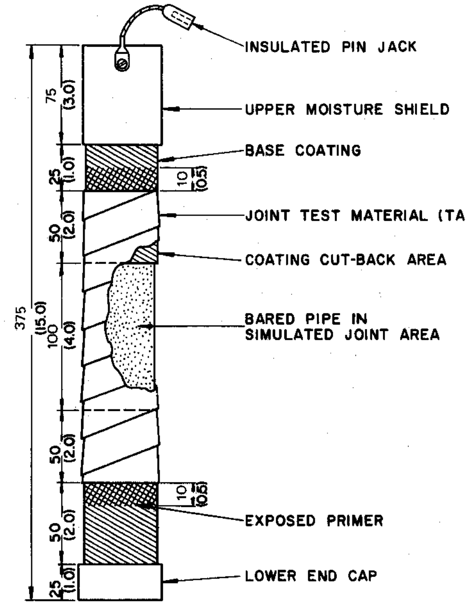
7.8 The upper 76.2 mm (3.0 in.) of the completed joint test specimen shall be coated with the material used for the lower end cap. This moisture shield can be conveniently made, in the case of some waxes and epoxies, with several successive brush or dip-applied applications. The thickness of the moisture shield should be approximately 3.175 mm (0.125 in.).

## 8. Testing Temperature

8.1 Perform all tests at a room temperature of 21 to 25°C (70 to 77°F).

## 9. Preliminary Test Measurements

9.1 *Coating and Joint Thickness*—Measure and record the thickness of the base coating which lies exposed at each end of the test joint. Measurements shall be made in accordance with Test Method G 12. In a like manner, measure and record the average thickness of the joint covering.



NOTE—All dimensions are in millimetres with inches in parentheses.

FIG. 2 Joint Test Specimen

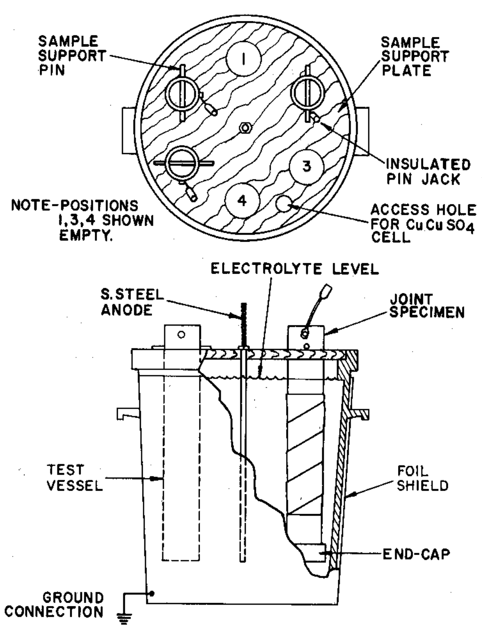


FIG. 3 Joint Test Cell

## 10. Procedure for Leakage Current Measurements

10.1 Suspend the joint test specimens in the test vessel, observing the clearances specified in 5.1.1 and 5.1.2. Fill the vessel with electrolyte, bringing the fluid level up to a point midway between the end cap and the lower edge of the joint material.

10.2 Connect one terminal of the ohmmeter to the test specimen and the other terminal to the central anode. The anode should be in contact with the electrolyte. Measure the apparent sample-to-anode resistance in ohms. The reading shall remain above 1000 MΩ for 15 min. Readings below this value probably indicates a faulty end-cap seal which should be repaired before the joint specimen is totally immersed.

10.3 Totally immerse the joint test specimens up to the lower edge of the moisture shield. This level shall be maintained throughout the test period by regular additions of tap water. The test shall be performed at room temperature.

10.4 Connect each joint test specimen in series with a 1000-Ω ± 1 % (1-W) resistor, a suitable switch, and the negative (−) terminal of the voltage source.

10.5 Connect the central stainless steel anode to the positive (+) terminal of the voltage source.

10.6 Energize the voltage source. Adjust the voltage to each specimen by connecting a voltmeter between each sample and the Cu-$CuSO_4$ half cell and varying the voltage output until 6.0 V are measured between sample and half cell (see Fig. 1).

10.7 As soon as the circuit is energized and voltage adjusted to each joint test specimen, measure and record the voltage across each 1000-Ω shunt resistor.

10.8 Measure and record the closed circuit potential, $E_B$, and open circuit potential, $E_o$, of each joint specimen with reference to the saturated Cu-$CuSO_4$ half cell.

NOTE 4—Any drop in closed circuit potential ($E_B$) of a joint specimen greater than 20 % of the terminal voltage ($E_t$) probably indicates a developing current leak in the joint area. In a like manner, any open circuit potential ($E_o$) greater than 0.05 V may indicate the presence of a developing conductive path across the test joint. (Both voltages are referenced to the Cu-CuSO$_4$ half cell.)

10.9 Using the d-c voltmeter in 5.4 measure and record the voltage appearing across the terminals of the voltage supply.

10.10 Continue, on a periodic basis, the measurements specified in 10.7 through 10.9. Calculate the apparent joint resistance by the method described in 12.1.

10.11 Prior to removal, reexamine a suspected joint failure to confirm that any observed drop in joint resistance is not due to a faulty end-cap seal. This can best be accomplished by removing the specimen from the test cell and allowing it to dry. When dry, reimmerse it to just above the level of the lower end cap and check the integrity of the cap by the method outlined in Section 8.

## 11. Procedure for Measurement of Capacitance and Dissipation Factor

11.1 The series capacitance and dissipation factor between each joint sample and the central stainless-steel anode shall also be measured. These measurements shall be made at the time of initial immersion and continued on an identical schedule with the voltage readings. They shall be made in the following manner:

11.2 Temporarily disconnect the test specimen from the voltage source. Connect the impedance bridge between the test specimen and stainless-steel anode. Energize the bridge and measure the equivalent series capacitance, *C*, and dissipation factor, *DF*, or the test specimen using a "null" technique to balance the measuring bridge.

11.3 A continued rise in joint capacitance and dissipation factor from the values recorded at the time of initial immersion, will indicate progressive absorption of water by the joint material.

11.4 The joint test shall continue for 180 days or until the joint resistance falls to a value of 4600 Ω for 1 m$^2$ (50 000 Ω for 1 ft$^2$) of immersed joint surface.

## 12. Calculations

12.1 *Joint Resistance*—Calculate the resistance, $R_j$, for the joint sample as follows:

$$R_j = 1000\,A[(E_B - E_o)/E_s]$$

where

$R_j$ = resistance, Ω/cm$^2$ (Ω/ft$^2$),
$A$ = immersed sample area (excluding end cap), cm$^2$ (ft$^2$),
$E_B$ = closed circuit potential of sample (reference Cu-CuSO$_4$), V (switch closed),
$E_o$ = open circuit potential of sample (reference Cu-CuSO$_4$), V (switch open), and
$E_s$ = voltage across 1000-Ω shunt resistor, V, (switch closed).

## 13. Report

13.1 All test reports shall include the following:

13.1.1 Complete identification of the joint specimen including:

13.1.1.1 Name and code number of the base coating,

13.1.1.2 Thickness of the base coating,

13.1.1.3 Name and code number of the joint material, including type (that is tape, two-part mix, etc.),

13.1.1.4 Method of application of joint material, including amount of overlap.

13.1.1.5 Where applicable, the width and thickness of the joint material (tapes),

13.1.1.6 Where applicable, the name, code number, and type of primer,

13.1.1.7 Where applicable, the method of application of primer,

13.1.1.8 Average thickness of the joint material, as applied, and

13.1.1.9 Dates of starting and terminating the test and other information that may be pertinent.

13.2 The following readings shall be recorded on a periodic basis:

13.2.1 Voltage across the 1000-Ω shunt resistor, $E_S$,

13.2.2 Closed-circuit potential of the joint specimen referenced to the Cu-CuSO$_4$ half cell, $E_B$,

13.2.3 Open-circuit potential of the joint specimen referenced to the Cu-CuSO$_4$ half cell, $E_o$,

13.2.4 Terminal voltage at the power supply, and

13.2.5 Calculated value of joint resistance, $R_j$.

13.2.6 Initial series capacitance of the joint sample, and

13.2.7 Initial dissipation factor of the joint sample.

NOTE 5—The frequency of experimental readings will depend upon the performance of each joint specimen. Specimens that show a high rate of change may require more frequent watching if the point of failure is to be recorded. Normally, weekly readings have been found to be adequate. Joint performance can be conveniently followed through plots of Joint Resistance versus Time and Dissipation Factor versus Time in semilogarithmic coordinates, and Capacitance Change versus Time in rectangular coordinates.

## 14. Precision

14.1 Due to the range of joint coating formulations, thicknesses, densities, etc. found among commercially available materials, the over-all accuracy and reproducibility of test results by these methods will tend to be poorer than those expected on straight runs of coated pipe. The fact that most joint materials are hand-applied introduces an inherent factor of variability into the test results. The basic purpose of these methods is to provide a means for the comparative screening of combinations of joint materials and base coatings for insulating effectiveness.

14.2 The precision (reproducibility) of the joint resistance determination by these methods is considered to be such that when two tests are performed consecutively on the same specimen and under identical conditions of application, the difference between the two results may normally be expected not to exceed ±10 % of their mean.

## 15. Keywords

15.1 capacitance; coating; current; dissipation factor; fittings; immersion; joint; leakage; patches; pipeline; resistance

Designation: G 19 – 88

# Standard Test Method for Disbonding Characteristics of Pipeline Coatings by Direct Soil Burial[1]

This standard is issued under the fixed designation G 19; 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 describes the determination of the relative disbonding characteristics of damaged coatings on steel pipe by cathodic protection potentials in direct soil burial. This test method is intended to apply to the testing of all types of nonmetallic pipeline coatings and tapes including thermoplastics, thermoset, and bituminous materials.

1.2 Results may vary widely when test sites are in different geographical areas of the country, and even in different localities.

1.3 This test method is limited to nonconducting, or nonmetallic pipe coatings and is not applicable to conducting materials such as zinc coatings on steel pipe.

1.4 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 The values stated in SI units to three significant decimals are to be regarded as the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

G 62 Test Methods for Holiday Detection in Pipeline Coatings[2]

## 3. Summary of Test Method

3.1 Apparatus and materials are described whereby protective coatings on steel pipe are subjected to disbonding by an electrical stress. Specimens with intentionally damaged areas are buried in soil at an outdoor site and electrically connected to a magnesium anode. After test, the disbonded coating is removed, the exposed area measured, and comparisons are made to other specimens similarly exposed.

## 4. Significance and Use

4.1 Coated pipe is seldom, if ever, buried without some damage to the coating. Hence, an actual soil-burial test can contribute significant data, provided the method of testing is controlled and the test specimen monitored and the relationship between the area disbonded, the current demand, and the mode of failure is fully understood.

4.2 Means are provided for measuring and following the electrical potential and current flow and relating these data to the final measurement of disbonded area.

## 5. Apparatus

5.1 *Anode*—A standard packaged magnesium anode, minimum 4.082 kg (9 lb), with a factory-sealed, 4107-cmil (14-gage Awg) minimum, insulated copper wire shall be used. A solution potential of not less than −1.45 V with respect to a copper-copper sulfate reference electrode is required. Use sufficient anodes to maintain required potential.

5.2 *Connectors*—The wiring circuit from anode to test specimen and from specimen to reference electrode should be 4107-cmil (14-gage Awg) minimum insulated copper wire. Attach the wires to the test specimen as shown in Fig. 1 by soldering or brazing at the air-exposed end, and coat the place of attachment with insulating material. A junction box is optional for connecting the resistor in series between the anode and the test specimen.

5.3 The instruments used shall include the following:

5.3.1 *Voltmeter*, a suitable instrument such as a high impedance (>10mΩ) analog multimeter having a sensitivity of 50 000 Ω/V minimum and a multiple range from 0.01 to 2 V for direct current is used for measuring the potential between specimen and the reference electrode,[3] current between specimen and anode, and the resistance of the circuit. The same instrument shall be used for measuring current between specimen and anode. Alligator clips on the leads are permissible.

5.3.2 *Volt-Ohm-Meter* for measuring resistance of the circuit.

5.3.3 *Reference Electrode*, consisting of a copper-copper sulfate half cell in a conventional glass or plastic tube with porous plug construction, but preferably not over 19.05 mm (¾ in.) in diameter, having a potential of −0.316 V with respect to a standard hydrogen electrode. A calomel electrode may be used, but measurements made with it should be converted to the copper-copper sulfate reference electrode for reporting, by adding −0.092 V to the observed reading.

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nometallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 27, 1988. Published July 1988. Originally published as G 19 –71 T. Last previous edition G 19 – 83.

[2] *Annual Book of ASTM Standards*, Vol 06.02.

[3] A pipe-to-soil Voltmeter-Ammeter, Agra Engineering Co., Tulsa, Okla., has been found suitable for this test.

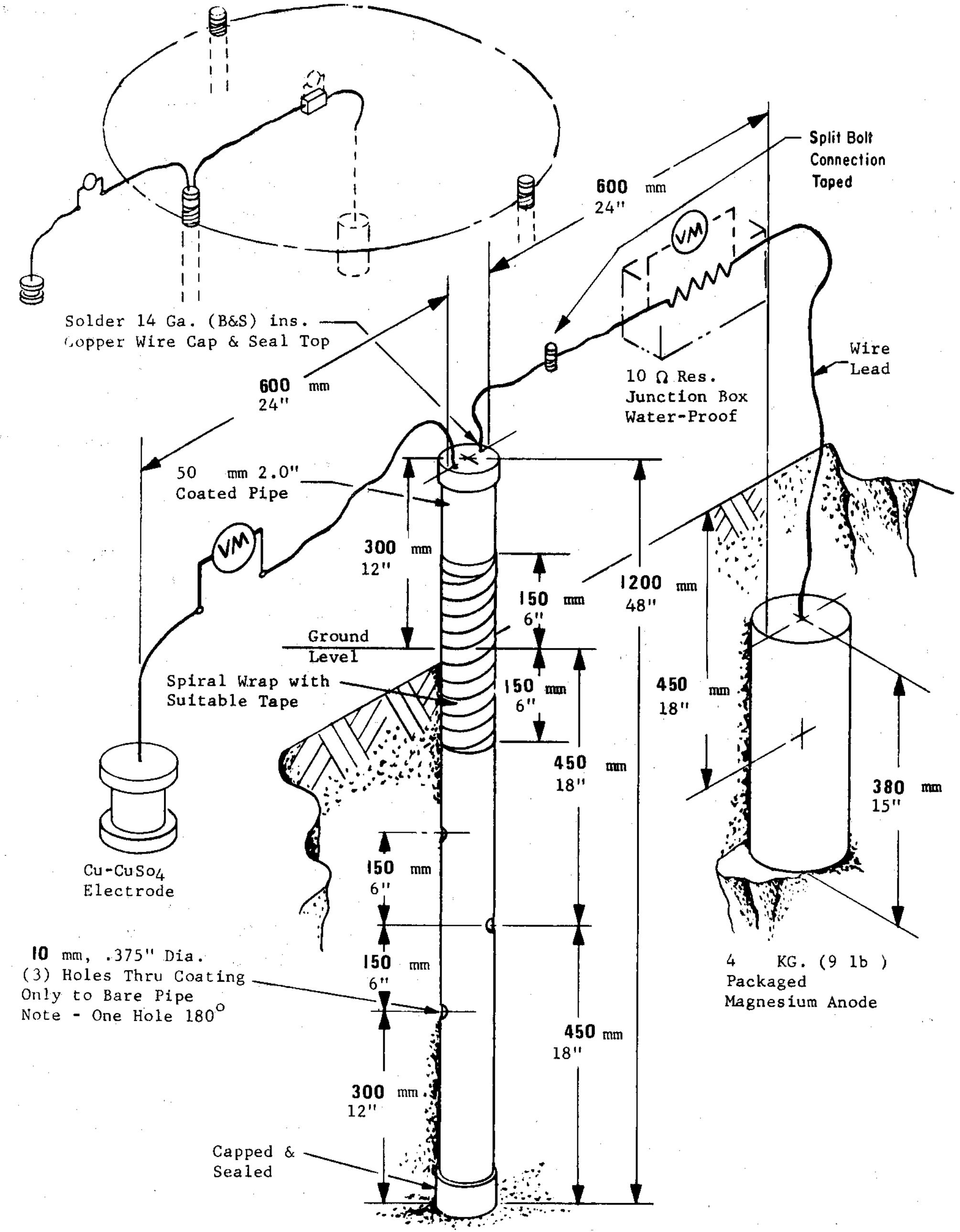

FIG. 1 Three Specimens in a Circle with a Common Anode

5.3.4 A suitable instrument is used to measure the soil resistivity using the four-pin method.[4] Pins should be spaced 762 mm (30 in.) apart.

5.3.5 *Thickness Gages*, to be used in accordance with Test Method G 12.

5.3.6 *Holiday Tools*—Holidays in the specimen are made with conventional drills of the required diameter. A 9.525-mm (0.375-in.) drill modified by substantially reducing the cone angle has been found effective in preventing perforation of thin-wall pipe or tubing. A sharp-pointed knife with a safe handle is required for removing disbonded coatings to make physical examinations. A micrometer-type depth gage is used for measuring coating thicknesses at the edge of holidays.

5.3.7 *Holiday Detectors*—Selected in accordance with Test Method G 62.

## 6. Test Specimen

6.1 A 1219.2 mm (48-in.) long specimen shall be prepared with its surface preparation and coating procedures equivalent to that of production coated pipe. Only holiday-free specimens may be used in this test and four samples should be prepared.

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[4] A Vibroground instrument, Associated Research Inc., 3758 Belmont Ave., Chicago, Ill., has been found suitable for measuring soil resistivity.

6.2 Measure the coating thickness of all specimens at four points 90 deg apart and approximately 355.6 mm (14 in.) from each end.

6.3 Cap and seal one end of each specimen. Check the seal for current leakage before and after test by immersion in 1 weight percent sodium chloride solution for ½ h. Test with an ohmmeter sensitive to at least 1 MΩ.

6.4 Provide the unsealed end of the pipe with two lengths of insulated 4107-cmil (14-gage Awg) copper wire as in 5.2.

6.5 Make 3 holidays 304.8 mm (12 in.), 457.2 mm (18 in.), and 609.6 mm (24 in.), respectively, from the sealed end of the pipe by drilling holes through the coating so that the drill will fully enter the steel. The center holiday is to face the anode with the remaining two holidays facing 180 deg away from the anode. The drill diameter may not be less than two times the coating thickness, and never smaller than 9.525 mm (0.375 in.) in diameter. Do not perforate the steel wall of the pipe. With thin-wall steel pipe where there is danger of perforating the pipe, start the holiday with a standard 60-deg cone-point drill and finish with a substantially reduced cone-angle drill.

6.6 To prevent mechanical damage, apply a spiral wrap of suitable tape from approximately 152.4 mm (6 in.) from the top of the pipe specimen to 457.2 mm (18 in.) from the top. When the test specimens are buried, the taped area will extend 152.4 mm above and 152.4 mm below ground.

6.7 The specimen test area will consist of the area between the edge of the bottom end seal and the ground level. The bottom end-seal area is not considered part of the area tested. Any suitable diameter and length of pipe may be used, but the buried area should not be less than 23 227.2 mm$^2$ (36 in.$^2$). An area of 93 000 mm$^2$ (1 ft$^2$) has been found preferable.

## 7. Test Site

7.1 Any level location may be used, provided the site will not be disturbed for the duration of the test.

## 8. Procedure

8.1 Lay out the test site with stakes so that the anode location will be the center of a circle as shown in Fig. 1. Locate the pipe specimens 20° apart around the circumference so that the surface of the anode when placed in the center will be 609.6 mm (24 in.) from the nearest holiday of each specimen equally. Three specimens can thus be protected by one anode.

8.2 *Installation of Test Specimens:*

8.2.1 Dig suitable holes with an auger or posthole digger to accommodate the anode and test specimens at the test site.

8.2.2 Insert the anode at the center of the circle. The center of the anode should be 457.2 mm (18 in.) below ground. Pipe specimens should be inserted so that 304.8 mm (12 in.) are above the ground level, and oriented so that the single intentional holiday faces the anode and the remaining two holidays are 180 deg away from the anode. Maintain a distance of 609.6 mm (24 in.) between the surface of the anode and the nearest specimen and holiday.

8.2.3 Refill all holes with soil or a soil and water slurry. Firmly tamp the soil so that it is in intimate contact with the specimen and anode. No wood or other foreign material should contact the pipe coating or the anode.

8.3 *Electrical Measurements:*

8.3.1 Determine in several areas the soil resistance in ohm-centimetres by the four-pin method.

8.3.2 Measure the initial pipe to soil potential with reference to a copper-copper sulfate half cell with the electrode 609.6 mm (24 in.) from the pipe as shown in Fig. 1. Record the closed-circuit potentials.

8.3.3 Connect the anode lead to one test specimen lead at the junction box with a 10 Ω ± 1 % wire-wound resistor connected in series between the anode and test specimen.

8.3.4 Measure the voltage. $E$, across the 10-Ω resistor, $R$, and convert to current, $I$ as follows:

$$I = E/R = E/10$$

8.3.5 Measure the polarized potential, in volts with the analog multimeter described in 5.3.8 connected between the test specimen and the reference electrode as follows:

8.3.5.1 Disconnect the anode from the test specimen while closely observing the analog multimeter. As the instrument pointer falls, it will dwell significantly at the polarized value before receding further. The dwell point is the polarized potential.

8.3.6 Determine electrical measurements at 30-day intervals for a test period of 18 months or longer depending on soil conditions.

## 9. Report

9.1 The report shall include the following:

9.1.1 Complete identification of the test specimens, including name and code number of coating, size of the pipe, source, production date, production run number, and any other information that may be pertinent to identification.

9.1.2 Date of starting and of terminating the test.

9.1.3 Coating thickness of the test specimen before testing.

9.1.4 After subtracting the initial holiday areas, report the total disbonded area, average disbonded area, and disbonded area at each holiday in square millimetres (square inches) after testing.

9.1.5 Condition of the pipe surface under the disbonded coating.

9.1.6 Pipe-to-soil potentials, initial readings and at 30-day intervals.

9.1.7 Current readings between anode and pipe specimens, initial readings and 30-day intervals.

9.1.8 Soil resistivity readings in ohm-centimetres.

9.1.9 Polarized potentials, initial readings and 30-day intervals.

## 10. Precision

10.1 Precision data are limited to two adjacent specimens taken from the same production-coated pipe, assuming that the production process was uniform with respect to pipe surface condition and coating material. Specimens that were not adjacent in the as-produced condition, or were taken from different lengths of pipe may represent differing process conditions.

Designation: G 20 - 88

# Standard Test Method for Chemical Resistance of Pipeline Coatings[1]

## 1. Scope

1.1 This test method is intended for evaluating the resistance of pipe coating materials when exposed to various concentrations of reagents or suspected soil contaminants. The test serves as a guide to investigators wishing to compare the relative merits of pipe-coating materials in specific environments. The choice of reagents, concentrations, duration of immersion, temperature of test, and properties to be reported are necessarily arbitrary and should be chosen to reflect conditions known to exist along the pipeline right-of-way.

1.2 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method 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 543 Test Method for Resistance of Plastics to Chemical Reagents[2]

D 883 Terminology Relating to Plastics[2]

G 8 Test Methods for Cathodic Disbonding of Pipeline Coatings[3]

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[3]

G 17 Test Method for Penetration Resistance of Pipeline Coatings (Blunt Rod)[3]

## 3. Summary of Test Method

3.1 This test method consists of an immersion-type test in a closed container where coated pipe specimens are in long-term contact with both the liquid and vapor phase of the test reagent. Specimens exposed in this manner are inspected for visible signs of chemical attack. Subsequent tests for cathodic disbonding in accordance with Test Method G 8, or penetration under load in accordance with Test Method G 17, may be applied to determine if the specimens have undergone any loss of mechanical or bonding properties.

## 4. Significance and Use

4.1 The data obtained for short-term tests are of interest only in eliminating the most unsuitable materials or for indicating a probable order of resistance in any particular media.

4.2 Test conditions should take into account the manner and duration of immersion, the reagent, the temperature of the system, the area exposed above and below the liquid level, and other performance factors selected for the particular test.

## 5. Apparatus

5.1 *Thickness Gage*, capable of measuring the coating thickness in the manner prescribed by Test Method G 12.

5.2 *Test Container*—A transparent closed container, sized to completely encase the pipe specimen and large enough to provide adequate exposure to both the liquid and vapor states of reagent.

NOTE 1—For example, a 2-L (2.0-qt) capacity, Mason-type jar with a 70-mm (2.75-in.) diameter neck has been found suitable for use with 2 in. pipe and is illustrated in Fig. 1.

5.2.1 To avoid pressure build-up within the test containers, the threaded cup shall be replaced with a solid-rubber stopper. A positive venting device, such as a water seal, shall be used when testing with volatile solvents at elevated temperatures.

5.2.2 A separate container shall be used for each test specimen.

5.3 *Oven or Constant-Temperature Room or Bath*—To ensure uniformity of test results, the test cells and specimen shall be maintained at the test temperature ±5°C (9°F) over the duration of the test period.

5.4 *Auxiliary Testing Devices*—Supplemental equipment used to determine specific mechanical properties of specimens before and after immersion shall conform to the requirements prescribed in the applicable ASTM test method.

## 6. Test Specimen

6.1 The test specimen shall be prepared with its surface preparation and coating procedures equivalent to that of coated pipe. A control specimen shall be retained for comparison.

6.2 In order to utilize the test container specified in 4.2, the pipe specimen, with end caps, shall be restricted to a maximum overall length of 180 mm (7.0 in.) and a diameter of 65 mm (2.5 in.).

6.3 Both ends of the pipe specimens shall be plugged with inert stoppers and coated with an epoxy-base coating material.

6.4 *Specimens with Holidays:*

6.4.1 A holiday shall be made in the surface of the coated pipe specimen at a point 25 % of the distance between the

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[1] This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vols 06.02.

end caps. It shall be made by drilling a radial hole through the coating so that the angular cone point of the drill will fully enter the steel where the cylindrical portion of the drill meets the steel surface. The drill diameter shall not be less than three times the coating thickness, but it shall never be smaller than 6 mm (0.25 in.) in diameter. The steel wall of the pipe shall not be perforated.

6.4.2 A second identical holiday shall be drilled into the coating surface at a point 25 % of the distance between the end caps of the specimen (see Fig. 1). Both holidays should lie in the same pipe axis.

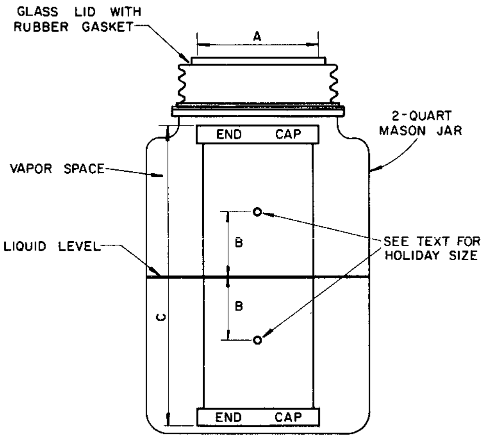
6.5 Specimens without intentional holidays shall also be prepared for testing.

NOTE 2—Intentional holidays shall be made in only those specimens for which a comparison of disbonding properties is desired. Other samples used for supplemental mechanical testing shall be run without holidays.

## 7. Reagents

7.1 The reagents selected for coating-resistance tests should be those anticipated to occur in the environment or in the product being carried in the pipeline at the temperatures and in the concentrations expected. The numbers in parentheses refer to the list of standard reagents given in Section 4 of Test Method D 543.

7.1.1 *Acetic Acid* (5 %) (4.4.2).
7.1.2 *Acetone* (4.4.3).
7.1.3 *Carbon Disulfide.*
7.1.4 *Gasoline.*
7.1.5 *Hydrochloric Acid* (10 %) (4.4.23).
7.1.6 *Kerosine* (4.4.28).
7.1.7 *Lime Water, Saturated.*
7.1.8 *Methyl Alcohol* (4.4.29).
7.1.9 *Methyl Ethyl Ketone.*
7.1.10 *Nitric Acid* (10 %) (4.4.33).
7.1.11 *Sodium Carbonate Solution* (20 %) (4.4.38).
7.1.12 *Sodium Chloride Solution* (10 %) (4.4.40).
7.1.13 *Sodium Hydroxide Solution* (10 %) (4.4.42).



Dimensions

*A*—70 mm (2.75 in.) Jar Opening
*B*—45 mm (1.75 in.) Holiday Location
*C*—90 mm (7.00 in.) Length of Pipe Specimen

NOTE—Dimensions shown for 2 in. IPS pipe.

**FIG. 1 Chemical Immersion Test Schematic of Test Cell**

7.1.14 *Sulfuric Acid* (30 %) (4.4.46).
7.1.15 *Toluene* (4.4.48).
7.1.16 *Transformer Oil* (4.4.49).
7.1.17 *Trichlorethylene.*
7.1.18 Other selected environments.

## 8. Precautions

8.1 Safety precautions should be taken to avoid personal contact, to eliminate toxic vapors, and to guard against explosion hazards in accordance with the hazardous nature of the particular reagents being used.

## 9. Procedure

9.1 Place a single specimen in a vertical position in each test container.

9.2 Fill the container with the selected reagent so that the liquid level covers one half of the coated pipe specimen up to a point midway between the two intentional holidays. Stopper the 704 Container to prevent evaporation of the reagent and against contamination.

9.3 Maintain the reagent level at the original level.

9.4 The basic immersion test for a particular coating material shall consist of twelve specimens, six with holidays and six without, each in an individual test cell. Remove duplicate samples of each from the test at 30, 60, and 90 day intervals. Additional investigations of pre- and post immersion mechanical properties will require additional specimens. It is recommended that in all cases provisions be made for duplicate test specimens.

9.5 Remove the specimen after 30, 60, and 90 days of immersion. Wash with running water all specimens removed from acid, alkali, or other aqueous solutions and wipe them dry with a soft, clean, cotton cloth or paper tissue. Specimens removed from volatile solvents such as acetone, alcohol, etc., need no rinsing before wiping dry.

9.6 Observe and report before, immediately after, and 2 h later the appearance of specimen after exposure to reagent on the basis of visual examination for evidence of loss of gloss, developed texture, decomposition, discoloration, softening, swelling, injury, bubbling, blistering, cracking, solubility, etc. as defined in D 883.

9.7 Mechanically probe each of the intentional holidays in accordance with Section 8.3.3 of Test Method G 8 to see if there has been any loss of coating bond during the test period.

9.8 Perform other planned mechanical tests to determine if any degradation of coating properties has occurred through reagent exposure.

## 10. Report

10.1 The report shall include the following:

10.1.1 Complete identification of the material tested, including type, source, manufacturer's code, and previous history,

10.1.2 Method of preparing test specimen,

10.1.3 Temperature of test,

10.1.4 Description of media, including ASTM designation,

10.1.5 Duration of immersion,

10.1.6 Outside diameter of test specimen,

10.1.7 Initial thickness of coating,

10.1.8 General appearance of specimen after immersion,

10.1.9 Immersion area in square millimetres (square inches),

10.1.10 Vapor phase area in square millimetres (square inches), and

10.1.11 Disbonded area after immersion in square millimetres (square inches).

## 11. Precision and Bias

11.1 As there are no direct measurements involved with this test method, no precision and bias statement is necessary. Measurements made by procedures for other test methods should refer to the precision and bias statements therein.