

FED. TEST METHOD STD. NO. 141C
January 24, 1986

FEDERAL TEST METHOD STANDARD

PAINT, VARNISH, LACQUER, AND RELATED MATERIALS;

METHODS FOR SAMPLING AND TESTING

AUTHORITY: This standard is issued pursuant to the Federal Property and Administration Service Act of 1949, as amended, and its application to the purchase of commodities referred to herein is mandatory on all Federal agencies.

SECTION 1

SCOPE AND CONTENTS

1. **SCOPE.** This standard elaborates the methods used to determine the physical and chemical properties of paint, varnish, lacquer, and related materials to evaluate their conformance to Federal and Military specifications. The purpose of this standard is to eliminate unnecessary or undesirable variation in general inspection, sampling, and testing procedures. This standard or methods contained herein shall become a portion of any particular specification. If any variation or modifications of the methods in this standard are required by any specification, the procedures listed in the specification will take precedence. Any conflict between this standard and any particular specification shall be resolved in favor of the specification.

2. **CONTENTS.** The contents of this standard are arranged in sections as follows:

Section

1. Scope and Contents.
2. Form and Numbering System.
3. Alphabetical Index of Test Methods.
4. Numerical Index of Test Methods.
5. Numerical Index of Canceled Test Methods.
6. Numerical Index of ASTM Methods to be Used in Place of Canceled Fed. Test Method Std No. 141 Test Methods.
7. Notes on the Use of this Standard.
8. Reagents, Water, and Ethyl Alcohol.
9. Routine and Referee Testing Conditions.
1000. Sampling for Inspection and Testing.
2000. Preparation of Panels and Application of Test Films.
3000. Package Stability Tests.
4000. Physical Tests of Coating Materials and Coating Ingredients.
5000. Chemical Tests of Coating Materials and Their Ingredients.
6000. Physical and Chemical Tests of Dried Films.
7000. Analytical Tests of Coating Materials and Their Ingredients.

FSC 8010

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SECTION 2

FORM AND NUMBERING SYSTEM

1. SCOPE. This section contains a brief description of the form and the system of numbering within the individual methods, and also the manner in which new methods are added and old methods revised.

2. FORM. The binding of this standard permits separation for convenience in adding new methods and revising existing methods.

2.1 Revision of existing methods.

2.1.1 Numbering system used. When a technical change or modification is issued, it will be identified by adding a decimal point to the basic method number or by incrementing the decimal by one.

2.1.2 Revision precedence. When a revision is issued on a Standard 141 method, it automatically supersedes the previous method. The particular revision in force at the time of invitation to bid shall be the method used for testing.

2.2 New methods. A placement of new methods in this standard is based on the following criteria:

a. The character or purpose of the test method indicates the specific section to which it belongs.

b. A method number is assigned so that the new method is located close to methods of similar or related type.

2.3 Use of ASTM test methods. American Society for Testing and Materials (ASTM) methods listed in section 6 of this standard have been accepted as substitutes for the applicable canceled Federal test methods and shall be used whenever the Fed. Test Method Std. No. 141 method number is referenced. When superseded 141 methods are to be cited in the future, the accepted ASTM method shall be cited directly.

3. INDEXES AND METHOD FORMAT

3.1 The indexes.

3.1.1 Alphabetical index (section 3). In the alphabetical index, each current method is listed alphabetically by its primary purpose as indicated in its title.

3.1.2 Numerical index (section 4). The numerical index lists all current methods in numerical order.

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3.1.3 Numerical index of canceled test methods (section 5). An index of canceled, deleted, or superseded test methods is listed with cross-reference to the superseding Fed. Test Method Std. No. 141 or accepted ASTM test methods where applicable.

3.1.4 Numerical index of accepted ASTM test methods (section 6). The ASTM test methods accepted are listed with cross-reference to canceled Fed. Test Method Std. No. 141 test methods.

3.2 Method format. Whenever possible, the methods contain the first five of the following six paragraphs. Data on precision are included whenever available.

3.2.1 Scope. The scope of each method is given in paragraph 1. It describes the purpose of the method.

3.2.2 Apparatus. The apparatus is listed in paragraph 2. Unusual apparatus is listed, along with standardization techniques and construction requirements, if applicable.

3.2.3 Reagents. All reagents required in a method are listed in paragraph 3. Details are given for the preparation of reagents which must be manufactured specifically for a particular method.

3.2.4 Procedure. The procedure to be followed is detailed in paragraph 4. The procedure generally includes three major sections: preparation of the test specimen, performance of the test involved, and calculation of results.

3.2.5 Notes. Any pertinent notes are listed in paragraph 5. These notes will include caution warnings, comments on variations in results, and similar comments.

3.2.6 Precision. Data on precision, both within one laboratory and between different laboratories, are listed in paragraph 6.

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Application of Flowed Films	2151	-	-
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Kauri Reduction Test	4151	-	D 1642 Method B
Rosin-Pentaerythritol Ester Reduction Test	4152	-	D 1642 Method A
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Specific Gravity of Pigment (Centrifuge Method)	4182	-	-
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Specific Gravity of Solid Resins	4185	-	-
Oil Absorption (Rub Out)	4191	-	D 281
Oil Absorption (Gardner-Coleman)	4192	-	-
Miscibility Test for Pastes-in-Oil	4201	-	-
Mixing Properties of Aluminum Mixing Varnishes	4202	-	-
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Bronzing Test of Celluloses	4205	-	-
Compatibility with Basic Pigment	4206	-	-
Compatibility with Zinc Oxide	4207	-	-
Evaluating Degree of Settling of Paint	4208	-	D 869
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Color of Transparent Liquids (Barrett Scale)	4244	-	-	-
Color of Transparent Liquids (Lovibond Scale)	4245	-	-	-
Color of Transparent Liquids (Saybolt Scale)	4246	-	-	D 156
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Color of Transparent Liquids (Gardner Color Standards of 1953)	4248	-	-	D 1544
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Distillation of Liquid Naval Stores	4302	-	D 1078
Distillation of Aromatic Hydrocarbons	4303	-	D 1078
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Separation of Volatile Matter (Ordinary Distillation)	4351	-	-
Odor Test	4401	-	D 1296
Heating Test (Gel Time)	4441	-	D 1955
Quality Test (Tung Oil)	4442	-	D 1964
Leafing Test (Aluminum)	4451	-	D 480
Pour Point	4452	-	Sec 6-12 D 97
Cloud Point	4452	-	D 2500
Loss on Heating (Fatty Oils)	4461	-	D 1960
Loss on Ignition	4462	-	D 1208
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Softening Point by Ring and Ball Apparatus	4495	-	E 28
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Acid Number (Varnishes)	5073	-	D 1639
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Polymerization Residue	5121	-	D 233
Aromatic Hydrocarbon Content (Spectrophotometric Method)	5131	-	-
Break Test (Oils)	5161	-	D 1952
Foots Test	5162	-	D 1954
Ester and Ketone Content of Lacquer Thinner	5171	-	-
Aniline Point	5181	-	D 611
Mixed Aniline Point	5182	-	D 611
Kauri-Butanol Value	5191	-	D 1133
Nitrocellulose in Floor Sealer	5201	-	-
Nitrocellulose in Nonvolatile Vehicles	5202	-	-
Nitrocellulose in Presence of Glycol Sebacate (First Alternate)	5203	-	-
Nitrocellulose in Presence of Glycol Sebacate (Second Alternate)	5204	-	-
Water Solubility (Pigments)	5241	-	D 1208 Sec 3
Water Solubility (Alcohols)	5242	-	-
Water Solubility (Solvents and Plasticizers)	5243	-	-
Water Solubility (Finished Materials)	5244	-	-

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SECTION 5

NUMERICAL INDEX OF CANCELED TEST METHODS

Title of canceled test method	Canceled		Use	
	FTMS No. 141 Method No.	FTMS No. 141 Method No.	FTMS No. 141 Method No.	ASTM Method No.
Acidity (Dopes)	5251	-	-	-
Acidity (Lacquer Solvents and Diluents)	5252	-	-	D 1613
Acidity - Mineral Acid	5253	-	-	-
Acidity - Mineral Acid (After Distillation)	5254	-	-	-
Acidity of Aromatic Hydrocarbons	5255	-	-	D 847
Ash (Pigments and Other Solids)	5261	-	-	D 1208 Sec 2
Ash (Clear Materials)	5262	-	-	D 1951
Ash (Vehicle Extracted from Pigmented Materials)	5263	-	-	D 1951
Ash (Liquid Driers)	5265	-	-	D 1951
Silica Content of Silicone Resin Vehicles	5266	-	-	-
Plasticizer and Resin in Nonvolatile Solids	5291	-	-	-
Sulfur Compounds in Volatile Thinners	5311	-	-	D 853
Acid Wash of Volatile Thinners	5321	-	-	-
Acid Wash of Aromatic Hydrocarbons	5322	-	-	-
Sulfuric Acid Solubility and Paraffin Content of Volatile Thinners	5331	-	-	-
Cloud Point	5341	-	-	D 2500
Doctor Test	5351	-	-	D 484 Sec 4C
Nitro Compounds in Cellulose	5391	-	-	-
Spot Test for Nitrate in Doped Fabrics	5392	-	-	-
Paraffin Content of Aromatic Hydrocarbons	5411	-	-	-
Solidification Point of Benzene	5421	-	-	-
Immersion Resistance	6011	-	-	D 1308 6.4
Cellulose Film Test	6021	-	-	-
Salt Spray (Fog) Test	6061	-	-	B 117
Method for Testing Coated Metal Panels at 100 Percent Relative Humidity	6071	-	-	D 2247

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Title of canceled test method	Canceled FTMS No. 141 Method No.	Use FTMS No. 141 Method No.	Use ASTM Method No.
Spotting Resistance	6081	-	D 1308 6.2 & 6.3
Flatness	6102	-	-
45-Degree, 0-Degree Directional Reflectance	6121	-	E 97
Luminance Factor of Retroreflective Materials, Instrumental Measurements	6125	Use FTMS No. 370	
Scrubbing Resistance of Oil Paints	6143	-	-
Accelerated Weathering (Open Arc Apparatus)	6151	-	G 23, D 822
Accelerated Weathering (Enclosed Arc Apparatus)	6152	-	G 23, D 822
Conducting Exterior Exposure Tests of Paints on Metals	6160	-	D 1014
Conducting Exterior Exposure Tests of Paints on Wood	6161	-	D 1006
Brittleness of Doped Fabrics	6162	-	-
Moisture Vapor Permeability of Organic Coating Films	6171	-	D 1653
Dry Film Thickness (Magnetic Type Gage)	6181	-	D 1186
Dry Film Thickness of Cast Films	6182	-	-
Dry Film Thickness (Mechanical Type Gage)	6183	-	D 1005
Abrasion Resistance (Falling Sand)	6191	-	D 968
Print Test	6211	-	D 2091
Indentation Hardness of Organic Coatings	6212	-	D 1474
Blocking Test	6216	-	-
Flexibility (Percent Elongation - Conical Mandrel)	6222	-	D 522
Flexibility (Percent Elongation - Cylindrical Mandrel)	6222	-	D 1737
Elongation and Tensile Strength of Free Films	6224	-	D 2370
Elongation of Cast Films	6225	-	D 2370
Impact Flexibility	6226	-	D 2794
Infrared Reflectance (From Photograph)	6243	-	-

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NUMERICAL INDEX OF CANCELED TEST METHODS

Title of canceled test method	Canceled FTMS No. 141 Method No.	Use FTMS No. 141 Method No.	Use ASTM Method No.
Tautness of Coped Fabrics	6281	-	-
Bursting Strength of Doped Fabrics	6291	-	-
Adhesion (Parallel Groove Method)	6302	-	D 2197 Method B
Adhesion of Coatings with Scrape-Adhesion Apparatus	6303	-	D 2197 Method A
Marking by Masking Tape	6311	-	-
Adhesion and Strippability	6317	-	-
Performance Tests for Floor Sealers	6331	-	-
Rubbing Test	6332	-	-
Removal Power and Removability	6352	-	-
Degree of Resistance to Chalking of Exterior Paints	6411	-	D 659
Degree of Resistance to Checking of Exterior Paints	6421	-	D 660
Degree of Resistance to Erosion of Exterior Paints	6431	-	D 662
Degree of Resistance to Flaking (Scaling) of Exterior Paints	6441	-	D 772
Rusting Obtained with Paint on Iron or Steel Surface, Degree of Resistance to.	6451	-	D 610
Degree of Blistering of Paints	6461	-	D 714
Degree of Resistance to Cracking of Exterior Paints	6471	-	D 661
Unsaponifiable Matter in Drying Oils and Fatty Acids	7011	-	D 1965
Unsaponifiable Matter in Alkyd Resins and Resin Solutions	7012	-	D 1397
Chlorine Content of Vinyl-Alkyd Resins	7015	-	D 1156
Phthalic Anhydride Content of Alkyd Resins	7021	-	D 563
Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids	7022	-	D 1306
Phthalic Anhydride Content of Vinyl Alkyd Resins	7023	-	-
Phthalic Anhydride Content of Alkyd Resins (Spectrophotometric)	7025	-	D 1307
Melamine-Formaldehyde Resin in Alkyds	7027	-	-

XTX

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Title of canceled test method	Canceled FTMS No. 141 Method No.	Use FTMS No. 141 Method No.	Use ASTM Method No.
Oil Acids Content of Alkyd Resins	7031	-	D 1398
Analysis of Basic Carbonate White Lead Pigment	7041	-	D 1301
Analysis of Basic Sulfate White Lead Pigment	7051	-	D 1301
Analysis of Blue Lead Pigment	7061	-	-
Colorimetric Determination of Red Lead	7072	-	-
Analysis of Titanium Dioxide Pigment (Jones Reduction)	7081	-	D 1394
Analysis of Titanium Dioxide Pigment (Zinc Amalgam)	7082	-	-
Analysis of Titanium Dioxide Pigment (Aluminum Reduction)	7083	-	D 1394
Analysis of Titanium Dioxide Pigment (Titration with Ferric Salt Solution)	7084	-	-
Analysis of Zinc Oxide Pigment (Outside Indicator)	7091	-	D 34
Analysis of Zinc Oxide Pigment (Inside Indicator)	7092	-	-
Analysis of Lithopone Pigment	7101	-	-
Analysis of Antimony Oxide Pigment	7106	-	D 2350
Analysis of Chrome Green Pigment (Perchloric Acid)	7112	-	-
Analysis of Chromium Oxide Green Pigment	7121	-	D 126 Par. 38
Analysis of Chromium Oxide Green Pigment (Perchloric Acid)	7122	-	-
Analysis of Molybdate Orange	7133	-	D 2218
Analysis of Cadmium Pigment	7135	-	-
Analysis of Yellow Ocher	7141	-	D 50
Analysis of Metallic Brown	7151	-	D 50
Analysis of Para Red, Toluidine Red, and Lithol Red Toner Pigments	7191	-	D 970
Analysis of Ultramarine Blue Pigment	7201	-	D 1135
Analysis of Zinc Yellow Pigment	7211	-	D 444
Analysis of Zinc Dust (Metallic Zinc Powder)	7221	-	D 521

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Title of canceled test method	Canceled FTMS No. 141 Method No.	Use FTMS No. 141 Method No.	Use ASTM Method No.
Metals Content of Driers	7231	-	D 564
Analysis for Total Copper in Pigments	7232	-	D 283
Analysis of Aluminum in Aluminum Powder and Aluminum Paste	7233	-	D 480
Analysis of Magnesium Silicate Pigment	7251	-	D 717
Analysis of Calcium Carbonate	7253	-	D 34
Analysis of Pigment Extracted from Graphite Paints	7321	-	-
Distillation of Solvents from Enamels and Lacquers	7355	-	D 3272
Epoxy Content of Epoxy Resins	7403	-	D 1652
Available NCO (Isocyanate) in Urethanes	7421	-	D 2572
Free Toluene Diisocyanate in Urethanes	7426	-	D 2615
Identification of Drying Oils (Gas-Liquid Chromatography)	7501	-	D 2800, D 2245, D 1983

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NUMERICAL INDEX OF ASTM METHODS TO BE USED IN PLACE OF
 CANCELED FED. TEST METHOD STD. NO. 141 TEST METHODS

ASTM Method No.	FTMS No. 141 Method No.
B 117	6061
D 5	4273
D 29	4255
D 34	7091
D 34	7253
D 50	7141
D 50	7151
D 56	4291
D 56	4292
D 88	4285
D 92	4294
D 93	4293
D 97	4452
D 126	7121
D 153	4181
D 156	4246
D 185	4101
D 233	5121
D 279	4572
D 280	4071
D 281	4191
D 283	7232
D 332	4222
D 358	2031
D 387	4220
D 387	4221
D 444	7211
D 480	4451
D 480	7233
D 484	5351
D 521	7221
D 522	6222
D 562	4281
D 563	7021
D 564	7231
D 611	5181
D 611	5182

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ASTM Method No.	FTMS No. 141 Method No.
D 659	6411
D 660	6421
D 661	6471
D 662	6431
D 714	6461
D 717	7251
D 772	6441
D 822	6151
D 822	6152
D 823	2121
D 823	4341
D 847	5255
D 853	5311
D 869	4208
D 891	4183
D 937	4273
D 968	6191
D 970	7191
D 1005	6183
D 1006	6161
D 1014	6160
D 1078	4301
D 1078	4302
D 1078	4303
D 1133	5191
D 1135	7201
D 1156	7015
D 1186	6181
D 1200	4282
D 1208	4462
D 1208	5111
D 1208	5241
D 1208	5261
D 1209	4243
D 1296	4401
D 1301	7041
D 1301	7051
D 1306	7022

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 CANCELED FED. TEST METHOD STD. NO. 141 TEST METHODS

ASTM Method No.	FTMS No. 141 Method No.
D 1307	7025
D 1308	6011
D 1308	6081
D 1343	4272
D 1353	4045
D 1364	4082
D 1394	7081
D 1394	7083
D 1397	7012
D 1398	7031
D 1474	6212
D 1475	4184
D 1476	4083
D 1542	5031
D 1542	5032
D 1544	4248
D 1545	4271
D 1613	5252
D 1616	5101
D 1616	5102
D 1639	5071
D 1639	5072
D 1639	5073
D 1642	4151
D 1642	4152
D 1643	4161
D 1643	4162
D 1643	4171
D 1652	7403
D 1653	6171
D 1720	4204
D 1724	5011
D 1729	4249
D 1732	2015
D 1737	6222
D 1849	3018
D 1951	5262
D 1951	5263

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NUMERICAL INDEX OF ASTM METHODS TO BE USED IN PLACE OF
 CANCELED FED. TEST METHOD STD. NO. 141 TEST METHODS

ASTM Method No.	FTMS No. 141 Method No.
D 1951	5265
D 1952	5161
D 1954	5162
D 1955	4441
D 1959	5061
D 1960	4461
D 1962	5081
D 1964	4442
D 1965	7011
D 1983	7501
D 2091	6211
D 2197	6302
D 2197	6303
D 2218	7133
D 2243	3012
D 2245	7501
D 2247	6071
D 2350	7106
D 2369	4041
D 2370	6224
D 2370	6225
D 2500	4452
D 2500	5341
D 2572	7421
D 2615	7426
D 2698	4022
D 2698	4032
D 2698	4052
D 2794	6226
D 2800	7501
D 3272	7355
D 3539	4492
E 28	4495
E 97	6121
G 23	6151
G 23	6152

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January 24, 1986

SECTION 7.

NOTES ON THE USE OF THIS STANDARD

1. **PRODUCT SPECIFICATION.** Product specifications for paint, varnish, lacquer, and related materials shall specify by number which method shall be used to test for a particular property. Many of the methods in this standard give desired conditions for the test involved, in which case, assuming they are applicable to the product under test, no further information need be specified. In some instances, the specification shall stipulate the test conditions, properties to be checked (if there are more than one), and the test periods involved.

2. **SOURCE FOR SECURING FEDERAL SPECIFICATIONS AND STANDARDS**

Activities outside the Federal Government may obtain copies of Federal Specifications, Standards, and Handbooks as outlined under General Information in the Index of Federal Specifications, Standards, and Commercial Item Descriptions and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications, Standards, and Commercial Item Descriptions from established distribution points in their agencies.

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SECTION 8

REAGENTS, WATER, AND ETHYL ALCOHOL

1. SCOPE. This section covers the general requirements for the purity of reagents and water used in the test methods of this standard. Detailed information for a particular reagent is given in the method in which it is used.

2. REAGENTS. Unless otherwise indicated, all reagents shall conform to the American Chemical Society specifications for reagent chemicals, published in "Reagent Chemicals, ACS Specifications," Fifth Edition (1974), and available from the American Chemical Society, 1155 Sixteenth Street, N.W., Washington, D.C. 20036, telephone (202) 872-4600. Where such specifications are not available, commercial chemicals having the highest purity shall be used, and if necessary, these chemicals shall be analyzed before use by proper analytical methods.

3. WATER. Unless otherwise indicated, references to water for use in the preparation of reagents and in the analytical procedures shall be understood to mean freshly distilled water.

4. ETHYL ALCOHOL. Ethyl alcohol shall be either absolute ethanol (100 percent or 200 proof), 95 percent ethanol (containing 5 percent of water, 190 proof), or alcohol denatured according to formulas of the Internal Revenue Service (see 5). The particular grade of ethanol to be used shall be specified in each method.

5. DENATURED ALCOHOL. Denatured alcohol corresponding to the following formulas is prepared by adding the designated denaturant to 100 gallons of 95 percent (190 proof) ethyl alcohol:

<u>Formula Number</u>	<u>Denaturant (gallons)</u>
1	wood alcohol (5)
2B	benzene (0.5)
3A	methyl alcohol (5)
12A	benzene (5)
13A	ethyl ether (10)
23A	acetone (10)
28	benzene (10)
30	methyl alcohol (10)
32	ethyl ether (5)
35	ethyl acetate (35)
35A	ethyl acetate (5)

6. NBS STANDARD REFERENCE MATERIALS. Information, prices, and ordering instructions for NBS standard reference materials are given in NBS Special Publication 260, entitled "Catalog of NBS Standard Reference Materials", available from the National Bureau of Standards, Office of Standard Reference Materials, Chemistry B-311, Washington, D.C., 20234; telephone (301) 921-2045.

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SECTION 9

ROUTINE AND REFEREE TESTING CONDITIONS

1. CONDITIONING AND TESTING

1.1 Routine testing. Unless otherwise specified in the product specification or test method, all physical tests on coating materials or dried test films thereof shall be made at 21 to 32° C (70 to 90° F).

1.2 Referee testing. In case of dispute or disagreement between laboratories, and unless otherwise specified, the coating material or test film thereof shall be conditioned in an atmosphere of 50 \pm 4 percent relative humidity and at a temperature of 23 \pm 1° C (73 \pm 2° F). It is intended that the laboratory be maintained as closely as possible to 23° C (73° F) and a relative humidity of 50 percent. The tolerances indicated are meant to cover variations from the specified conditions which may occur at different locations in the room.

1.3 Time of conditions. Unless otherwise specified in the product specification or test method, the coating material or test film thereof and the testing equipment shall be allowed to reach equilibrium and then tested at the conditions specified in 1.2.

1.4 Lighting and ventilating. Unless otherwise specified, lighting and ventilating conditions shall be those normally maintained in a room used for paint testing purposes.

MILITARY INTEREST:

Custodians

Army - ME
 Navy - SH
 Air Force - 20

Review Activities

Army - ER, CR, MI, CE
 Navy - YD, AS

User Activity

NAVY - MS
 DLA - ES

CIVIL AGENCY COORDINATING ACTIVITIES:

GSA
 HCC
 OSS
 NBS

PREPARING ACTIVITY:

Army - ME
 Project 8010-1064

METHOD 1011
January 24, 1986

INSPECTION (GENERAL)

1. DUTIES OF GOVERNMENT INSPECTOR.

1.1 The duties of the Government inspector are varied and depend on the inspection requirements of the order or contract. The requirement may be any one of the following:

1.1 Inspection during process of manufacture. In this type of inspection, the Government inspector may be required to witness all steps in the processing operations, take samples of raw materials used, sample the finished product, and witness the filling of the containers. He may conduct or witness tests and analyses on the samples at the manufacturer's plant or in a laboratory arranged for by the manufacturer, and then accept or reject the batch involved, or he may send the samples to a Government laboratory for analyses and tests. Material is often held in tanks until tests on the representative sample are completed. Containers are filled only after the Government inspector signifies that tests are satisfactory. The Government inspector then may be required to witness the filling, determining that the material has been properly mixed, and ascertain that the specified containers are properly filled, marked, and shipped.

1.2 Inspection prior to shipment. In this type of inspection, materials are generally packaged and ready for shipment at the time of arrival of the Government inspector. However, in some instances when large amounts of material are involved, the manufacturer may not fill containers, but will hold the batch in a large tank until the Government inspector arrives. All the duties of 1.1 other than witnessing the processing operations may apply.

1.3 Inspection at destination. In this type of inspection, packaged materials which have arrived at destination are to be examined and sampled.

1.4 Special assignments. Inspection may also include the performance of any special assignment under the applicable product specification. Typical of these are the inspection of the contents of a representative sample of paint to determine whether hard settling has taken place in the container, whether the material has obviously deteriorated to the point of uselessness, and similar acts. In the case of products purchased in concentrated form which must be thinned for use, the Government inspector may be required to transmit the manufacturer's thinning directions to the test laboratory if a sample is taken from a large container marked with such directions.

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METHOD 1022
January 24, 1986

SAMPLING FOR INSPECTION AND TESTING

1. SCOPE

1.1 This method describes sampling procedures for inspection and testing of paints, varnishes, lacquers, solvents, and related products, and the procedures for the acceptance or rejection of these products.

2. DEFINITIONS

2.1 Inspection. As used in this standard, inspection refers to visual observation, and to facile and rapid measurement, which requires no equipment or only very simple equipment (scale, meter stick, etc.), and which does not necessitate destruction of the packaging nor alteration of the product.

Examples of inspection include examination of preparation for delivery, condition in containers, number of units involved, and type, class, grade, color, or other visual considerations of the units as may be called out in the product specification.

2.1.1 Types of inspection.

2.1.1.1 Inspection during process of manufacture. In this type of inspection, the inspector may be required to witness all steps in the processing operations, take samples of raw materials used, sample the finished product, and witness the filling of the containers. He may conduct or witness tests and analyses on the samples at the manufacturer's plant or in a laboratory arranged for by the manufacturer, and then accept or reject the batch involved, or he may send the samples to a laboratory for analyses and tests. Material is often held in tanks until tests on the representative sample are completed. Containers are filled only after the inspector signifies that tests are satisfactory. The inspector then may be required to witness the filling, determining that the material has been properly mixed, and ascertain that the specified containers are properly filled, marked, and shipped.

2.1.1.2 Inspection prior to shipment. In this type of inspection, materials are generally packaged and ready for shipment at the time of arrival of the inspector. However, in some instances when large amounts of material are involved, the manufacturer may not fill containers, but will hold the batch in a large tank until the inspector arrives.

2.1.1.3 Inspection at destination. In this type of inspection, packaged materials which have arrived at destination are to be examined and sampled.

2.1.1.4 Special assignments. Inspection may also include the performance of any special assignment under the applicable product specification. Typical of these are the inspection of the contents of a representative sample of paint to determine whether hard settling has taken place in the container, whether the material has obviously deteriorated to the point of uselessness, and similar

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METHOD 1022

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acts. In the case of products purchased in concentrated form which must be thinned for use, the inspector may be required to transmit the manufacturer's thinning directions to the test laboratory if a sample is taken from a large container marked with such directions.

2.2 Test. As used in this standard, test refers to a measurement of physical or chemical characteristic which requires specialized equipment, trained analysts, or a long period of time, and which may or may not necessitate alteration of the product. Examples of tests include viscosity, working properties, drying times, adhesion, and all others that are called out in the product specification.

2.3 Delivery. Unless otherwise specified, this represents the quantity of material of the same units, type, class, grade, color, composition, etc., purchased for delivery at one specified time.

2.4 Lot. A lot is a delivery, a portion of a delivery, or a multiple of deliveries composed of material of the same type, class, grade, composition, color, etc. A lot is rejected or accepted as a whole on the basis of inspection, test, or both, carried out on a sample or samples from a lot. Unless otherwise specified, a lot of various materials shall be defined as follows.

2.4.1 Liquid paints, enamels, resin solutions, removers, varnishes, drier solutions, pastes (in bulk), and similar products: A lot shall be a manufacturer's single batch, i.e., all raw materials processed at one time or in one operation or sequence of operations to a finished product.

2.4.2 Solvents, oils, hard resins, dry powders or pigments, and materials packaged in tubes or Semkits (of one or two components), cartridges, or pressurized (aerosol) dispensers: Inasmuch as the individual batch identity may be lost or of little significance, lot definition for these products shall be the same as that for "delivery" (see 2.3).

2.4.3 Liquids in bulk. Where procurements are made for bulk shipments of liquids, each tankcar shall be an individual lot.

2.4.4 Size. For purposes of sampling, and unless otherwise specified the lot shall be as large as possible, subject to the considerations above, and of such size as to permit representative sampling in accordance with MIL-STD-105 (see 5.1).

2.4.5 There may be occasions wherein lot formation will not conveniently fall within the above definitions. In such cases, sound judgment must be made as to lot formation, using the above as guidelines in such manner as to be commensurate with the provisions of MIL-STD-105. The lot shall be so formed that its components are easily accessible for sampling in accordance with the specified plan.

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2.4.6 Change in lot formation. No material shall be added to or removed from a lot once the inspector has begun to draw samples therefrom. However, if the inspector notices some damaged units (by accident or other circumstances not related to the manufacturing process) before beginning to sample, and if the damage is of such character that it is apparent to him that no other similarly damaged units are distributed throughout the lot, then the inspector shall have this damaged material removed from the lot before drawing a sample.

2.5 Unit. For purposes of sampling, and unless otherwise specified, lots shall be expressed in units as follows.

2.5.1 Liquids: U.S. gallons.

2.5.2 Pigments, powders, hard resins, highly viscous pastes in bulk, etc.: pounds (avoirdupois).

2.5.3 Materials packaged in tubes, pressurized (aerosol) dispensers, Semkits, cartridges, etc.: Each container shall be a separate unit. Where tubes, cartridges, Semkits, etc., are parts of multiple-component kits, each kit shall be a separate unit.

3. SAMPLING PLANS

3.1 Sampling plans, inspection levels, and acceptable quality levels (AQL) (see 5.1). For inspection and tests, each lot (see 2.4) shall be sampled in accordance with MIL-STD-105.

3.2 Inspection.

3.2.1 Inspection of preparation for delivery. Unless otherwise specified, each lot of material, in units packaged for delivery, shall be inspected in accordance with PPP-P-1892 (see 5.2). Inspection of each lot shall be made in accordance with MIL-STD-105, inspection level I and an AQL of 1.5 percent defective.

3.2.2 For end item inspection of material not involving tests, other than as in 3.2.1, and unless otherwise specified, each lot shall be inspected in accordance with MIL-STD-105, inspection level S-2, with an AQL of 4.0 percent defective.

3.2.3 Liquids in bulk (see 2.4.3) do not require inspection as described in 3.2.1 and 3.2.2, but inspection shall be made of tankcar or transport vehicle to ensure that appropriate DOT shipping markings are present (see 5.3).

3.3 Tests. Unless otherwise specified, sampling of the end item for tests shall be as follows.

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3.3.1 For most materials, each lot shall be sampled in accordance with MIL-STD-105, inspection level S-2 and AQL of 2.5 percent defective.

3.3.2 For materials in pressurized (aerosol) containers, each lot shall be sampled in accordance with MIL-STD-105, inspection level I, and AQL of 1.5 percent defective.

3.3.3 Materials in bulk (see 2.4.3) do not lend themselves to sampling as described in 3.3.1 and 3.3.2 (see 4.5.5).

4. PROCEDURE

4.1 General considerations.

4.1.1 Samples are taken for the purpose of having representative quantities, of economical and convenient size, from each lot of material for inspection and tests, so that it may be ascertained if the materials meet the requirements of the product specification, and also to determine uniformity within a lot that has been packaged prior to inspection.

4.1.2 No set of directions for sampling, however explicit, can take the place of judgment, skill, and previous experience on the part of persons actually engaged in the sampling and in the supervision of the sampling. These directions are intended to supplement this experience and particularly to serve as a guide in the selection of the method which is to be used in common by each of two or more contracting parties.

4.2 Method of sampling. The lots, assembled as described in 2.4, shall be sampled according to the applicable plan described in paragraph 3. For lots of materials, samples shall be selected at random so that they are representative of the lot.

4.3 Sample size. The sample shall be of such size as to permit the performance of all inspections and tests. In most cases, 1 quart of liquid or 1 pound of dry material is sufficient.

4.4 Original containers. To the extent possible, it is advisable that original, unopened containers within each lot be selected as samples. When individual containers are less than the 1-quart, 1-pound, or other required size, a sufficient number of containers shall be selected to equal the required size. Obviously it is not always convenient or economical to have samples of very large size, such as 5 gallons, 50 pounds, or even 1 gallon, if the lot is small or the materials relatively expensive. In these cases, care must be exercised so that samples are uniform and representative of the lot of material (see 4.5).

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4.5 Uniformity of samples.

4.5.1 Clear liquids filled for delivery. Clear liquids, such as oils, varnishes, solvents, drier solutions, etc., shall be stirred so as to be uniform. Care must be taken so that any separation, sediment, gel, or other matter indicative of nonuniformity is included in the sample.

4.5.2 Pigmented liquids in filled containers. These materials should be stirred thoroughly so as to be uniform. Where there is settling, or separation of constituents, these should be reincorporated by "boxing." This consists of having a clean container of the same size, into which is poured the liquid portion from the container being sampled. The remaining settled portion is stirred into a thin paste as uniform as possible. The removed liquid is then poured slowly and with stirring back into the original container. The contents are finally poured back and forth from container to container until a representative sample can be taken.

4.5.3 Materials in drums. Solvents, clear coatings, paint removers, etc., in drums may be mixed to uniformity by rolling, inverting, or rocking the drums. Pigmented coatings in drums do not conveniently lend themselves to "boxing" as described in 4.5.2, and an alternative sampling procedure must be considered (see 4.5.7). The same applies for pastes in unit containers larger than 1 gallon.

4.5.4 Hot plastic materials. These coatings, whether pigmented or not, must be melted by heat and stirred thoroughly before being sampled. This is inconvenient and possibly dangerous. If these coatings are packaged in units larger than 1 gallon, an alternative sampling procedure (see 4.5.7) must be considered.

4.5.5 Clear liquids for bulk shipment. Liquids in bulk such as solvents, oils, varnishes, paint removers, etc., are normally in tankcars or storage tanks and should be made as representative as possible by agitation or circulation. Then, if possible, samples should be taken from the top, middle, and bottom of the tank.

4.5.6 Dry materials. Ordinarily pigments, powders, hard resins, etc., are more likely to be uniform than pigmented liquids. Care must be exercised, however, to ensure that samples of these materials are representative of the lots being inspected and tested.

4.5.6.1 Pigments and powders. Small containers, such as those about 1 gallon in volume, should be "boxed" as described in 4.5.2. Since moisture content and size of agglomerates may vary in large containers, samples should be taken from different parts of the containers. For sampling very large containers of these materials, an alternative sampling procedure (see 4.5.7) should be considered.

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4.5.6.2 Hard resins. Where these materials are in lumps, samples should be representative of the various size lumps observed. Where the resin is a solid mass in a container, it may be sampled as described for hot plastics in 4.5.4. If heating is known to change the composition of the material, an alternative sampling procedure (see 4.5.7) should be considered.

4.5.7 Alternative sampling procedure. Where it is impractical, inconvenient, or dangerous to take samples as described above, and where permitted, samples may be taken in the manufacturer's plant during filling operations or production line as applicable. In such cases samples should be taken near the beginning, in the middle, and near the end of the operation. Sampling in this manner must be supervised by a representative of the purchaser or by an individual delegated by that representative.

4.5.8 Pressurized (aerosol) dispensers. For obvious reasons samples cannot be taken from coatings packaged in pressurized dispensers. The filled containers shall be taken as samples in accordance with 2.4.2., 2.5.3, 3.1, 3.2.1, and 4.4. The filled containers are subject to inspections as required by the product specifications. Also, product specifications normally permit some tests to be performed on the coating concentrate prior to its packaging. Unless specifically stipulated, however, coating concentrates do not constitute samples for inspection and tests as defined herein.

4.5.9 Composite samples. Composites of the samples taken as directed herein shall not be used in inspections as here defined, inasmuch as inspection is intended for observing conditions of products as they exist physically. While not recommended, composites may occasionally be permitted for economy in testing. Where permitted, however, a composite must be representative only of an individual lot of material.

4.6 Disposition of samples. Unless otherwise specified (see 4.4 and product specification), each sample taken as directed herein shall be sealed in a clean, dry container and marked so as to clearly identify the lot of material involved. Unless otherwise specified, each sample shall be inspected and tested in accordance with the product specification. Each inspection and test value, as well as the average value, for each sample shall be reported, and failure of any sample to meet all requirements in the product specification shall be cause for rejection of the sample.

4.7 Termination of sampling. When, in the course of sampling, the material is found to have serious obvious defects (see method 3011 et. al.), sampling shall be terminated and resumed only after defects have been corrected or the defective material replaced.

4.8 Time of sampling. Samples shall be taken during or as soon as possible after manufacture of the material.

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5. NOTES

5.1 Military standard: MIL-STD-105, Sampling Procedures and Tables for Inspection by Attributes.

5.2 Federal specification: PPP-P-1892, Paint, Varnish, Lacquer, and Related Materials; Packaging, Packing, and Marking of.

5.3 Code of Federal Regulations, 49 CFR 171--177, Department of Transportation, Hazardous Materials Regulations.

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CONTRACTOR INSPECTION RESPONSIBILITY

1. SCOPE.

1.1 This method covers the requirements for contractor inspection responsibility as outlined below:

2. QUALITY CONTROL PROVISIONS

2.1 Inspection and testing. The contractor is responsible for the performance of all inspection and testing requirements as specified in the specification for the commodity. Except as otherwise specified, the contractor may utilize his own or any other inspection and testing facilities and services acceptable to the Government. Records of the examination and tests shall be kept complete and available to the Government. The Government reserves the right to perform any of the inspection and testing set forth in the specification and contractual documents where such is deemed necessary to ensure that supplies conform to prescribed requirements.

2.2 Quality control system. The contractor shall maintain an effective quality control system. This system shall provide for adequate controls of quality throughout all areas of contract performance. Descriptive procedures shall be provided and maintained to illustrate the entire process of receipt and handling of ingredient materials, and the manufacture, inspection, testing, packaging, packing, marking, and shipping of finished product. The contractor shall provide for or maintain at least a minimum of accurate laboratory testing equipment in satisfactory operating condition sufficient to perform the required acceptance tests and to ensure that the products conform to contract requirements.

3. EXTENT OF TESTING

3.1 Over 250 gallons or 2,000 pounds, as applicable (see 8.1). The contractor shall have all acceptance tests made as required by the specification and the contract and shall submit the laboratory test report when the contract or order provides for more than 250 gallons or 2,000 pounds of one material. When the contractor has a past history of good quality control as determined by the Government quality control activity:

- a. With 90 percent of the quantity completely conforming to specification requirements;
- b. With the 10 percent of the quantity nonconforming having only minor discrepancies with no gross divergence from specification requirements;
- c. With reasonable conformity with Government test results;

then testing of up to four lots in five of the same material, regardless of contract or order, may be reduced to that of 3.2.

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3.2 50 - 250 gallons or 400 - 2,000 pounds. Reduced testing will be permitted on contracts of 50 gallons or 400 pounds and over but not exceeding 250 gallons or 2,000 pounds, provided the formulation used has been shown to comply with all requirements by prior testing. For paints and similar pigmented and clear products, the submitted test report shall cover the following characteristics, as applicable:

- a. Application properties.
- b. Drying or curing time.
- c. Appearance of dry film (gloss, color, etc.).
- d. Opacity.
- e. Fineness of grind.
- f. Weight per gallon.
- g. Viscosity.
- h. Percent nonvolatile.
- i. Strength properties (adhesives).

For other materials, test may be reduced to generally accepted plant control procedures and other tests indicative of quality that can be completed within 2 days time with common laboratory equipment.

3.3 Under 50 gallons or 400 pounds. When the quantity is less than 50 gallons or 400 pounds, the contractor may furnish a certificate indicating compliance with all requirements of the specification in lieu of a test report, provided the formulation used has been shown to comply with all requirements by prior testing. Unless otherwise specified in the contract or order, material furnished shall have a 1-year unqualified shelf life beginning with date of manufacture marked on container, and must be guaranteed usable for the intended purpose after normal mixing, stirring, or shaking.

3.4 If at any time Government testing indicates significant differences when checked against the contractor's test reports, the contractor will be advised and his quality control system will be considered to be in a noncomparable status. He will then be required to submit laboratory tests on a more complete testing basis on each lot, dependent on the nature of the difference, until comparability is reestablished. This action shall be effective regardless of material then being manufactured or the contract under which it is being procured.

4. PAINT CHIP REQUIREMENT

4.1 For each lot of all coatings, the contractor shall furnish the Government representative a coated card which shows the color, gloss, and general appearance of the material covered by the lot. The card shall be no smaller than 3 by 5 inches. For acceptance, the color must match, within specification limits, the color card furnished for the contract or order by the Government representative.

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5. TESTING PROCEDURES AND REPORTS

5.1 All tests performed shall be in accordance with the applicable specifications. The contractor is cautioned to refer to and to comply with all modifications of the specifications cited in the contractual documents. If testing is not done by the contractor, it shall be his responsibility to furnish his designated testing facility with all pertinent contract information and modifications to make certain that the testing establishment is adequately informed to accomplish all tests and make accurate reports. Quantitative test results shall be reported to the same number of significant digits as are used in stating the requirements of that property in the commodity specification. Qualitative values shall be definitely stated. Results should not be reported simply as "complies" or "satisfactory," but in the same manner as the requirements. The test methods shall be reported by reference to the applicable paragraph in the product specification or the method number when referenced to a Federal or other test method standard.

6. INGREDIENTS--CERTIFICATE OF COMPLIANCE

6.1 When the specification has mandatory ingredient requirements and the quantity of finished product is 50 gallons or 400 pounds or over, the contractor shall make available to the Government representative a certificate of compliance from the ingredient supplier for each ingredient, as applicable. The certificate of compliance shall cite the data or the location and availability of data, i.e. the quality control data upon which the certificate is based. For frequently replenished ingredients, normally mixed, blended, or combined in tanks or bins, the contractor shall make available a record of ingredient supplier's certificate of compliance for each replenishment. For products of critical end use such as ship-bottom paints, ingredients shall be sampled and subjected to verification tests in a Government laboratory prior to start of manufacture, when so required by the specification.

7. SAMPLES

7.1 Unless otherwise specified, sampling shall be in accordance with Method 1022.

8. NOTES

8.1 While most paints and related products are bought by volume (gallons, etc.), some products such as pigments, hard resins and, possibly, pastes are bought by weight (pounds, etc.). The weights called out in this method are considered convenient and representative amounts or lots for determining the extent of testing required. No specific mathematical relationship is implied by the gallons and pounds called out in this method.

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Section 2000

PREPARATION OF PANELS AND APPLICATION OF TEST FILMS

METHOD 2011.2
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PREPARATION OF STEEL PANELS

1. SCOPE

1.1 This method prescribes the type of steel panels to be used and the procedures to be followed in their preparation for testing paint, varnish, lacquer, and related products.

Four procedures of panel preparation are covered, as follows: Procedure A, Panels for Testing on Steel without Chemical Treatment. Procedure B, Panels for Testing on Phosphoric Acid-Cleaned Steel. Procedure C, Panels for Testing on Chemically Treated Steel. Procedure D, Panels for Testing on Mechanically Cleaned Steel.

2. EQUIPMENT

2.1 Test panels. The test panels shall be prepared from new cold-rolled steel, rust-free No. 20 gage (0.0375 in.), unless otherwise agreed upon between the purchaser and the supplier, but in any case, not thinner than No. 24 gage (0.025 in.). The steel shall be cold-rolled carbon steel strip conforming to ASTM A 109 and shall have a Rockwell Hardness of 55 to 75 on the "B" scale. It shall have a surface roughness of 30 to 50 microinches (arithmetical average) as rolled. Flat polishing of this steel surface to a 15-to-20-microinch (arithmetical average) maximum shall be allowed (note 1). The steel shall have been coated at the mill with a suitable rust-preventive compound for shipment or storage. After cutting the steel into test panels, the coating shall be removed as described in procedure A. Supplier-cleaned panels shall be wrapped in a paper impregnated with dicyclohexylammonium nitrate, or its equal, and then sealed in a waterproof bag or envelope. If recoating the cleaned panel with a suitable rust-preventive compound is required, then the inner wrap may be a neutral pH paper or polyethylene. The panels shall be not less than 2-3/4 by 5-7/8 inches in size. Edges and corners shall be smooth and rounded.

Note 1. Flat polishing is a method of uniformly polishing the steel surface with abrasive belts mechanically to remove surface contaminations that may influence the uniformity of test results obtained with steel test panels. To insure complete removal of surface contamination, the original surface shall be completely removed as determined by visual inspection; in any case, a minimum of 0.0003 inch of surface shall be removed. Care must be used in the operation of the polishing apparatus and after cleaning the test panel to insure that grit and steel particles are not left on the polished surface.

3. SOLVENTS AND REAGENTS

3.1 Denatured alcohol (95 percent). Specially denatured alcohol corresponding to any of the following formulas of the U.S. Bureau of Internal Revenue; SD 2-B, SD 3-A, SD 12-A, SD 13-A, SD 23-A, SD 28, SD 30, SD 32, SD 35, or SD 35-A.

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3.2 Ammonium hydroxide (sp gr. 0.90). Concentrated ammonium hydroxide (NH_4OH).

3.3 Methyl alcohol (methanol), conforming to the specifications for methyl alcohol (A.C.S grade).

3.4 Phosphoric acid (1:1). Mix equal volumes of concentrated phosphoric acid (H_3PO_4 , 85 percent) and distilled water.

3.5 Solvent mixture. Prepare a solvent mixture consisting either of equal volumes aliphatic naphtha (TT-N-95) and xylene (ASTM D 845) or of three volumes of naphtha and one volume of propylene glycol monomethyl ether.

Note 2: Other combinations of organic solvents that will produce a surface equal in cleanliness to the solvent mixtures mentioned in 3.5 may be used if agreed upon by the purchaser and the supplier.

3.6 Trichlorethylene conforming to and maintained in accordance with the specification for trichlorethylene (O-T-634).

4. PROCEDURE

4.1 Procedure A. Panels for testing on steel without chemical treatment. Wipe rust free panels vigorously on both sides with a cloth wet with the solvent to remove the gross film of oil. Clean the panels as described in 4.1.1, solvent spray; 4.1.2, vapor degreasing; or 4.1.3, solvent brush. Spot sanding of the test panel surface is not permitted.

4.1.1 Solvent spray cleaning. Spray the solvent mixture (see 3.5), downward at an acute angle to the panel surface, using a spray gun operated with the fluid tip wide open and the atomizing tip almost completely closed. Preferred practice is to support the panels on a rack at an angle of approximately 25 degrees from the vertical and to hold the spray gun so that the jet is directed vertically downward. The bottoms of the panels should rest upon projections, such as nails, that permit the solvent to drain from the bottom edge. Direct the solvent over the surface of the panel in an oscillating rinsing manner, moving the spray progressively from the top to the bottom of the panel until all soluble and loosely adhering soil has been washed off. The minimum treatment shall consist of five washings, three on the side to be tested, and two on the reverse side. Alternate the washings, starting and ending with the side to be tested. Allow the panels to dry at room temperature until all solvent has evaporated (approximately 15 min.). Then rub the panels briskly with a clean, lintless cloth to remove any adhering dirt or smut. After this treatment, dip 1 panel of each 20, momentarily, into distilled water. If it does not retain a continuous film (that is, shows a water break), repeat the entire cleaning process (note 3). Then coat the clean panels immediately or store them in a

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desiccator until needed. Handle supplier-cleaned panels as clean panels when the original exterior waterproof wrapper is not broken. Do not touch the panels with the fingers during the complete process of preparation.

Note 3: It may sometimes be necessary to immerse the panels in methyl alcohol to obtain a water break free surface. When this is required, immerse the panels in methyl alcohol, remove, dry at a temperature of 125° to 200° F, and allow to cool to room temperature before coating or storing.

4.1.2 Vapor degreasing. Suspend the panels in a vapor phase-type degreaser containing stabilized trichloroethylene and allow them to remain above the boiling solvent until they attain the temperature of the vapor, at which time condensation on the panels no longer occurs. Allow the panels to cool to room temperature, rub briskly with a clean, lintless cloth, immerse in methyl alcohol, dry, and check for water break as described in 4.1.1.

4.1.3 Solvent brush cleaning. Power brush scrub the panel with the solvent mixture (see 3.5) until all soluble and loosely adhering soil has been washed off; then flush with clean solvent. Allow the panels to dry at room temperature or force dry at a temperature no higher than 300° F. Allow the panels to cool to room temperature, rub the panels briskly with a clean, lintless cloth, and check for water break as described in 4.1.1.

4.2 Procedure B. Panels for testing on phosphoric acid cleaned steel unless otherwise specified shall be treated per 4.2.1.

4.2.1 Phosphoric acid coating. Clean standard rust-free panels as described in procedure A, including the rubbing with a clean, lintless cloth. After rubbing, dip the panels in phosphoric acid (1:1) at 80° +5° F for 1 minute (note 4). Rinse the panels immediately by spraying with distilled water for a minimum of 15 seconds and note whether a continuous film is formed. Spray all panels that retain a continuous film (that is, show no water break) once with 95 percent denatured alcohol to which has been added 1 percent, by volume of ammonium hydroxide (sp. gr. 0.90). Dry the panels at a temperature between 125° and 200° F and coat them immediately or store them in a desiccator until needed. Do not touch the panels with the fingers during the process of preparation.

Note 4. Do not use phosphoric acid solution to clean more than 100 square feet of panel surface per liter of solution.

4.2.2 Phosphoric acid etch. Clean standard rust-free panels as described in procedure A including the rubbing with clean lintless cloth. After rubbing, immerse the panels in phosphate cleaner of the following composition at room temperature for 1 minute.

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Formula:

3 milliliters of A.C.S. reagent grade phosphoric acid.
1 milliliter Tergitol Penetrant No. 4 (Union Carbide and Carbon Corporation).
Water to make 1,000 milliliters.

Rinse the panels under running warm water and immerse for 1 minute in the solution of chromic acid 0.1 percent at $85^{\circ} \pm 5^{\circ} \text{ C}$ ($185^{\circ} \pm 9^{\circ} \text{ F}$). Blot panel dry with absorbent paper or cloth and heat in an oven at $105^{\circ} \pm 2^{\circ} \text{ C}$ ($221^{\circ} \pm 3.6^{\circ} \text{ F}$) for 5 minutes. Cool to room temperature and coat the panels immediately or store them as in procedure A until needed.

4.3 Procedure C. Panels for testing on chemically-treated steel.

4.3.1 Use standard rust-free panels. Coat the panels with a phosphate coating or other conversion coating representative of production performance. The conditions of treatment shall be as agreed upon between the purchaser and the supplier.

4.4 Procedure D. Mechanical cleaning.

4.4.1 The steel panel surface shall be buffed by means of 240 grit aluminum oxide abrasive with cloth or paper backing so as to produce a smooth surface of 10 to 20 microinches root mean square (r.m.s.). The use of wet or dry cloths or wet or dry papers is prohibited. The final abrasion marks shall be in the direction to the length of the panel. The sanded panel shall be further cleaned to remove dust from the abrasion operation using clean, dry absorbent tissue or cloth and then cleaned as per procedure A. When specified, this may be followed by procedure B or C, or other chemical treatment.

METHOD 2012.2
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PREPARATION OF TIN PANELS

1. SCOPE

1.1 This method prescribes the type of tin panels to be used and the procedure to follow in their preparation for testing paints, varnish, lacquer, and related products.

2. EQUIPMENT

2.1 Test panels general. The finished tinned panel shall conform to the requirements of QQ-T-425 for class A2 (best coke), grade 1, 31 gage ICL tin plate having a base weight per base box of 100 pounds and equivalent weight per square foot of 0.44 pound.

2.2 Flexibility tests. The tinplate specified in 2.1 shall be made by plating manufacturer's standard gage No. 31 (0.0107 inch), soft skin rolled temper (condition 4) steel sheet of nonaging quality conforming to ASTM A 109.

2.2.1 Bending property. Panels (3 to 5 inches) cut at random from the plated sheet, when bent through an arc of 180 degrees over mandrels from 1/8 to 1 inch in diameter, shall conform to the circumference of each mandrel without any deformation such as bulging, buckling, or breaking.

3. CLEANING SOLUTIONS

3.1 Solvent mixture. Prepare a solvent mixture consisting either of equal volumes of aliphatic naphtha (TT-N-95) and xylene (ASTM D 845) or of three volumes of aliphatic naphtha and one volume of propylene glycol monomethyl ether.

3.2 Trichlorethylene conforming to and maintained in accordance with the specification for trichlorethylene (O-T-634)

Note 1: Other combinations of organic solvents that will produce a surface equal in cleanliness to the solvent mixtures mentioned in 3.1 may be used if agreed upon by the purchaser and the supplier.

4. PROCEDURE

4.1 The panels shall be cleaned by any method in procedure A of 2011.

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METHOD 2013.1
January 24, 1986

PREPARATION OF ALUMINUM ALLOY PANELS

1. SCOPE

1.1 This method covers the treatment necessary for preparation of aluminum or aluminum-alloy surfaces for painting.

2. EQUIPMENT

2.1 Test panels.

2.1.1 Type of metal. The metal shall conform to either QQ-A-250/4, Aluminum Alloy 2024, Plate and Sheet; QQ-A-250/5, Aluminum Alloy Alclad 2024, Plate and Sheet; QQ-A-250/12, Aluminum Alloy 7075, Plate and Sheet; QQ-A-250/13, Aluminum Alloy Alclad 7075, (Aluminum - 5.6 Zinc - 2.5 Magnesium - 1.6 Copper) Plate and Sheet; or other as specified in the product specification.

2.1.2 Thickness of metal. The metal shall be of the thickness specified, but in no case less than 0.020 inch.

2.1.3 Size of panel. The test panels shall be cut to the specified size, which shall be not less than 2-3/4 by 5-7/8 inches (3- by 6-inch size preferred). All edges and corners shall be smooth and uniformly rounded.

2.1.4 Panels for exterior exposure. Panel dimensions shall be as specified. Panels often used for exterior exposure are of the following sizes, designated as size a or size b:

Size a. Approximately 5 inches wide, 16 inches long, and from 0.032 to 0.040 inch thick.

Size b. Approximately 4 inches wide, 12 inches long, and from 0.032 to 0.063 inch thick.

When cleated panels are specified they shall conform to the requirements of figure 1.

3. REAGENTS AND CHEMICALS

As indicated in section 4.

4. PROCEDURE FOR CLEANING AND PREPARATION OF TEST PANELS

4.1 Preliminary cleaning. All panels shall be given a preliminary cleaning by vapor degreasing or solvent washed as in 2011; and then be treated in accordance with 4.2 or 4.3 as specified in the product specification. Chlorinated hydrocarbons and high pH material shall not be used.

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4.2 Panels to be anodized. Immerse the panels in a hot solution of mild inhibited alkaline cleaner for the specified time and at the specified temperature, and then thoroughly rinse with water. The panels shall then be anodized in accordance with MIL-A-8625 or the method otherwise specified. The following methods have been found satisfactory for cleaning treatment.

a. Immerse for 5 to 10 seconds at a temperature of 150° to 160° F in 6.7 ounces per gallon (50 grams per liter) of caustic soda solution saturated with fluosilicate.

b. Immerse for 10 minutes at just below boiling in a solution of 4 ounces per gallon of the concentrate given in table I.

TABLE I. Cleaner concentrate

Ingredient	Percent by weight
Sodium metasilicate	41
Sodium trisilicate (soluble)	54
Synthetic soap	5

4.3 Panels not to be anodized.^{1/} For control^{2/} or routine testing, aluminum alloy panels will require no other preparation than that specified in 4.1. Clad aluminum alloy panels shall be further treated with an alcoholic phosphoric acid cleaner of the composition given in table II. The alcoholic phosphoric acid cleaner may be applied by scrubbing the panels with a bristle brush for a period of 2 minutes or by dipping for the same period of time. The cleaner is then removed by rinsing thoroughly with water and drying.

TABLE II. Alcoholic-phosphoric acid cleaner.

Ingredient	Percent by weight
Butyl alcohol	35
Water	22
Isopropyl alcohol	25
Phosphoric acid (85 percent by weight)	18

^{1/} Unless otherwise specified, for use, panels shall be prepared by cleaning with alcoholic phosphoric acid and water rinsing as described in table II, and then shall be given a chemical film conforming to MIL-C-5541 applied to the panels in compliance with the directions of the manufacturers.

^{2/} When the behavior of the sample is to be compared with that of a control material, all test panels shall be prepared in the same manner.

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METHOD 2021
January 24, 1986

PREPARATION OF GLASS PANELS

1. SCOPE

1.1 This method covers the preparation of glass panels used to evaluate quality of coatings.

2. EQUIPMENT

2.1 Type of glass.

2.1.1 Window glass, the surface of which is reasonably plane and free of irregularities.

2.1.2 Clear plate glass not less than 3/16-inch thick.

2.1.3 Plate glass that has been ground uniformly on one side with 1F carborundum.

2.1.4 Black carrara glass, one side of which has been polished to a smooth, high-gloss surface.

2.1.5 White carrara glass, one side of which has been polished to a smooth, high-gloss surface.

2.2 Test panels.

2.2.1 Size. Since the nature of the test governs the type and size of the test panel to be used, this information shall be included in the specific test method. Three- by six-inch panels are recommended.

3. REAGENTS

3.1 Methyl alcohol, A.C.S. reagent grade.

4. PROCEDURE

4.1 Cleaning.

4.1.1 New panels. The panels shall be cleaned by means described in 2011 until no water break occurs. Absence of water break is determined by immersing the panels in A.C.S. reagent grade methyl alcohol (methanol), removing and drying the panels at 52° to 93° C (125.6° to 199.4° F). After the panels have been cooled to room temperature, they shall be dipped momentarily into distilled water and allowed to drain in a vertical position. While draining, the panels shall be covered with a continuous film of water (that is, show no water break).

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Again dip the panels in the alcohol, allow them to drain and dry. The panels shall be used immediately or stored in a desiccator over a suitable desiccant until needed.

4.1.2 Used panels. Remove the old paint, etc., using a paint and varnish remover of the organic solvent type, or a suitable solvent mixture. Do not use a spatula, sandpaper, or other instrument or treatment that might scratch or etch the surface. Complete the cleaning as prescribed in 4.1.1.

METHOD 2051
January 24, 1986

PREPARATION OF CONCRETE PANELS

1. SCOPE

1.1 This method describes the procedure and materials required in making concrete and masonry panels for use in testing paints.

2. APPARATUS

2.1 Test panels.

2.1.1 The test panels shall be made by one of the following procedures.

2.1.2 Procedure A. Panels shall be prepared from a mix of three parts of Ottawa sand (20-30 mesh) and one part of white portland cement (ASTM C 150, type I), by weight. Mix thoroughly with sufficient water to obtain a trowelling consistency and cast into panels measuring 6 by 6 by 2 inches. The top surfaces of the panels shall be "trowel cut". Allow the panels to harden in the air for 24 hours, then cure in water for 14 days. After this time, the panels may be stored in an atmosphere free of chemical fumes until used.

2.1.3 Procedure B. Panels shall be prepared from a mix of 1 part of portland cement (ASTM C 150, type I) and 1 part of graded Ottawa silica sand, conforming to the requirements of 4 of method 2001 of Fed. Test Method Std. No. 158, by volume. Mix thoroughly with sufficient water to obtain trowelling consistency and cast into panels measuring 3 by 3 by 1/2-inch. The top surface of the panels shall be trowelled smooth, but not excessively; three trowellings are adequate. The panels shall be allowed to harden in air for at least 2 weeks. The panels may be prepared in advance and held ready for use; however, panels which have aged for more than 6 months shall not be used. The surface glaze shall be removed with No. 1 sandpaper before the panels are used.

2.1.4 Procedure C. Panels shall be prepared from a mix of three parts of concrete sand (SS-A-281) and one part of portland cement (ASTM C 150, type I) by weight. Mix thoroughly with sufficient water to obtain a trowelling consistency and cast into panels measuring 6 by 3 by 1/2-inch. The top surface of the panels shall be screened and lightly trowelled using a steel trowel. Three and one-half to 4 hours later, trowel to a smooth finish. Allow the panels to harden in air for 24 hours and then cure in water for 7 days. The panels shall be thoroughly dried in air before using. They may be prepared in advance and held ready to use. The surface glaze shall be removed with No. 1 sandpaper before the panels are used.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 See section 2.

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METHOD 2081
January 24, 1986

PREPARATION OF GYPSUM WALLBOARD PANELS

1. SCOPE

1.1 This method provides a procedure for preparing a gypsum wallboard test panel which encompasses many of the operational problems encountered with this structured material. This method eliminates as many variables as possible in the preparation of panels.

2. APPARATUS

2.1 Gypsum wallboard meeting SS-L-30.

2.2 Joint cement such as manufactured by U.S. Gypsum Co. or National Gypsum Co.

2.3 Nails. Any commercial nail recommended for sheet rock.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Fabricate a 24- by 24-inch test panel by butting the beveled edges of two 12- by 24-inch sections of gypsum wallboard (see 2.1) and backing the joint with a 2-1/4- by 3/4- by 24-inch strip of wood. Nail the wallboard to the wood strip with a commercial sheet rock nail. The nails shall be countersunk. Fill the nail holes and the joint with a joint cement (see 2.2), trowelling it smooth and feathering it into the surrounding areas. The panel shall be allowed to dry 72 hours before being used for testing. If any cracks develop, refill them with joint cement and allow an additional 24 hours to dry.

5. NOTES

5.1 None

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO 141C

METHOD 2112
January 24, 1986

APPLICATION BY ROLLER

1. SCOPE

1.1 This method describes the application of coating materials by means of a roller.

2. APPARATUS

2.1 Paint roller with 10 mm pile, and accessories, meeting H-R-550.

2.2 Gypsum wallboard panel, prepared according to method 2081.

3. REAGENTS

3.1 None

4. PROCEDURE

4.1 Thoroughly wet the roller with the material under test. Remove excess paint from the roller.

4.2 Apply from a Luer syringe to a gypsum board panel, prepared as described in method 2081 unless otherwise specified, a volume of material calculated to give the dried film thickness specified in the product specification. Weigh the syringe before and after the application to determine the weight of material applied.

4.3 Roll the material so that the roller passes completely over all the surface twice in the vertical and twice in the horizontal direction.

4.4 Weigh the roller to ensure that the weight is approximately the same at the end as at the beginning.

5. NOTES

5.1 None

6. PRECISION

6.1 No data

FED. TEST METHOD STD. NO. 141C

METHOD 2131.1
January 24, 1986

APPLICATION OF SPRAYED FILMS

1. SCOPE

1.1 This method covers procedures for both the manual and automatic application of finishing materials by using spraying apparatus.

2. MANUAL APPLICATION

2.1 Cold spray.

2.1.1 Reduction of material. When dilution is necessary, the material shall be reduced with the specified thinner to the required solids content or viscosity. Where no viscosity is specified, spray-outs performed with standard type guns shall be done at a viscosity of 17 to 25 seconds in a No. 4 Ford cup at 25° C (see ASTM D 1200) for pigmented materials, and 0.65 to 1.00 stokes at 25° C (see ASTM D 1545) for clear materials.

2.1.2 Application. The air pressure shall be kept constant and the air line provided with a moisture trap. The flow, gun distance from the test panel, and spray pattern shall be so adjusted that the deposited film is kept smooth, level, and wet. Application shall be made so as to produce a dry film thickness of (a) 0.0008 to 0.0011 or (b) 0.003 \pm 0.0003 inch depending on the nature of material applied or the number of coats and thickness specified in the product specification.

2.2 Hot spray.

2.2.1 Application. The packaged material shall be applied with an approved hot spray unit. When dilution is necessary, the material shall be reduced with the specified thinner to the required viscosity for hot spray application. The conditions of operation with respect to temperature of heating unit, at spray gun, and of atomization air; pressure of atomization air and feed tank; type and adjustment of air cap and fluid tip on spray gun; distance of gun from work; and rate of flow of material at spray nozzle shall be as specified.

3. AUTOMATIC APPLICATION

Any automatic spraying machine which combines reciprocating rectilinear motion with variable speed controls and means of adjusting distance from spray gun to test specimen may be used. The speed of the traversing mechanism and the distance from the spray gun to the test specimen shall be varied until satisfactory spraying properties and the specified dry film thickness are obtained. There shall be no change in operating conditions after correct settings have been determined. Application shall be to the number of coats or thickness specified in the product specification or as specified in 2.1.2.

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METHOD 2141.1
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APPLICATION OF BRUSHED FILMS

1. SCOPE

1.1 This method covers the procedure for application of brushed films for evaluation of coatings.

1.2 High quality, clean, flat brushes free of loose bristles and having a minimum bristle length of 2 inches shall be used. The brushes shall have good softness and flexibility and shall be carefully cleaned after each day's use. Only brushes which have been thoroughly broken in, rather than brand new brushes, shall be used in the preparation of test panels. In general, the size and type of the test panel will more or less govern the size of brush to be used. For example, a 1- to 1-1/2-inch brush is suitable for areas of less than 1 square foot, a 2- to 2-1/2-inch brush for areas of 1 to 4 square feet and a 4- to 5-inch brush for areas of 8 or more square feet.

2. APPLICATION

2.1 The material shall be applied by brush at a spreading rate normal for the material being tested by a prewetted brush. A Luer-type glass syringe shall be filled with the material under test and distributed over the panel quickly and uniformly spread at a predetermined or specified rate or wet film thickness. The brush shall be wetted with the coating tested by dipping about 2/3 of the brush bristles into the coating and worked out on a clean surface. Repeat if necessary until a well-wetted brush is obtained. Then weigh the wetted brush and the Luer syringe filled with the coating material. Reweigh the brush and syringe after application of coating. Spreading rate, if required, can be determined with this data. The brush application shall be by up and down strokes, then crosswise and finally with up and down strokes lightly. The above operation shall be done at temperatures between 70° and 90° F unless otherwise specified. Allow the brush painted panels to dry in a horizontal position.

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METHOD 2161
January 24, 1986

APPLICATION OF FILM WITH FILM APPLICATOR
(MAGNETIC CHUCK)

1. SCOPE

1.1 This method covers the apparatus for and the application of a paint film with film applicator.

2. APPARATUS

2.1 Film applicator. The applicator shall be made of nonmagnetic metal (18/8 stainless steel has been found satisfactory). The applicator shall spread a 2-inch wide or wider film and shall have such clearance that the thickness of the resulting dried film of the material under test will be as specified in the product specification. The Bird applicator or equal has been found satisfactory for this purpose.

2.2 Magnetic chuck. The magnetic chuck shall be of the permanent magnetic type equipped with a control handle and so designed that a magnetic field can be established at the working surface of the chuck at the will of the operator by mechanically raising or lowering the magnet. The area of the working surface on the chuck shall be greater than the area of the panel and the chuck shall have sufficient magnetic strength to hold the panel in contact with the chuck throughout the area of the panel.

2.3 Panel. The type of panels used shall be (a) smooth plate glass, (b) opaque white glass, (c) metals panels, and (d) as specified on the product specification. The size of the panels shall be as specified in the product specification.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Pour an excess of the coating being tested on the panel just in front of the film applicator. Pull the film applicator down the panel with a continuous and smooth motion. The film so cast should be smooth, homogeneous, and free of irregularities.

4.2 Use of magnetic chuck. To hold metal panels firm and flat during application of coating, using a magnetic chuck has been found desirable. The handle of the magnetic chuck shall be adjusted to the "off" position. A sheet of paper (newspaper is suitable) shall be laid on the chuck. The specified metal panel shall be placed on the paper and the handle adjusted to the "on" position. The metal panel shall be previously cleaned by procedure in 2011. An excess quantity

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of the sample shall be placed on one end of the panel and a film spread by means of the applicator. The handle shall be adjusted to the "off" position and the panel removed. The test panel shall be supported in a horizontal position until the coating has dried.

METHOD 2162
January 24, 1986

APPLICATION OF FILM WITH FILM APPLICATOR
USING SUCTION PANEL HOLDER

1. SCOPE

1.1 This method covers procedures for applying the film by operating the applicator either by hand or by a motor-driven mechanism.

2. HAND APPLICATION

2.1 Apparatus

2.1.1 Suction panel holder. The panel holder consists essentially of a suction box provided with a straight edge to facilitate the use of the applicator. The suction box may be one of the following constructions:

2.1.1.1 A plane plate glass panel with small holes drilled at appropriate intervals, cemented or fitted on an air-tight wooden or metal box. The suction box should be of proper size to accommodate the test panel. Figure 1 covers a design suitable for use with a 3- by 5-inch panel.

2.1.1.2 A hollow aluminum or steel one piece casting with a smoothly ground, plane surface into which are drilled small holes at appropriate intervals.^{1/}

2.1.2 Film applicator. The applicator shall be made of metal, preferably noncorrosive, and shall be accurately constructed and smoothly finished. It shall spread a film of uniform width and shall have such clearance that the thickness of the resulting wet film of coating applied will be as specified in the product specification.

2.1.3 Test panels. The panels shall be smooth and flat and may be cold rolled steel, tinplate, paper (hiding power chart), or glass as specified in the product specification.

2.2 Procedure. Place the properly prepared test panel on the panel holder with a piece of thin paper under the edge of the panel nearest the operator and extending out beyond the holder (The paper serves to catch the excess material from the drawdown.) Connect the panel holder to the laboratory suction line or a water aspirator. Then place an excess quantity of the finishing material at one end of the panel in front of the film applicator. Spread the material the length of the panel by means of the applicator, one side of which is guided by the straight-edge. Turn off the suction, remove and discard the paper, and dry the test film in a horizontal position, as specified in the product specification.

^{1/} The Bird Vacuum Plate sold by Bird and Sons, Inc., East Walpole, Mass., meets these requirements.

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3. MECHANICAL APPLICATION

3.1 Apparatus. The fundamental parts of the automatic film applicator^{2/} are the base plate, the doctor blade, and the drive mechanism similar to those illustrated in figure 2.

3.1.1 Base plate. The base plate illustrated in figure 1 is a 9- by 12-inch hollow aluminum casting containing seventy-five, 3/32-inch holes spaced 1 inch on centers across the width and 1-3/4 inches on centers along the length of the plate.^{3/}

3.1.2 Doctor blade. The doctor blade may be any one of the usual types.

3.1.3 Test panels. Same as in 2.1.3.

3.1.4 Drive mechanism. The drive motor may be any motor of adequate power. Shafts with gears to accommodate the driving chain for the blade shall be placed at each end of the base plate. One of the shafts shall be connected to the motor through a gear speed reducer to give a rate of travel of the blade of 1 \pm 0.02 inch per second. A holder for the blade shall be held in position by pins which fit into the chains. The holder shall be designed to accept weights for loading the doctor blade. Weights of approximately 1,000 g have been used. A mechanism shall be provided to stop the blade movement automatically at the end of the drawdown.

3.2 Procedure. Place the properly prepared test panel on the base plate. Apply suction to the base plate from the laboratory suction line or a water aspirator, when necessary to hold the test panel to the base plate. Place the doctor blade in position on the test panel and apply a suitable amount of the finishing material in front of the blade. Start the mechanism, which automatically draws the doctor blade the length of the test panel. Turn off the suction and dry the test film in a horizontal position as specified in the product specification.

3.3 Precision. A precision of \pm 0.10 mil (0.0001 inch) is obtainable in the thickness of the applied films.

^{2/} A machine of this description is sold by the Gardner Laboratory Inc., 2431 Linden Lane, Silver Spring, MD.

^{3/} Another type of base plate with hollow channels near the edges of the upper surface or holes along the edges only for application of the vacuum may be used to avoid possible dimpling of the substrate to which the test coating is applied.

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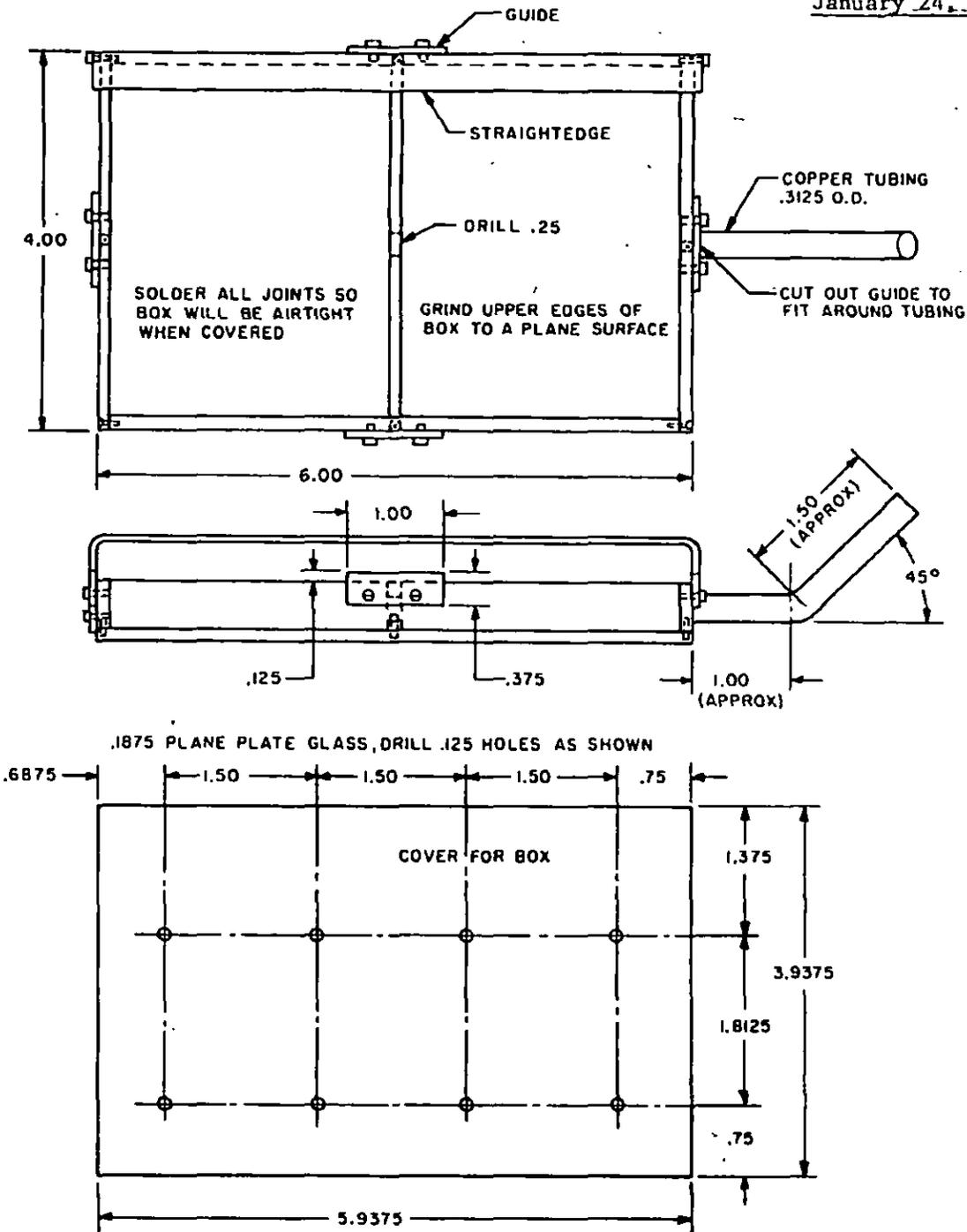


FIGURE 1. Suction panel holder.

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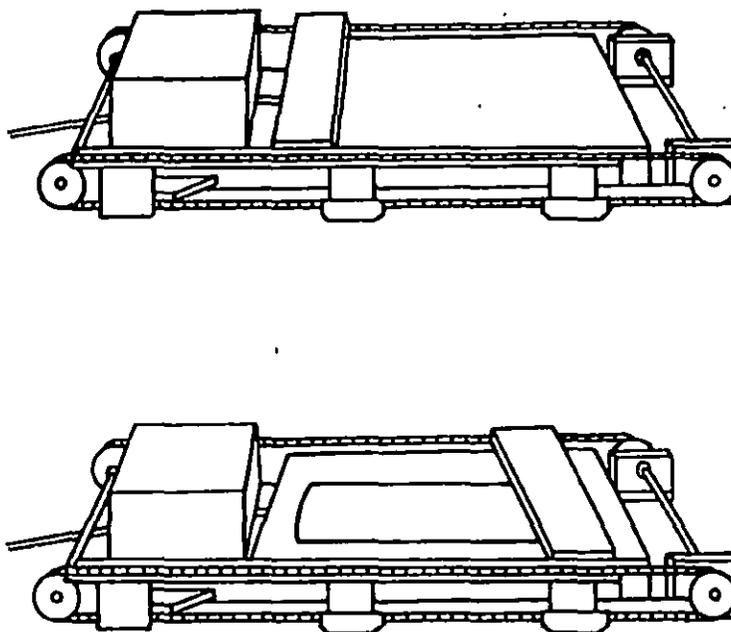


FIGURE 2. Motor-driven blade film applicator.

X-4465

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Section 3000
PACKAGE STABILITY TESTS

METHOD 3011.2
January 24, 1986

CONDITION IN CONTAINER

1. SCOPE

1.1 This method covers procedures for determining changes in properties of paints and related materials after packaging.

2. APPARATUS

2.1 No special apparatus required.

3. REAGENTS

3.1 None required.

4. PROCEDURES

4.1 General. Before agitating the contents of the container in which the material was originally packaged or transmitted for test, open the container by suitable means and determine the condition of the material by one of the following applicable procedures against specified requirements.

4.1.1 Ready-mixed pigmented materials (paints, enamels, etc.). Note whether skinning has occurred and remove any continuous skin with a spatula by first carefully cutting the skin free from the container. Lower a stiff spatula or paddle into the container and observe if the material is abnormally thick or livered and to what extent settling or caking exists. Soft caking can usually be broken up just by stirring. When firm or hard caking is encountered, the supernatant liquid shall be poured into a clean container and then added back in small amounts with continuous stirring until the pigment has been reincorporated to form a homogeneous material suitable for use or until it is determined that the pigment cannot be reincorporated by hand stirring. After the material has been thoroughly stirred, examine for presence of gell bodies or undispersed conglomerates as it flows from the spatula or paddle. Immediately after stirring examine the surface of the material for floating pigments. Flow some of the material on a tin or glass panel, allow to drain, in a vertical position and examine for loss of drying ability, color drift, seeding, and coarse particle. Check for change in viscosity per applicable method such as ASTM D 562.

4.1.2 Ready-for-use clear materials (varnishes, lacquers, oils, etc.). Examine for skinning, thickening, livering gelatinous masses, ingredient separation, and other specially defined characteristics that may be objectionable for the specified material such as in 4.1.5. Examination shall be made with the use of a spatula or paddle or by transferring the material to a clear glass container.

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4.1.3 Pastes-in-oil and concentrates. Lower the spatula or paddle into the material and pass it over the bottom of the container to determine the extent of caking. Add slowly with constant stirring the specified thinning material and determine whether the paste or concentrate breaks up readily to a smooth, homogeneous mixture of satisfactory brushing consistence.

4.1.4 Dry materials (pigments, toners, etc.). Transfer a representative amount of the material from the container to a mixing sheet. Note whether the material is homogeneous and has the physical characteristics normal to it. Abnormal lumps or conglomerates shall be friable or capable of being broken up or separated to approximately individual particle size, normal for the material, by pressure between the thumb and fingers or by light tapping with a blunt instrument. Examine for discoloration, dirt and other signs of contamination or adulteration.

4.1.5 Undesirable qualities.

4.1.5.1 The following characteristics in the material are considered undesirable and objectionable under all conditions.

- a. Curdling.
- b. Agglomerates.
- c. Gelling.
- d. Seeding.
- e. Putrefaction.
- f. Gassing.
- g. Livering.

4.1.5.2 The following characteristics are considered undesirable and objectionable if excessive and not capable of being reworked to an acceptable package condition per applicable method.

- a. Caking (ASTM D 869).
- b. Settling (ASTM D 869).
- c. Separation.

4.1.5.3 Marked changes in the following characteristics are not desirable but small changes within specification limits may be permitted.

- a. Coarse particles
- b. Viscosity. See method 4287.
- c. Loss of drying ability (method 4061).
- d. Color drift (method 6123).

5. NOTES

5.1 None

6. PRECISION

6.1 No data. —

FED. TEST METHOD STD. NO. 141C

METHOD 3Q19.1
January 24, 1986

STORAGE STABILITY AT THERMAL EXTREMES

1. SCOPE

1.1 This method provides a procedure for determining the stability of materials which would be subjected to unusual storage conditions.

1.2 This method may be used as an accelerated storage test (see 5.1).

2. APPARATUS

2.1 A commercial friction top, 1-pint tinplate can, lined as usual for the product under test.

2.2 Stormer viscosimeter.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of sample.

4.1.1 Fill the can with the product under test. Seal the can. The container shall be exposed to hot or cold storage as specified in the product specification.

4.2 Hot storage.

4.2.1 If hot storage is to be used, the material under the test shall be treated as follows, unless otherwise specified. The material under test shall be held at $120^{\circ} \pm 1^{\circ} F$ for 168 hours. At the beginning of the test the material shall be brought to temperature in approximately 1 hour. At the end of the test the material shall be brought to room temperature ($77^{\circ} \pm 1^{\circ} F$) in approximately 1 hour.

4.3 Cold storage.

4.3.1 If the cold storage is to be used, the material under test shall be treated as follows, unless otherwise specified. The material under test shall be held at $10^{\circ} \pm 1^{\circ} F$ for 168 hours. At the beginning of the test the material shall be brought to temperature in approximately 1 hour. At the end of the test the material shall be brought to room temperature ($77^{\circ} \pm 1^{\circ} F$) in approximately 1 hour.

4.4 Evaluation.

4.4.1 Any change such as the following shall be noted.

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4.4.1.1 Viscosity. The viscosity shall be run before and after with a Stormer viscosimeter as per ASTM D 562. Any change shall be noted.

4.4.1.2 Any putrefaction shall be noted.

4.4.1.3 A drawdown or some similar test shall be made for visual determination of agglomeration.

5. NOTES

5.1 Certain types of storage failure will not be apparent from this test, i.e., certain bacteria causing putrefaction are inhibited at 120° F.

6. PRECISION

6.1 No data.

METHOD 3021.1
January 24, 1986

SKINNING (PARTIALLY FILLED CONTAINER)

1. SCOPE

1.1 This method provides a procedure for determining the skinning characteristics of a material.

2. APPARATUS

2.1 Cabinet. A storage cabinet, box, or drawer of sufficient interior dimensions to allow upright storage of the sample containers. The storage area will be kept dark and at 22.2° to 26.7° C (72° to 80° F).

2.2 Container. An 8-ounce wide-mouth glass jar, approximately 4-1/2 inches in height and 2 inches in diameter, shall be used as the container.

2.3 Measuring device. A suitable volumetric measuring device to measure 6 ounces ± 0.5 ounces.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Unless otherwise specified, a 6-ounce sample of the coating material shall be measured into the glass jar. The cover shall be secured on tightly and the jar inverted momentarily. The jar shall then be placed in an upright position in the dark (placing it under a box or in a drawer is satisfactory). The test shall be made at 22.2° to 26.7° C (72° to 80° F). The sample shall not be agitated or disturbed until inspected. The coating material shall be examined for skinning at the specified time interval.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 3022.1
January 24, 1986

STORAGE STABILITY (FILLED CONTAINER)

1. SCOPE

1.1 It is the objective of this method to determine the storage stability (filled container) in terms of the undesirable qualities that may be produced during a specified time and condition of storage.

2. APPARATUS

2.1 An 8-ounce wide-mouth glass jar, approximately 4-1/2 inches in height and 2 inches in diameter, shall be used as the container.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 The jar shall be filled with the material under test. The cover shall be secured on tightly and the jar inverted momentarily. The jar shall then be placed in an upright position in the dark (placing it under a box or in a drawer is satisfactory). The test shall be made at 22.2° to 26.7° C (72° to 80° F). The sample shall not be agitated or disturbed until inspected.

4.2 Recommended storage period (see 5.1) as follows:

1. Lacquers and dopes - 6 months.
2. Enamels and varnishes - 6 months.
3. Oil paints - 12 months.
4. Other lengths of time peculiar to a product, its use or unusual storage conditions.

4.3 Undesirable qualities. The following characteristics in the material are considered undesirable and objectionable.

1. Caking (ASTM D 869).
2. Curdling.
3. Agglomerates.
4. Coarse particles (method 4092).
5. Gelling
6. Viscosity (ASTM D 562).
7. Settling (ASTM D 869).
8. Seeds.
9. Putrefaction.
10. Skinning (method 3021).
11. Gas.

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12. Livering.
13. Separation.
14. Loss of drying ability upon aging (method 4061).
15. Color drift upon aging (method 6123).

4.4 Reincorporation of pigment in the liquid.

4.4.1 After inspection without stirring for 1, 5, 7, 9, 10, 11, 12, and 13 of 4.3, reseal the container and agitate the jar for 5 minutes on a paint shaker. Open can and examine for compliance with 2, 3, 4, 6, 8, 14, and 15 of 4.3. If pigment is not fully redispersed, agitate until full dispersion is reached, if possible, and reexamine.

5. NOTES

5.1 This is the recommended storage period for the test and has no relationship to the actual storage life intended.

6. PRECISION

6.1 No data.

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Section 4000

PHYSICAL TESTS OF COATING MATERIALS
AND COATING INGREDIENTS

METHOD 4021.1
January 24, 1986

PIGMENT CONTENT (ORDINARY CENTRIFUGE)^{1/}

1. SCOPE

1.1 This method provides a procedure for determining the pigment content of all materials, except cellulosic lacquers and vinyl paints which contain toluidine, chrome green, and iron blue.

2. APPARATUS

2.1 Weighing device. An analytical or torsion balance sensitive to at least 1 division on indicator per milligram (mg) and appropriately accurate weights.

2.2 Centrifuge. A centrifuge with a multi-unit head, a speed of around 2,000 r.p.m. and an individual tube capacity of 60 ml minimum.

2.3 Oven. A well ventilated gravity convection-type oven thermostatically controlled to the specified temperature $\pm 2^{\circ}$ C.

2.4 Sieve. A sieve conforming to the requirements for a No. 80 sieve, RR-S-366.

3. REAGENTS

3.1 Extraction mixture A.

- 10 volumes ethyl ether - A.C.S. Reagent grade.
- 6 volumes benzene - VV-B-231.
- 4 Volumes methyl alcohol - O-M-232.
- 1 volume acetone - O-A-51.

3.2 Extraction mixture B.

- 3 volumes toluene - TT-T-548.
- 4 volumes absolute ethyl alcohol.
- 3 volumes acetone - O-A-51.

3.3 Extraction mixture C.

- 1 volume toluene - TT-T-548.
- 1 volume acetone - O-A-51.

3.4 Extraction mixture D.

- 1 volume ethyl ether A.C.S. reagent grade.
- 3 volumes petroleum ether - O-E-751.

3.5 Petroleum ether. O-E-751.

3.6 Alcohol. Absolute ethyl alcohol.

^{1/} This method yields slightly lower results than the supercentrifuge method.

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3.7 Acetone. Acetone conforming to the requirements of O-A-51.

3.8 Mineral spirits. Mineral spirits conforming to the requirements of grade I, TT-T-291.

4. PROCEDURE

4.1 Weigh accurately, about 15 grams (g) of the sample into a weighed centrifuge tube. Add 20 to 30 milliliters (ml) of the extraction mixture specified in the product specification or indicated in 5.1; mix thoroughly with a glass rod, wash the rod with more of the mixture, and add enough of the mixture to make a total of 60 ml in the tube. Place the tube in the container of a centrifuge, surround with water, and counterbalance the container of the opposite arm with a similar tube or a tube with water. Centrifuge until well settled. Decant the clear supernatant liquid. Repeat the extraction twice with 40 ml of the extraction mixture. After drawing off the liquid, hold the tube in a nearly horizontal position, tap the side of the tube to dislodge the pigment cake from the bottom of the glass tube, set the tube in a beaker of water at room temperature and gradually raise the temperature, or on top of a warm oven for 10 minutes, then in an oven at $105^{\circ} \pm 2^{\circ} \text{ C}$ ($221^{\circ} \pm 3.6^{\circ} \text{ F}$) for 2 hours. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through a No. 80 sieve to remove any "skins", and preserve in a stoppered tube for pigment analysis.

5. NOTES

5.1 The following reagents and procedures are suggested for the guidance of the analyst in extracting pigments from pastes, colors-in-oil, paints, enamels, and lacquers.

5.1.1 Group 1 - Pigments.

All whites.
Ultramarine blue.
Metallic brown.
Chromium oxide green.
Chrome yellow, chrome orange, and molybdate orange.
Bright red, Indian red, and mineral red iron oxides.
Venetian red.
Yellow ochre.
Umbers and siennas.
Yellow iron oxide.
Carbon black.

5.1.1.1 This group of pigments can usually be extracted with mixture A. In cases where the pigment does not separate satisfactorily from raw and boiled linseed oil vehicles, petroleum ether may be used.

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5.1.2 Group 2 - Pigments.

Lampblack.
Carbon black.
Toluidine.

5.1.2.1 Petroleum ether may be used to extract these pigments from linseed oil vehicles.

5.1.2.2 Mixture D may be used for toluidine enamels. In some cases it may be necessary to increase the ratio of ethyl ether to petroleum ether.

5.1.3 Group 3 - Pigments.

Chrome green.
Iron blue.

5.1.3.1 These pigments may be extracted from linseed oil vehicles as follows: Place 10 to 15 g of the sample in a weighed centrifuge tube. Add 5 ml of mineral spirits conforming to TT-T-291, grade I, and mix thoroughly with a glass rod. Add 45 ml of hot, absolute alcohol and mix thoroughly by stirring. Keep the tube and contents in a hot water bath while stirring. Remove the stirring rod and wash any adhering pigment back into the tube with hot alcohol. Centrifuge, while hot, for approximately 2 minutes. Pour off the clear solvent and oil layer and rinse the tube with hot alcohol without disturbing the pigment cake. Repeat the extraction three times. Dry the tube and contents at $105^{\circ} \pm 2^{\circ}$ C ($221^{\circ} \pm 3.6^{\circ}$ F) to constant weight.

5.1.3.2 Extraction mixture B may be used in extracting these pigments from varnish and resin vehicles. If the blue portion of the pigment does not separate upon centrifuging, increase the proportion of alcohol in the mixture until there is just sufficient to cause separation. In cases where the vehicle does not dissolve properly, decrease the proportion of alcohol until solution is just obtained.

5.2 Cellulosic lacquers and vinyl paints.

5.2.1 Extraction mixture C may be used for lacquers containing groups 1 and 2 pigments with the exception of toluidine. If satisfactory separation is not obtained, the mixture may be modified with other appropriate solvents as required.

5.2.2 No method is available for the extraction of toluidine or the group 3 pigments. Information as to the presence of group 3 pigments may be obtained by cautiously ashing a portion of the nonvolatile matter and examining the ash for iron and chromium.

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5.3 Water-thinned paints.

5.3.1 Extract twice with distilled water, then twice with acetone and then with mixture C until vehicle is removed. This method cannot be used with some types of water-thinned paints.

6. PRECISION

6.1 No data.

METHOD 4031
January 24, 1986

NONVOLATILE VEHICLE ISOLATION (ORDINARY CENTRIFUGE)

1. SCOPE

1.1 This method is to be used in conjunction with method 4021 to obtain the nonvolatile vehicle.

2. APPARATUS

2.1 Steam bath.

3. REAGENTS

3.1 As in method 4021.

4. PROCEDURE

4.1 Proceed as in method 4021, but retain all decanted liquid portions, combine and evaporate on a steam bath until all volatile matter has been removed. The isolated vehicle is then used for other tests.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 4042.1
January 24, 1986

VOLATILE AND NONVOLATILE CONTENT (VACUUM OVEN)

1. SCOPE

1.1 This method has application where a solvent is difficultly driven off at 105° C (221° F) in an ordinary convection type oven. It also has application where the evaporation temperature has to be maintained at a low level because of thermal decomposition in the product under test.

2. APPARATUS

2.1 Vacuum oven. Capable of maintaining a continuous vacuum of 29 \pm 0.5 inches of Hg.

2.2 Aluminum weighing dish. A flat-bottomed pan, 60 mm in diameter made of aluminum foil.

3. REAGENTS

3.1 ACS Toluene.

4. PROCEDURE

4.1 Place a portion of the thoroughly mixed sample in a dropping bottle and weigh to the nearest one-tenth mg. Weigh one of the 60 mm aluminum dishes with fourth-decimal accuracy. Transfer a small sample that does not exceed 0.3 g to the dish, determine its exact weight by loss in weight of the bottle. Dissolve the sample in 2 ml of A.C.S. reagent grade toluene (see 5.1) and dry in a vacuum oven. Unless otherwise specified, the test specimen shall be heated for 2 hours at 105° C \pm 2° C (221° F \pm 3.6° F) and under a continuous vacuum of 29 \pm 0.5 inches. Remove the dish from the oven and upon cooling reweigh it to the nearest one-tenth mg. From the weight of the residue in the dish and the weight of the sample taken, calculate the percent nonvolatile or volatile as required.

5. NOTES

5.1 It is important that the sample dissolve in the 2 ml of solvent added. If a resin is encountered that is insoluble in toluene, a suitable mixture such as equal volumes of toluene with ethyl alcohol or acetone will be satisfactory. Thin water emulsion paints with 2 ml of water.

6. PRECISION

6.1 No data.

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METHOD 4044
January 24, 1986

NONVOLATILE CONTENT OF DOPES AND LACQUERS

1. SCOPE

1.1 This method gives a procedure for the determination of the nonvolatile content of dopes and lacquers.

2. APPARATUS

2.1 Weighing device. An analytical balance sensitive to at least 1 division on the indicator per 0.1 mg and appropriately accurate weights.

2.2 Desiccator. An ordinary laboratory desiccator equipped with a sample shelf and utilizing a suitable desiccant.

2.3 Dish. A flat bottom disposable aluminum dish approximately 2 inches in diameter.

2.4 Oven. A well ventilated gravity convection-type oven thermostatically controlled to the specified temperature $\pm 2^{\circ}$ C.

3. REAGENTS

3.1 Acetone. Acetone conforming to the requirements of O-A-51.

4. PROCEDURE

4.1 Place a portion of the thoroughly mixed sample in a stoppered flask or weighing bottle and weigh to the nearest 0.1 mg. Weigh to the nearest 0.1 mg a small disposable aluminum dish, approximately 2 inches in diameter. Transfer a quantity of the sample that will contain approximately 0.5 g of nonvolatile matter from the weighing container to the dish. Reweigh the container and calculate the weight of the sample taken. Add 2 ml of acetone, conforming to O-A-51, to the sample in the dish and mix by gentle swirling. Allow the dish and its contents to stand at 21° C to 32° C (70° F to 90° F) for between 12 and 24 hours; then heat the dish and its contents in an oven at 60° C $\pm 2^{\circ}$ C (140° F $\pm 3.6^{\circ}$ F) for 2 hours. Remove the dish from the oven, cool in a desiccator and weigh. Replace in the oven and reheat at the same temperature for one hour, cool in a desiccator and reweigh. If necessary, repeat to a constant weight of within 0.001 g. From the weight of the residue in the dish and the weight of the sample taken, calculate the percentage of nonvolatile.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 4051.1
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VEHICLE SOLIDS (ORDINARY CENTRIFUGE)

1. SCOPE

1.1 This method gives a procedure whereby the vehicle solids may be computed.

2. APPARATUS

2.1 The apparatus required in this method are a combination of that required in methods 4021 and ASTM D 2369.

3. REAGENTS

3.1 The reagents required in this method are a combination of those required in methods 4021 and ASTM D 2369.

4. PROCEDURE

4.1 Determine the percentage of pigment in the sample by method 4021 and its percentage of nonvolatile matter by ASTM D 2369. From this data calculate the percentage of solids in the vehicle using the following equation:

$$\text{Percent solids in vehicle} = \frac{A - B}{100 - B} \times 100$$

where:

A = percentage of nonvolatile in the sample.

B = percentage of pigment in the sample.

5. NOTES

5.1 This method is subject to the same limitations as method 4021.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 4053.1
January 24, 1986

NONVOLATILE VEHICLE CONTENT

1. SCOPE

1.1 This method gives a procedure whereby the nonvolatile vehicle content may be calculated using the sum of the volatile matter and pigment solids content.

2. APPARATUS

2.1 The appropriate apparatus from the specific methods chosen shall be used.

3. REAGENTS

3.1 The appropriate reagents from the specific methods chosen shall be used.

4. PROCEDURE

4.1 Add the percentage of volatile matter in the sample as determined by ASTM D 2369 or method 4042 to its pigment percentage as determined by method 4021 or ASTM D 2698. Subtract this sum from 100 to obtain the percentage of nonvolatile vehicle in the sample.

5. NOTES

5.1 This method is limited only by the limitations placed on the methods which are used.

6. PRECISION

6.1 No data.

METHOD 4061.2
January 24, 1986

DRYING TIME

1. SCOPE

1.1 This method determines the various stages and rates of film formation in the drying or curing of organic coatings normally used under conditions of ambient room temperature.

2. FILM THICKNESS AND MANNER OF DRYING

2.1 Thickness of dried film. The film thickness and application method to be used shall be stated in the product specification. If the specification does not give this information, table I shall be used.

TABLE I. Dry film thickness.

Test material	Dry film thickness, mm	Notes
Clear coatings	.0304 \pm .0051	See 4.1. Add driers a minimum of 24 hours before test.
Drying oils	.0304 \pm .0051	
Varnishes	.0254 \pm .0025	See 4.1. See 4.1.
Lacquers	.0254 \pm .0025	
Resin solutions	.0254 \pm .0025	
Enamels	.0304 \pm .0051	
Oil-based paints	.0457 \pm .0051	
Water-thinned paints	.0304 \pm .0051	
Rubber-based paints	.0304 \pm .0051	
New synthetics	.0304 \pm .0051	

2.2 Air-drying.

2.2.1 Routine testing. Air-drying shall be done in a well-ventilated room or chamber free from drafts and dust, and in diffused light (not in direct sunlight). An illumination of approximately 25 foot-candles is preferred for drying oil films. The temperature of the air shall be between 21° C and 28° C and the relative humidity shall be 50 \pm 5 percent. If the time of drying is not within the specified limits of the product specification, repeat the test under the atmospheric conditions specified in 2.2.2.

2.2.2 Referee testing. In case of dispute or disagreement between laboratories, air-drying shall be done in an atmosphere of 50 \pm 4 percent relative humidity and at a temperature of 23° C \pm 1° C.

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2.3 Baking. Baking shall be done in a forced draft laboratory baking oven capable of maintaining the specified temperature within a range of $\pm 2^{\circ}$ C. The product specification shall specify the specific temperature for the product.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Preparation of test specimens.

4.1.1 All test specimens shall be prepared in duplicate and tested by an operator properly skilled in the methods to be used.

4.1.2 The materials to be tested shall be applied on clean glass panels or on another specific substrate of suitable dimension. The coated panels shall be placed in a horizontal position and shall be shielded from excessive dust accumulation while drying. Ground glass plates are more suitable for coatings that tend to crawl, such as low viscosity drying oils. Suitable plates can be prepared by roughening the surface of polished glass by grinding a paste of silicone carbide (Grit 1-F) and water between two glass plates.

4.1.3 The test films preferably shall be cast with a doctor blade having a clearance sufficient to give the recommended dry film thickness in table I. The allowable dry film thickness variation obtained with the blade shall not lie outside the limit of 0.002 mm over the area of the film to be tested. When a suitable doctor blade is not available, or it has been agreed upon to apply the film in some other manner, the various conventional and automatic methods of spray, dip, thicknesses conform to the above requirements.

4.1.4 The dry film thickness of the test films shall be measured with the proper film thickness gauge. When plates of small area are used, measurement of dry film thickness can be made by weight of plates before and after coating and calculation from plate area and coating solids.

4.2 Performance of test.

4.2.1 The film shall be tested at points not less than 10 mm from the edges. Tests shall be made at regular drying intervals, such as 5 minutes, 15 minutes, or 1 hour, depending on the speed of drying. The different stages of drying, defined herein, shall be determined as follows:

4.2.2 Set-to-touch. An intermediate stage in the drying of coatings, paints, or varnishes which is reached when gentle pressure of the finger shows a tacky condition of the film, but none of the material adheres to the finger.

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4.2.3 Dust-free. This stage of drying shall be determined by one of the following methods, as specified.

4.2.3.1 Cotton fiber method. Separate a number of individual fibers from a mass of absorbent cotton. At regular drying intervals, drop several of the cotton fibers from a height of 25 mm onto the film. The dust-free time is the earliest time that the cotton fibers may be removed by blowing lightly across the surface of the film.

4.2.3.2 Calcium carbonate method. Lay off along its long edge equal portions of the test film and record the drying time of each portion before dusting. Sprinkle a small amount of finely powdered calcium carbonate on the film. Repeat the operation on different portions of the film at regular intervals. After the film has dried tack-free (see 4.2.4), remove the calcium carbonate dust by blowing with a gentle stream of air and wiping with a soft cloth or camel's-hair brush. The coating shall be considered dust free at the time interval that the calcium carbonate can be removed completely.

4.2.4 Tack-free.

4.2.4.1 Mechanical method (see 5.1).

4.2.4.1.1 Apparatus. The tack tester to be used in this method comprises essentially a base or surface-contacting portion 25 mm square and a counterbalancing portion 25 by 50 mm in area. Both portions are made up from a continuous metal strip 0.406 to 0.457 mm in thickness. To prepare the apparatus for use, the base is fitted with several thicknesses of masking tape and paper strips to provide a means of attaching aluminum foil, and the angle of the counterbalancing strip is so adjusted that a weight of 5 g placed in the geometric center of the base is just sufficient to overcome the unbalanced force.

4.2.4.1.2 Procedure. At regular drying intervals make preliminary tests of the state of tackiness of the film by placing a strip of aluminum foil (152 by 25 by 0.012 mm) on the film and exerting firm finger pressure against the foil. Pressure of the finger-tip against the foil shall not be greater than that required to obtain a contact area of 3 to 5 mm in diameter. When the foil can be removed without noticeable pull, proceed with the tack tester. Place the base of the tester on a portion of the film not previously used and place a 300-g weight on the base. Remove the weight at the end of 5 seconds. The film shall be considered tack-free when the tack tester tips over immediately upon removal of the weight.

4.2.5 Free from after-tack. Some varnishes retain a tacky condition after the dry-through period (see 4.2.6) has been reached. This condition should not be confused with the tack-free drying stage (see 4.2.4) which occurs previously to the dry-through stage. This state of drying shall be determined by the following method.

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4.2.5.1 Apparatus.

4.2.5.1.1 Test paper. Kraft paper test sheets (90# basis weight) that when conditioned in accordance with the standard method of the Technical Association and the Pulp and Paper Industry, conforms to the following requirements (see 5.2).

Basis weight (24 x 36/500), pounds		90	+4.5
Thickness, inch		0.0070	+0.0005
Smoothness, Gurley, R.C.	T.S.	40	+15
	W.S.	60	+20
Smoothness, Sheffield	T.S.	210	+50
	W.S.	130	+30
Water resistance, average, seconds		120	+30
Bursting strength, points		75	- 105

4.2.5.1.2 Bearing weight. A steel cylinder 50 mm in diameter, weighing 2.8 kg.

4.2.5.2 Procedure. Lay a 51- by 70-mm piece of the test paper on the film, and place the cylinder on the paper. At the end of 5 seconds remove the cylinder and invert the test panel. The film shall be considered free from after-tack when the paper drops off the test film within 10 seconds.

4.2.6 Dry.

4.2.6.1 Paints and enamels. Test the film with a finger using moderate pressure. The film shall be considered dry when the soft tacky condition no longer exists and the film feels firm.

4.2.6.2 Drying oils. Lightly rub the finger across the surface of the test films. The film shall be considered dry when it no longer adheres to the finger and does not rub up appreciably.

4.2.7 Dry-through for varnish, lacquers and enamels. Place the test panel in a horizontal position at a height such that, when the thumb is placed horizontally on the film, the arm of the operator is in a vertical straight line from wrist to shoulder. Bear downward on the film with the thumb, exerting the maximum pressure of the arm, at the same time turning the thumb through an angle of 90 degrees in the plane of the film. The film shall be considered dry-through when no loosening detachment, wrinkling, or other distortion of the film occurs.

4.2.8 Dry-hard time. With the end of the thumb resting on the test film and the forefinger supporting the test panel, exert a maximum downward pressure (without twisting) of the thumb on the film. Lightly polish the contacted area with a soft cloth. The film shall be considered dry-hard when any mark left by the thumb is completely removed by the polishing operation.

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4.2.9 Dry-to-recoat. A film shall be considered dry for recoating when a second coat or specified topcoat can be applied without the development of any film irregularities, such as lifting or loss of adhesion of the first coat, and the drying time of the second coat does not exceed the maximum specified (if any) for the first coat.

5. NOTES

5.1 The standard tack tester is fully described in U.S. Patent 2,406,989, dated September 3, 1946.

5.2 Paper meeting these requirements may be obtained from the Gunned Industries Association, 415 Lexington Avenue, New York, NY 10017.

6. PRECISION

6.1 Because of the subjective nature of the drying time test, the agreement to be expected between laboratories depends upon their understanding of the terms used, and cannot be established with certainty. Within any laboratory, the agreement depends upon the material being tested, some coatings being much sharper in their end point than others, but duplicate determinations should agree within 10 percent of the time of drying.

6.2 Critical factors in the measurement of drying time of coatings are room temperature, relative humidity, and rate of air flow over the sample.

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METHOD 4091.1
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COARSE PARTICLES AND SKINS IN OIL-BASE PAINTS AND PASTES

1. SCOPE

1.1 This method provides a means to determine the amount of skins and agglomerated material in oil-base paints and pastes.

2. APPARATUS

2.1 Sieve. A No. 325 (44 micron) sieve having a diameter of 3 inches and conforming to RR-S-366.

2.2 Brush. A 1-inch camel-hair brush.

2.3 Weighing device. An analytical or torsion balance sensitive to at least 1 division on indicator per mg, and appropriately accurate weights.

2.4 Desiccator. An ordinary laboratory desiccator equipped with a sample shelf and utilizing a suitable desiccant.

2.5 Dish. A porcelain dish, approximately 8 inches in diameter.

2.6 Oven. A well ventilated gravity convection-type oven thermostatically controlled to the specified temperature $\pm 2^{\circ}$ C.

3. REAGENTS

3.1 Kerosene. Kerosene conforming to ASTM D 3699.

3.2 Extraction mixture.

10 volumes ethyl ether - A.C.S. reagent grade

6 volumes benzene - VV-B-231.

4 volumes methyl alcohol - O-M-232.

1 volume acetone - O-A-51.

4. PROCEDURE

4.1 Thoroughly clean and dry the sieve in an oven at 105° C $\pm 2^{\circ}$ C (221° F $\pm 3.6^{\circ}$ F). Cool the sieve in a desiccator and weigh it to the nearest mg. Weigh to the nearest mg an amount of the sample containing approximately 10 g of pigment (25 g of pigment in case of white leads and red lead) into a 250-ml beaker. Add 100 ml of kerosene if sample is a paste and 50 ml if it is a paint. Thoroughly mix the sample and kerosene, breaking up, but not grinding, all lumps with a flattened end of a stirring rod. Wet the sieve on both sides with kerosene. Slowly pour the contents of the beaker into the sieve. When the

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bottom of the sieve is covered start working the liquid through the sieve by passing the brush back and forth through the liquid. No more pressure shall be applied to the brush than that necessary to keep the tips of the bristles in contact with the bottom of the sieve. Transfer all pigment particles clinging to the stirring rod and walls of the beaker to the sieve using the brush and a stream of kerosene from a wash bottle. After the liquid and rinsings have passed through the sieve, place the sieve in an 8-inch porcelain dish containing 250 ml of kerosene, so that the sieve will be immersed to a depth of about 1/2 inch. Move the pigment remaining on the sieve around and over the bottom of the sieve by passing the brush across it at the rate of two strokes per second during two periods of 10 seconds each. After each 10-second period, raise the sieve and let the liquid run through. Clean the dish, add a new portion of kerosene and repeat the operation until the kerosene passing over the residue and through the sieve is clear and free of solid particles. When the washing appears to be complete, transfer about 200 ml of the wash kerosene to a 400-ml beaker. Stir the kerosene vigorously and place the beaker on a black surface in case of white pigments and on a white surface in the case of colored pigments. The washing shall be considered complete only when no particles collect about the middle of the bottom of the beaker. Wash the pigment particles adhering to the brush into the sieve using a stream of extraction mixture from a wash bottle. Finally wash the kerosene from the sieve with extraction mixture and dry the sieve for 1 hour at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ($221^{\circ}\text{F} \pm 3.6^{\circ}\text{F}$). Cool the sieve in a desiccator, weigh and calculate the percentage of coarse particles and skins.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 4092.1
January 24, 1986

COARSE PARTICLES AND SKINS IN SYNTHETIC VEHICLE
ENAMELS, LACQUERS AND DOPES

1. SCOPE

1.1 This method provides a means of determining the amount of skins and agglomerated material in synthetic vehicle enamels, lacquers, and dopes.

2. APPARATUS

2.1 Sieve. A No. 325 (44 micron) sieve having a diameter of 3 inches and conforming to RR-S-366.

2.2 Brush. A 1-inch camel-hair brush.

2.3 Weighing device. An analytical or torsion balance sensitive to at least 1 division on indicator per mg, and appropriately accurate weights.

2.4 Desiccator. An ordinary laboratory desiccator equipped with a sample shelf and utilizing a suitable desiccant.

2.5 Dish. A porcelain dish, approximately 8 inches in diameter.

2.6 Oven. A well ventilated gravity convection-type oven thermostatically controlled to the specified temperature $\pm 2^{\circ}$ C.

3. REAGENTS

3.1 Toluene. Toluene conforming to TT-T-548.

3.2 Extraction mixture A:

- 10 volumes ethyl ether - A.C.S. reagent grade.
- 6 volumes benzene - VV-B-231.
- 4 volumes methyl alcohol - O-M-232.
- 1 volume acetone - O-A-51.

3.3 Extraction mixture B:

1:1 mixture of xylene and methyl isobutyl ketone.

4. PROCEDURE

4.1 Thoroughly clean and dry the sieve in an oven at 105° C $\pm 2^{\circ}$ C (221° F $\pm 3.6^{\circ}$ F). Cool the sieve in a desiccator and weigh it to the nearest mg. Weigh to the nearest mg an amount of the sample containing approximately 10 g of

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pigment (25 g of pigment in case of white leads and red lead) into a 250-ml beaker. Add 100 ml of the appropriate solvent from 5.1 if sample is a paste and 50 ml if it is a paint. Thoroughly mix the sample and solvent, breaking up, but not grinding, all lumps with a flattened end of a stirring rod. Wet the sieve on both sides with the solvent. Slowly pour the contents of the beaker into the sieve. When the bottom of the sieve is covered start working the liquid through the sieve by passing the brush back and forth through the liquid. No more pressure shall be applied to the brush than that necessary to keep the tips of the bristles in contact with the bottom of the sieve. Transfer all pigment particles clinging to the stirring rod and walls of the beaker to the sieve using the brush and a stream of the solvent from a wash bottle. After the liquid and rinsings have passed through the sieve place the sieve in an 8-inch porcelain dish containing 250 ml of the solvent, so that the sieve will be immersed to a depth of about 1/2 inch. Move the pigment remaining on the sieve around and over the bottom of the sieve by passing the brush across it at the rate of two strokes per second during two periods of 10 seconds each. After each 10-second period, raise the sieve and let the liquid run through. Clean the dish, add a new portion of the solvent and repeat the operation until the solvent passing over the residue and through the sieve is clear and free of solid particles. When the washing appears to be complete, transfer about 200 ml of the wash solvent to a 400-ml beaker. Stir the solvent vigorously and place the beaker on a black surface in case of white pigments and on a white surface in the case of colored pigments. The washing shall be considered complete only when no particles collect about the middle of the bottom of the beaker. Wash the pigment particles adhering to the brush into the sieve using a stream of extraction mixture from a wash bottle. Finally wash the solvent from the sieve with extraction mixture and dry the sieve for 1 hour at 105° C \pm 2° C (221° F \pm 3.6° F). Cool the sieve in a desiccator, weigh and calculate the percentage of coarse particles and skins.

5. NOTES

5.1 Enamels. For enamels use toluene as the solvent.

5.2 Lacquers and dopes. For lacquers and dopes use extraction mixture A of 3.2.

5.3 In some paints and enamels toluene is not satisfactory and extraction mixture B of 3.3 should be used.

6. PRECISION

6.1 No data.

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METHOD 4121.1
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DRY OPACITY

1. SCOPE

1.1 This method covers two procedures for the application of the coating material to a specially prepared substrate and the subsequent determination of its contrast ratio at the specified spreading rate. Either procedure may be used for routine testing; however the procedure employing the doctor blade^{1/} shall be used in referee tests.

2. APPARATUS

2.1 Hiding power chart. The chart^{2/} shall be smooth-surfaced heavy paper sheet having a design area of 1 square foot. The design shall consist of alternate one-half inch black and white bands laid off in a diamond shape, with a black and white rectangular center. The paper shall be coated with a suitable varnish or lacquer, so as to render the surface impervious and resistant to paint liquids. The directional reflectance of the black portions shall be less than 2 percent and that of the white portion 80 \pm 1 percent, relative to MgO.

2.2 The suction box shall be of appropriate size and of either type of construction described below.

2.2.1 A plane plate glass panel with small holes drilled at appropriate intervals, cemented or fitted on an airtight wooden or metal box. The suction box should be of proper size to accommodate the test panel. Method 2162 gives a design suitable for use with a 3- by 5-inch panel.

2.2.2 A hollow aluminum or steel one piece casting with a smoothly ground plane surface into which are drilled small holes at appropriate intervals.^{3/}

2.3 Film applicator.

2.3.1 Brush. A 1-1/2-inch brush of high quality, clean, free of loose bristles and having a minimum bristle length of 2 inches. The brush shall have good softness and flexibility and shall be carefully cleaned after each day's use. Only brushes which have been thoroughly broken in, rather than brand new brushes, shall be used in the preparation of test panels.

- 1/ Results obtained by this procedure are more reliable because the mechanical method of forming films is more reproducible than hand-brushing.
- 2/ Charts (Form No. 03-B) obtained from the Moresst Co., 211 Centre Street, New York City, are satisfactory. Charts may also be obtained from the International Photo Co., 570 Seventh Avenue, New York City.
- 3/ The Bird Vacuum Plate sold by Bird & Sons, Inc., East Walpole, MA, meets these requirements.

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2.3.2 Doctor blade. A doctor blade, such as the Bird film applicator, which shall be made of metal, preferably noncorrosive, and shall be accurately constructed and smoothly finished, shall be used. It shall spread a film of uniform width and shall have such clearance that the thickness of the resulting wet film of coating applied will be as specified in the product specification.

2.4 Glass syringe. A 10-ml Luer syringe.

2.5 Weighing device. A balance or scale capable of weighing to the nearest 0.01 g.

2.6 Weighing device. An analytical or torsion balance sensitive to at least 1 division on indicator per mg, and appropriately accurate weights. A magnetically dampened balance is useful here.

2.7 Panels. Glass plates, such as window glass, with minimum dimensions of 12 inch by 12 inch.

3. REAGENTS

3.1 None required.

4. PROCEDURES

4.1 Procedure A. Brush application.

4.1.1 Preparation of brush. Dip the brush into the coating material and work until it does not pick up or lose much of the coating in brushing out a normal coat.

4.1.2 Sample measurement. Draw into the syringe the specified volume of the coating material and weigh the brush and syringe to the nearest 0.01 g.

4.1.3 Application of coating. Place the chart in a horizontal position and transfer the coating from the syringe to the charts, moving the syringe over the surface of the chart in such a manner that the stream of coating is fairly evenly distributed over the design area. Brush out the coating as uniformly as possible over the entire design area by brushing in one direction and then rotating the chart one-fourth revolution until the brushing has been completed in four directions. Immediately reweigh the brush and syringe and record the weight of coating applied. Prepare three or four test films following the same procedure but using quantities of the coating material slightly less and slightly more than the volume specified.

4.1.4 Drying of coating. Place the charts with the test coatings in a horizontal position and air dry for 24 hours under the condition described in the specification.

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4.1.5 Calculations.

4.1.5.1 Contrast ratio. Measure the daylight 45 degrees, 0 degrees directional reflectance of the films over both the black and white portions of the rectangular centers, by ASTM E 97. Calculate the contrast ratio for each film cast in 4.1.3 as follows:

$$\text{Contrast ratio} = \frac{\text{Reflectance over black}}{\text{Reflectance over white}}$$

4.1.5.2 Volume. Determine the specific gravity of the coating material as described in ASTM D 1475. From the weight of the coating applied and its specific gravity, compute the number of ml of coating per square foot in each film cast in 4.1.3.

4.1.6 Plotting of curve. Plot ml of coating material (see 4.1.5.2) versus dry contrast ratio (see 4.1.5.1). Draw a smooth curve through the mean of this series of points.

4.1.7 Interpretation of data. From the curve, read the contrast ratio at the specified volume. If the contrast ratio at this point is equal to or greater than that specified, the coating material passes the dry opacity requirements.

4.2 Procedure B. Doctor blade application.

4.2.1 Selection of doctor blades. Table I may be used as a guide in the selection of the proper doctor blades when the specification requirements are stated in terms of either ml per square foot or square feet per gallon. Experience will show which doctor blade is most likely to provide the spreading rate desired with the particular material being tested. On a number of interior flat paints for instance, which were examined, the calculated wet film thicknesses obtained were from approximately 0.0002 to 0.001 inch greater than the figure marked on the film applicators depending upon the one used and the coating material. This statement is given merely to caution the operator against assuming that the figure on the film applicator will necessarily represent the wet film thickness of any coating material laid down by it with this method, and does not mean that its usefulness is impaired, if this is recognized.

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TABLE I. Guide to selection of proper doctor blades.

Wet film thickness (inch)	Corresponding ml per square foot	Approximate spreading rate (square feet per gallon)
0.0015	3.5	1,070
0.002	4.7	800
0.0025	5.9	640
0.003	7.1	535
0.0035	8.3	460
0.004	9.4	400
0.005	11.8	320
0.006	14.2	265

The doctor blades shall be so selected as to give at least three fairly closely spaced points in the range of the specified spreading rate.

4.2.2 Preparation of substrate. Cut several hiding power charts (see 2.1) down the middle lengthwise and trim off one end of the swatch if necessary so that when placed against guides of the suction box it will just cover the last suction holes. Weigh each swatch to the nearest mg.

4.2.3 Application of coating. Apply a film of the coating material to each of the prepared substrates (see 4.2.2), using the selected doctor blades (see 4.2.1), as described (see 2.3.2). Immediately after the coating material has been placed in front of the doctor blade, raise the end of the suction box nearest the operator so that the coating material will flow to the back of the doctor blade, making sure that it flows into both corners. A spatula may be found useful in spreading the material if it is of high viscosity.

4.2.4 Examination of test film. The test films shall be examined by one of the following methods.

4.2.4.1 Method A, wet film. Weigh the coating charts to the nearest mg immediately after coating has been applied and compute the weight of each test film. This can be done with small error if the anticipated weight is calculated in advance and the approximate weights placed on the balance pan. A magnetically damped balance may be used to particular advantage. Dry the test specimen, as described in 4.1.4, and determine the number of square inches of each test film.

4.2.4.1.1 Calculations.

4.2.4.1.1.1 Contrast ratio. Determine the contrast ratio of each test film as described in 4.1.5.1.

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4.2.4.1.1.2 Volume. Determine the specific gravity of the coating material as described in ASTM D 1475 and calculate the volume of each test film in ml per square foot by the following equation:

$$Z = \frac{W \times 144}{S \times A}$$

where:

- Z = Milliliters of coating material per square foot.
- W = Weight of wet coating in grams.
- S = Specific gravity of the coating material.
- A = Area covered by the coating in square inches.

4.2.4.1.1.3 Plotting of curve. Plot a curve as described in 4.1.6 using the values obtained in 4.2.4.1.1.1 and 4.2.4.1.1.2.

4.2.4.1.1.4 Interpretation of data. Follow the procedure described in 4.1.7.

4.2.4.2 Method B, dry film. Dry the test specimens as described in 4.1.4, weigh and compute the weight of each dry test film. Determine the number of square inches in each test film.

4.2.4.2.1 Calculations.

4.2.4.2.1.1 Nonvolatile matter. Determine the fraction of nonvolatile matter in the coating material as follows: Weigh to the nearest mg a pair of glass panels. Sandwich about 2 g of coating material between them and reweigh. Separate the panels by sliding them apart and drying them under the same conditions and for the same period of time as the test films. Reweigh and compute the fraction of nonvolatile matter. This factor and the specific gravity of the coating material (see ASTM D 1475) are used in the following expression to calculate the rate at which the coating material was applied in terms of ml per square foot.

where:

- W = Weight, in grams, of dry test film.
- N = Nonvolatile content of the coating material, expressed as a decimal fraction.
- S = Specific gravity of the coating material.
- A = Area covered by the coating, in square inches.
- Z = Milliliters of coating material per square foot.

$$Z = \frac{W \times 144}{N \times S \times A}$$

Note. If the area of the test film is kept constant the above equation is simplified to:

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$$Z = \frac{W \times K}{N \times S}$$

and since for any given coating material, N and S are constant, the equation may be further simplified to

$$Z = W \times K_1$$

4.2.4.2.1.2 Contrast ratio. Determine the contrast ratio of each test film as described in 4.1.5.1.

4.2.4.2.1.3 Plotting of curve. Plot a curve as described in 4.1.6 using the values obtained in 4.2.4.2.1.1 and 4.2.4.2.1.2.

4.2.4.2.1.4 Interpretation of data. Follow the procedure described in 4.1.7.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 4122.2
January 24, 1986

HIDING POWER (CONTRAST RATIO) -

1. SCOPE

1.1 This method of test covers a procedure for the evaluation of hiding power by establishing a contrast ratio of directional reflectance of a specified dry film thickness coated over black carrara glass to the directional reflectance of the same dry film thickness of coated film over white carrara glass.

2. APPARATUS

2.1 Doctor blade. The doctor blade shall be selected for the desired film thickness by casting a film on a tin panel. The film is air-dried and the film thickness determined with a micrometer by ASTM D 1005 or other suitable method after an air-dry interval of 24 hours. This procedure is repeated until a doctor blade is obtained which will produce the dry film thickness specified in the detail specification.

2.2 Panels. Black and white carrara glass panels of adequate size shall be used. The uncoated black carrara glass shall have a daylight, 45 degrees, 0 degrees directional reflectance of not more than 1 percent, and the uncoated white carrara glass shall have a daylight, 45 degrees, 0 degrees directional reflectance of 86 \pm 2 percent (relative to MgO).

2.2 Micrometer. A micrometer or other suitable means of determining dry film thicknesses to the nearest 0.0001 inch.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 The doctor blade which gave the desired film thickness shall be utilized in casting the coating on the black and white carrara glass. A thoroughly representative portion of paint shall be cast on the carrara glass, using the selected doctor blade as described. An excess of the paint under test shall be poured just in front of the film applicator with the end of the plate nearest to the operator raised so that the paint will flow to the back and both corners of the film applicator. The panel shall be immediately lowered and the drawdown made, continuing the motion down, until the lower end of the panel is reached. The paint film shall then be dried in the prescribed manner in a dust free cabinet. The reflectance of the coated black and white carrara glass shall then be determined in accordance with ASTM E 97. The contrast ratio shall be calculated by dividing the reflectance value of the coated black panel by the reflectance of the coated white panel.

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METHOD 4122.2
January 24, 1986

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 4203.1
January 24, 1986

REDUCIBILITY AND DILUTION STABILITY

1. SCOPE

1.1 This method provides a procedure for determining the reducibility of a coating material with the recommended solvent for that material.

2. APPARATUS

2.1 A 100-ml stoppered graduated cylinder.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Material preparation.

4.1.1 The material shall be diluted with the amount of the solvent required in the product specification or according to the manufacturer's instructions, whichever applies.

4.1.2 Unless otherwise specified, all materials used in and during the test shall be at a temperature of $23^{\circ}\text{C} \pm 1.1^{\circ}\text{C}$ ($73.4^{\circ}\text{F} \pm 2^{\circ}\text{F}$).

4.2 Observation upon mixing. The material shall mix readily and easily without excess stirring or shaking.

4.3 Observations upon standing. Let the diluted material stand undisturbed for 4 hours unless otherwise specified in the product specification. At the end of the standing period, freedom from the following defects shall be observed:

Curdling.

Precipitation.

Separation.

4.4 If doubt exists as to the condition of the material after the standing period, flow an amount of the material onto a glass panel without agitating it. Any of the defects mentioned in 4.3 will then be observable.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 4241.1
January 24, 1986

COLOR OF TRANSPARENT LIQUIDS (ACID-POTASSIUM DICHROMATE SCALE)

1. SCOPE

1.1 This method covers the preparation and use of primary standards for the color specification of liquids, such as oils, varnishes, resin solutions, etc., used in organic coating materials.

2. APPARATUS

2.1 Tubes. Gardner bubble tubes^{1/} made of clear glass with a closed flat, even bottom and having the following dimensions.

2.1.1 A uniform internal diameter of 10.75 ± 0.025 mm throughout the length of the tube.

2.1.2 An internal length of 112 ± 0.05 mm.

2.1.3 An etched line around the outside of the tube 5 mm from the open end.

2.1.4 A second line around the outside of the tube 13 mm from the open end.

3. REAGENTS

3.1 A.C.S. reagent grade of potassium dichromate.

3.2 A.C.S. reagent grade sulfuric acid.

4. PROCEDURE

4.1 Preparation of standard. Dissolve the specified weight of the dichromate in 100 ml of the sulfuric acid. Gentle heat may be used, if necessary, to effect complete solution of the dichromate. Excessive temperatures or prolonged heating causes abnormal darkening of the solution.

4.2 Performance of the test. Fill one of the tubes with the material under test, and a second tube with the dichromate solution. Place the tubes close together, and holding them against a background substantially equal in illumination to that of a fairly light overcast northern sky, make the comparison by viewing the light transmitted through the liquids.

^{1/} Suitable tubes may be obtained from the Gardner Laboratory, Inc., 2431 Linden Lane, Silver Spring, MD.

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METHOD 4241.1
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5. NOTES

5.1 Solutions of potassium dichromate and sulfuric acid are not stable and must be freshly prepared for each test. Hence suitable stable secondary standards (see methods 4242 and ASTM D 1544) of equivalent color range may be used as a matter of convenience. However, in cases where the color of the material under examination is found to be darker than the secondary standard the dichromate solution shall be made up for the final decision.

6. PRECISION

6.1 No data.

METHOD 4242
January 24, 1986

COLOR OF TRANSPARENT LIQUIDS (HELLIGE SCALE)

1. SCOPE

1.1 This method covers the use of a series of glass color standards for the color specification of liquids, such as oils, varnishes, resin solutions, etc., used in organic coating materials.

2. APPARATUS

2.1 A Hellige color comparator.

2.1.1 Color standards. The Hellige glass color standards for varnish. These standards consist of eighteen filters mounted in two wheels. The filters in one wheel are numbered from 1 to 9 and in the other wheel 1L to 9L. Filter numbers 1L to 9L are lighter in color than the corresponding filters numbered 1 to 9.

2.2 Sample container. A glass tube, as Gardner bubble tubes^{1/}, made of clear glass with a closed flat, even bottom and having the following dimensions.

2.2.1 A uniform internal diameter of 10.75 \pm 0.025 mm throughout the length of the tube.

2.2.2 An internal length of 112 \pm 0.05 mm.

2.2.3 An etched line around the outside of the tube 5 mm from the open end.

2.2.4 A second line around the outside of the tube 13 mm from the open end.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Fill the tube with the material and place it in the comparator. Compare the color of the light transmitted by the material under test with that transmitted by the specified standard.

5. NOTES

5.1 The Lovibond equivalents of filters 1 through 9 are shown in the following table.

^{1/} Suitable tubes and reference standards may be obtained from the Gardner Laboratory, Inc., Bethesda, MD.

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6. PRECISION

6.1 No data.

Combinations of Lovibond glasses to approximate Hellige standards.

Hellige standard	Lovibond glasses	
	Red	Yellow
1	0.3	1.6
2	0.7	3.2
3	1.1	5.6
4	1.9	10.3
5	3.1	17.3
6	5.0	27.0
7	9.0	39.0
8	16.5	54.5
9	40.0	80.0

METHOD 4250.1
January 24, 1986

COLOR OF PIGMENTED COATINGS

1. SCOPE

1.1 This method covers a procedure for the comparison of the color of a dried film of pigmented organic coating material, with that of (1) a dried film similarly prepared from a liquid reference standard, (2) a previously prepared reference film, or (3) a designated color chip.

2. APPARATUS

2.1 None.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Preparation of film. Apply a film of the sample under test to the designated substrate in the manner and to the thickness specified in the product specification. If a liquid reference standard is specified, prepare a film of it in the same manner. Dry the test film, or the test and reference films, as the case may be, under the conditions prescribed in the applicable specification.

4.2 Comparison of color. Visually compare the color of the test film with that of the reference film as received or prepared in 4.1, or with the color chip by viewing them under an illumination substantially equal to that of a fairly light overcast northern sky. Arrange the two films or the test film and color chip beside each other on a table in front of a north light so that the illumination centers on an angle of about 45 degrees and the viewing is nearly normal to the surfaces to be compared. Prevent appreciable quantities of extraneous light from reaching the specimen by covering nearby bright walls and ceilings with black cloth. Exclude all adjacent colors from view by placing a piece of neutral paper with a square section cut from the center over the specimens, so that one-half of the square will be over the test film and the other half over the reference film or chip.

4.2.1 When it is necessary to compare films of different degrees of gloss, wet the films with water or heavy water-white mineral oil, if water does not wet the films, before making the comparison.

5. NOTES

5.1 If in doubt about the exactness of the color match, the test shall be made in accordance with ASTM D 2244 or other equivalent procedure and equipment. The unit or units of color difference allowable shall be stated in the product specification.

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METHOD 4250.1
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6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 4251
January 24, 1986

COLOR SPECIFICATION FROM SPECTROPHOTOMETRIC DATA

1. SCOPE

1.1 This method utilizes spectrophotometric data and the CIE (I.C.I.) standard observer^{1/2/} and coordinate system to specify colors of nonfluorescent specimens.

2. APPARATUS

2.1 A spectrophotometer shall be used to measure the relative spectral radiance factors of a specimen throughout the visible spectrum.

2.2 The directions of illuminating and viewing measured from the perpendicular to the illuminated face of the test specimen shall be within 10 degrees of those indicated in table I. The illuminating and viewing conditions may be interchanged without altering the results.

2.2.1 For reflectance measurements, instruments having different viewing geometries are permitted, namely either 45 degrees directional viewing, or diffuse viewing. Diffuse reflectance measurements of glossy specimens may be made with or without inclusion of the specular component of reflected flux.

2.3 Standard sources. Standard source A shall be used for the colors of specimens in incandescent-lamp light; standard source C for the colors of specimens in daylight. The relative spectral irradiances, H_λ , of these two standard sources have been combined with the spectral tristimulus values to obtain the values in tables II and III.

2.4 Standards.

2.4.1 For transparent specimens, the standard is the source.

2.4.2 For opaque specimens, the standard is a freshly prepared layer of magnesium oxide of 0.5 mm minimum thickness identically irradiated and viewed.^{3/}

TABLE I. Illuminating and viewing conditions.

Measurement	Geometric directions	
	Illuminating	Viewing
Transmittance	0°	180°
Reflectance	0°	45°
	0°	Diffuse

1/ Proceedings, Eighth Session, Commission Internationale de l'Eclairage, Cambridge, England, September 1931, pp. 19-29.

2/ D. B. Judd, the 1931 I.C.I. Standard Observer and Coordinate System for Colorimetry, J. Opt. Soc. Am., 23, pp. 357-374 (1933).

3/ Preparation and colorimetric properties of a magnesium-oxide reflectance standard as included in ASTM D 986.

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3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Calibration.

4.1.1 Check the calibration of the photometric scale at reasonable time intervals to insure accuracy over the entire range by measuring relative spectral radiance factors of spectrophotometric standards.^{4/}

4.1.2 Calibrate the wavelength scale at reasonable intervals by using spectrum sources or a didymium filter.

4.1.3 Use spectrometer slits sufficiently narrow so that slit-width error is negligible.

4.2 Handle test specimens carefully to avoid contamination or discoloration.

4.3 Determine relative spectral radiance factors throughout the visible spectrum at intervals sufficiently close so that the curve drawn by interpolation will not be changed by measurements at additional wavelengths. Ten-millimicron intervals are usually sufficient.

4.4 Computations.

4.4.1 Multiply for each wavelength the relative spectral radiance factors of the specimen, R_λ , by the products of spectral irradiance of the designated standard source, H_λ and the spectral tristimulus values, x_λ , y_λ , and z_λ , as contained in table II or III.

4.4.2 Sum the products computed in 4.4.1 and divide by 10^5 to obtain the specimen tristimulus values, X, Y, and Z. If required, calculate the chromaticity coordinates, x and y, from the following definitions:

$$x = X/S, y = Y/S, S = X + Y + Z$$

^{4/} Spectrophotometric standards may be purchased from the National Bureau of Standards.

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TABLE II. Computation form for CIE standard source A (2854° K).

λ	$\bar{x}(H_A)\lambda$	$\bar{y}(H_A)\lambda$	$\bar{z}(H_A)\lambda$	R λ	$\bar{x}(H_{AR})\lambda$	$\bar{y}(H_{AR})\lambda$	$\bar{z}(H_{AR})\lambda$
380	1		6	0.--- -- --			
90	5		23	... -- --			
400	19	1	93	... -- --			
10	71	2	340	... -- --			
20	262	8	1256	... -- --			
30	649	27	3167	... -- --			
40	926	61	4647	... -- --			
450	1031	117	5435	... -- --			
60	1019	210	5851	... -- --			
70	776	362	5116	... -- --			
80	428	622	3636	... -- --			
90	160	1039	2324	... -- --			
500	27	1792	1509	... -- --			
10	57	3080	969	... -- --			
20	425	4771	525	... -- --			
30	1214	6322	309	... -- --			
40	2313	7600	162	... -- --			
550	3732	8568	75	... -- --			
60	5510	9222	36	... -- --			
70	7571	9457	21	... -- --			
80	9719	9228	18	... -- --			
90	11579	8540	12	... -- --			
600	12704	7547	10	... -- --			
10	12669	6356	4	... -- --			
20	11373	5071	3	... -- --			
30	8980	3704		... -- --			
40	6558	2562		... -- --			
650	4336	1637		... -- --			
60	2628	972		... -- --			
70	1448	530		... -- --			
80	804	292		... -- --			
90	404	146		... -- --			
700	209	75		... -- --			
10	110	40		... -- --			
20	57	19		... -- --			
30	28	10		... -- --			
40	14	6		... -- --			
750	6	2		... -- --			
60	4	2		... -- --			
70	2			... -- --			
	109828	100000	35547	S= X + Y + Z	X=	Y=	Z=
	X_A	Y_A	Z_A				
	.4476	.4075	.1449				
	(x=X/S, y=Y/S, z=Z/S)				x=	y=	z=

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TABLE III. Computation form for CIE standard source C (average daylight).

λ	$\bar{x}(H_c)\lambda$	$\bar{y}(H_c)\lambda$	$\bar{z}(H_c)\lambda$	$R\lambda$	$\bar{x}(H_c R)\lambda$	$\bar{y}(H_c R)\lambda$	$\bar{z}(H_c R)\lambda$
380	4		20	0.---			
90	19		89	--- --			
400	85	2	404	--- --			
10	329	9	1570	--- --			
20	1238	37	5949	--- --			
30	2997	122	14628	--- --			
40	3975	262	19938	--- --			
450	3915	443	20638	--- --			
60	3362	694	19229	--- --			
70	2272	1058	14972	--- --			
80	1112	1618	9461	--- --			
90	363	2358	5274	--- --			
500	52	3401	2864	--- --			
10	89	4833	1520	--- --			
20	576	6462	712	--- --			
30	1523	7934	388	--- --			
40	2785	9149	195	--- --			
550	4282	9832	86	--- --			
60	5880	9841	39	--- --			
70	7322	9147	20	--- --			
80	8417	7992	16	--- --			
90	8984	6627	10	--- --			
600	8949	5316	7	--- --			
10	8325	4176	2	--- --			
20	7070	3153	2	--- --			
30	5309	2190		--- --			
40	3693	1443		--- --			
650	2349	886		--- --			
60	1361	504		--- --			
70	708	259		--- --			
80	369	134		--- --			
90	171	62		--- --			
700	82	29		--- --			
10	39	14		--- --			
20	19	6		--- --			
30	8	3		--- --			
40	4	2		--- --			
750	2	1		--- --			
60	1	1		--- --			
70	1			--- --			
	98041	100000	118103				
	x_c	y_c	z_c	$S = X + Y + Z$	$X =$	$Y =$	$Z =$
	.3101	.3163	.3736				
	$(x=X/S, y=Y/S, z=Z/S)$				$x =$	$y =$	$z =$

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4.5 Reporting results.

4.5.1 Report the tristimulus values, X, Y, and Z, or the relative luminance factor, Y, and the chromaticity coordinates, x and y.

4.5.2 Report the standard source used in the computations.

4.5.3 Report the illuminating and viewing conditions of measurement.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 4252
January 24, 1986

COLOR SPECIFICATION FROM PHOTOELECTRIC TRISTIMULUS DATA

1. SCOPE

1.1 This method utilizes data from photoelectric tristimulus colorimeters to specify colors of nonfluorescent specimens, in approximate accord with the 1931 CIE (I.C.I.) standard observer and standard source C (average daylight). Accurate color measurement of test specimens by this method is possible only with color standards that have been calibrated accurately from spectrophotometric data by method 4251 and that have spectral characteristics similar to those of the specimens.

2. APPARATUS

2.1 Components. The apparatus, in which specimens are illuminated and viewed under controlled geometric conditions, shall consist of an incandescent lamp source, one or more photocells, and an appropriate tristimulus-filter set.

2.2 Illuminating and viewing conditions. The directions of illuminating and viewing measured from the perpendicular to the illuminated face of the test specimen shall be within 10 degrees of those indicated in table I. The illuminating and viewing conditions may be interchanged without altering results.

2.2.1 For reflectance measurements, instruments having either of the viewing geometries listed in table I are permitted. Diffuse reflectance measurements of glossy specimens may be made with or without inclusion of the specular component of reflected flux.

2.3 Tristimulus-filter set. Filters of the tristimulus-filter set may be interposed in succession either between the source and the test specimen or between the test specimen and the viewing photocell. Alternatively, several photocells may be used and each filter of the set may have its own photocell. The filter set may be designed to produce source-filter photocell response functions which will give colorimeter readings directly proportional to the CIE tristimulus values, X, Y, and Z, or to produce response functions such that the CIE tristimulus values can be derived from the colorimeter readings.

TABLE I. Illuminating and viewing conditions.

Measurement	Geometric directions	
	Illuminating	Viewing
Transmittance	0°	180°
Reflectance	0°	45°

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2.4 Standard source. The equivalent of CIE standard source C shall be used to obtain the colors of specimens for average daylight.

2.5 Standards.

2.5.1 Primary standards.

2.5.1.1 For transparent specimens, the primary standard is the source.

2.5.1.2 For opaque specimens, the primary standard is a freshly prepared layer of magnesium oxide of 0.5 mm minimum thickness identically irradiated and viewed.

2.5.2 Secondary standards. Secondary standards having spectral characteristics similar to those of the test specimens shall be used since the usual source-filter-photocell combinations only approximate the CIE functions. Tristimulus values, X, Y, and Z for source C for master secondary standards shall be obtained according to method 4251. Working standards having nearly the same spectral character as a master standard may be calibrated by direct photoelectric comparison of tristimulus values.

2.5.2.1 For transparent specimens, glass filters may be used as secondary standards.

2.5.2.2 For opaque specimens, porcelain-enameled plaques known to be reasonably permanent in color characteristics may be used repeatedly or impermanent materials such as Munsell papers may be used and discarded frequently. The geometric conditions of standardization for reflectance standards should be the same as those of the colorimeter.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Select a standard having spectral characteristics similar to those of the test specimen.

4.2 Set the colorimeter to read the values assigned to this standard based on calibration relative to a primary standard.

4.3 Read the values of the test specimen.

4.4 Calculations.

4.4.1 Calculate the tristimulus values, X, Y, and Z, as prescribed in the instructions for use of the colorimeter.

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4.4.2 If required, calculate the chromaticity coordinates, x and y , from the following definitions:

$$x = X/S, \quad y = Y/S, \quad S = X+Y+Z$$

4.5 Report.

4.5.1 Report the tristimulus values, X , Y , and Z , or the luminance factor, Y , and the chromaticity coordinates, x and y .

4.5.2 Report the illuminating and viewing conditions of measurement.

5. NOTES

5.1 Definitions.

5.1.1 Color. An appearance characteristic of a specimen determined by the relative spectral irradiance from the source, the relative spectral radiance factor of the specimen, and the spectral tristimulus values of the CIE standard observer.

5.1.2 Relative luminance factor. The ratio of the luminance of a specimen for specified geometric conditions of illumination and view to the luminance of a standard. If the luminance of the standard is unity, the relative luminance factor is the tristimulus value, Y .

5.1.3 Tristimulus-filter set. A set of filters designed to provide receptor response functions for a specified source approximating the spectral tristimulus values of the CIE standard observer for source C.

6. PRECISION

6.1 No data.

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METHOD 4254.1
January 24, 1986

COLOR OF LAC RESIN AND SHELLAC VARNISH
BY COMPARISON OF SOLUTIONS

1. SCOPE

1.1 This method is intended for use in the evaluation of the color of solutions of dry lac resins or shellac varnishes by comparison with solutions of the standard resins or varnishes as agreed upon between buyer and seller.

2. APPARATUS

2.1 Glass tubes. As specified in 2.1 of ASTM D 1545.

2.2 Comparison standards. As agreed upon by the buyer and the seller.

2.3 Weighing device. A balance or scale capable of weighing to the nearest 0.1 g.

2.4 Flask. A 150 ml glass-stoppered Erlenmeyer flask.

3. REAGENTS

3.1 Alcohol. The alcohol used in "cutting" (dissolving) the dry lac resin or thinning the shellac varnish shall meet the requirements of either 95 percent (190 proof) ethyl alcohol, specially denatured according to formula No. 1, or in the proprietary solvent authorized for "cutting" shellac by the United States Bureau of Internal Revenue Service.

4. PROCEDURE

4.1 Lac resin. Weigh 10 \pm 0.1 g each of the sample of lac resin and the comparison resin into separate Erlenmeyer flasks. Add to each flask an amount of the alcohol equal to twice the weight of the resin and stopper the flasks. Keep the flasks at 21° C to 32° C (70° F to 90° F) and shake at frequent intervals until the resins are completely "cut". Allow the flasks to stand undisturbed for 1/2 hour and then fill separate glass tubes to their bottom lines with each of the solutions, being careful not to disturb any settlement that may have occurred in either flask. Place the tubes side by side and make the color comparison by viewing the light transmitted through the liquids while they are held against a background substantially equal in illumination to that of a fairly light overcast northern sky.

4.2 Shellac-varnish. Thoroughly agitate both the sample varnish and the comparison varnish and determine their nonvolatile contents by ASTM D 2369. Adjust the nonvolatile contents of both varnishes to 33-1/3 percent by weight by adding the alcohol to equal portions (approximately 30 g) of each varnish in separate Erlenmeyer flasks. Stopper the flasks, shake the contents thoroughly,

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and let stand undisturbed for 30 minutes. Continue with the procedure as prescribed in 4.1. Record whether the color of the sample is darker or not darker than that of the comparison standard.

5. NOTES

5.1 The shellac varnish used as the comparison standard must be stored in a tightly sealed glass container and in a dark place.

6. PRECISION

6.1 No data.

METHOD 4261
January 24, 1986

APPEARANCE OF TRANSPARENT LIQUIDS

1. SCOPE

1.1 This method provides a procedure for determining the appearance of clear materials of both a drying and nondrying nature. The materials are checked upon decantation, standing 24 hours, and after agitation.

2. APPARATUS

2.1 Tubes. Gardner bubble tubes made of clear glass with a closed flat, even bottom, a diameter of 10.75 \pm 0.025 mm and an internal length of 112 \pm 0.05 mm.

2.2 Water bath. A water bath capable of maintaining the specified temperature \pm 0.5° C and equipped with a stirring device.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Thoroughly mix the sample. Fill two test tubes of the same size (15 cm or (6 inches) to within 2.5 cm (1 inch) of the top with the sample. Stopper the tube with clean corks.

4.2 Materials other than drying oils. Immediately examine the contents of the tubes by transmitted light for haze, turbidity, hair, grain, clots; gell bodies, skins, and other insoluble matter that may be in suspension. Tilt one of the tubes at a small angle from the horizontal so that the air bubble will move slowly and permit observation in the thin moving film of any fine particles that might otherwise escape notice. Let the tubes stand undisturbed for 24 hours at 21° C to 32° C (70° F to 90° F) and examine for sediment. Shake one of the tubes vigorously and as soon as the air has escaped from the liquid compare the appearance of the liquids in the two tubes.

4.3 Drying oils. If the oil is cloudy at room temperature, warm the tubes and their contents in a water bath to 65° C (149° F) and hold for 5 minutes. Examine while warm by transmitted light and tilting the tubes as described in 4.2.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 4287
January 24, 1986

VISCOSITY (BROOKFIELD)

1. SCOPE

1.1 This method provides a procedure for determining viscosity of viscous material.

1.2 A fluid's viscosity is a measure or index of its internal friction. If a body is rotated within a liquid at a definite speed, shearing will be induced. The exact shearing within a liquid as induced by a rotating body will depend on (a) the rotation speed of the body; (b) the dimensions of the body, and (c) in many cases the dimensions of the container in which the body is held. The Brookfield Synchro-Lectric Viscosimeter is a rotational instrument which induces rotation of a spindle or rotor immersed in the liquid and attached to the instrument. The amount of force required to produce rotation may be read directly from the instrument's dial.

2. APPARATUS

2.1 Viscosimeter. A Brookfield Model RVF Viscosimeter with guard.

2.2 Sample container. A 600 ml Low Form Griffin Beaker.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of sample. Thoroughly mix and strain not less than 400 ml of the sample, allowing it to stand sufficient time (at least 1 hour) for all entrapped air to escape.

4.2 Performance of test.

4.2.1 Bring the material to be tested to $25.0^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ in the 600 ml beaker and maintain this temperature until testing is completed.

4.2.2 Attach the specified spindle to the lower shaft using care to avoid side thrusts on the shaft which may interfere with its alignment.

4.2.3 Immerse the spindle in the test sample, tilting the apparatus slightly at first to prevent the entrapment of air bubbles on the lower surface of the spindle. Immerse the spindle to the immersion groove cut in the spindle's shaft. The No. 7 spindle has a necked-down area instead of an immersion groove and should be immersed to the middle of this area.

4.2.4 Level the viscosimeter with the attached bubble level.

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4.2.5 Depress the clutch and turn on the viscosimeter, then release the clutch. Adjust the viscosimeter to the r.p.m. specified for the material under test. Allow the viscosimeter to run until the pointer has stabilized, usually 20 to 30 seconds or one revolution, depending upon the specified r.p.m. After the pointer has stabilized, depress the clutch and snap off the motor so that when it stops, the pointer will be in gear.

4.2.6 If check readings are required, restart the viscosimeter with the clutch still depressed at the original reading, then release. This will speed the check reading by reducing the time required for the pointer to stabilize.

4.3 Alternate method. Follow the procedure above, except place a correction shield around the guard to stimulate the boundaries of a 600 ml Low Form Griffin Beaker.

4.4 Results. Multiply the readings obtained by the appropriate factor which is determined by the spindle used, as well as the r.p.m. and which is supplied with the viscosimeter. This procedure will give the viscosity in centipoises.

5. NOTES

5.1 Required information.

5.1.1 The spindle to be used.

5.1.2 The r.p.m. to be used.

5.2 Care of the instrument.

5.2.1 The spindle shall be removed before cleaning. No metal or hard bristle brushes are to be used on the spindle.

5.2.2 Exercise care that at no time shall the lower shaft of the viscosimeter receive side thrusts because these may put the instrument out of alignment.

6. PRECISION

6.1 No data.

METHOD 4321.2
January 24, 1986

BRUSHING PROPERTIES

1. SCOPE

1.1 This method provides a means for determining the brushing properties of coatings. This test is quite subjective; however, someone experienced in the art can produce quite consistent results, particularly in the determination of the "drag" properties.

2. APPARATUS

2.1 Brush. A 2-1/2-inch wall brush of good quality.

2.2 Panel. A 2- by 2-foot cold-rolled steel or aluminum panel.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Application to metal panels.

4.1.1 Panel preparation.

4.1.1.1 The panel shall be solvent cleaned.

4.1.2 Application of material under test.

4.1.2.1 Prewet the brush with the coating to be applied. The idea is to have as much of the coating in the brush prior to beginning the test as there is at the finish of the test.

4.1.2.2 The coating shall be applied at 500 square feet per gallon unless otherwise specified.

4.2 Application to sheet rock panels.

4.2.1 Panel preparation.

4.2.1.1 The panel shall be a 2- by 4-foot piece of 1/4-inch gypsum sheet rock meeting SS-L-30.

4.2.1.2 Unless otherwise specified, the panel shall be primed with an undercoater meeting the minimum requirements of TT-S-179 (see 5.1).

4.2.1.3 Allow to dry at least 24 hours prior to applying the material under test.

FED. TEST METHOD STD. NO. 141C

METHOD 4321.2
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4.2.2 Application of material under test.

4.2.2.1 Apply the material at a spreading rate of 500 square feet per gallon unless otherwise specified.

4.2.2.2 The material shall be applied in sections, first brushing the coating using back and forth strokes, then coming back and leveling the material by brushing to 90 degrees of the first strokes.

4.2.2.3 Proceed to the next section always working to a "wet edge". Repeat this.

4.2.3 Place the panel in a vertical position.

4.3 Observations.

4.3.1 Note whether the material under test brushes easily and has good flowing and spreading qualities.

4.3.2 After the film has dried, the surface should be inspected for freedom of lap marks (see 5.2). Note shall be made of gloss variations and obvious brush marks.

5. NOTES

5.1 It should be realized that when the undercoater is the material under test, the top coat shall be a standard product and will be specified in the product specification.

5.2 Depending upon the product being tested, different characteristics will of necessity be stressed in the product specification.

6. PRECISION

6.1 No data.

METHOD 4331.1
January 24, 1986

SPRAYING PROPERTIES

1. SCOPE

1.1 The following method outlines a procedure for checking the spraying properties of coating materials. The method is very subjective and should be performed by an individual skilled in the art of using a spray gun.

2. APPARATUS

2.1 A steel panel 4 by 12 inches or larger.

2.2 A spray gun capable of spraying the material under test.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Panel preparation.

4.1.1 The steel panel shall be solvent cleaned.

4.2 Performance of test.

4.2.1 The material shall be reduced according to the instructions in the product specification.

4.2.2 While spraying, the gun shall be held perpendicular to the panel and moved in a straight line across the face of the panel.

4.2.3 The product specification shall state the distance from the test panel to the gun. For lacquer and other quick drying materials the distance shall be between 6 and 8 inches. For slower drying materials the distance shall be 8 to 10 inches.

4.2.4 Immediately place the panel in a nearly vertical position.

4.3 Observations.

4.3.1 On the wet panel freedom from the following shall be noted:

running
sagging
fogging

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4.3.2 The dried film shall be free of the following defects:

dusting	pimoling
floating	cratering
mottling	orange peel
bubbling	blushing
wrinkling	blooming
streaking	silking

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 4335
January 24, 1986

ROLLER COATING PROPERTIES

1. SCOPE

1.1 This method outlines a procedure for making an evaluation of a material's characteristics when applied by roller.

2. APPARATUS

2.1 Standard panels prepared as per method 2081.

2.2 The equipment listed in method 2112.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Panel preparation.

4.1.1 Prepare the test panel as described in method 2081.

4.1.2 Apply the roller coating in the manner described in method 2112.

4.2 Allow the panels to dry in a vertical position for the time and under the conditions required in the product specification.

4.3 Observation.

4.3.1 After the material under test is dry, note whether a sufficiently uniform film exists so as to prevent the visual detection of "dimples" because of localized lack of hiding.

4.3.2 Characteristics such as lapping, localized floating, and sagging shall be noted.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 4371
January 24, 1986

REFRACTIVE INDEX

1. SCOPE

1.1 This method gives a procedure which is particularly useful in detecting adulteration in oils containing substantial amounts of conjugation, such as tung, oiticica, and dehydrated castor oils (see 5.1).

1.2 Since refractive index varies with iodine value, this method can be used as a quick approximation of iodine value.

2. APPARATUS

2.1 An Abbe refractometer or other equally accurate instrument.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of test specimen. When the refractive index is required on the material as received, no preparation will be necessary. On the other hand, when the determination is to be made on a recovered ingredient, care shall be taken to remove all traces of impurities such as inorganic acids, solvents, etc.

4.2 Performance of test. Following the instructions for the operation of the instrument, determine the refractive index of the test specimen at the temperature specified in the product specification.

5. NOTES

5.1 Refractive index is not a very reliable means of specifying drying oils.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 4411.1
January 24, 1986

FINENESS OF GRIND

1. SCOPE

1.1 This method is intended to measure the degree of dispersion (commonly referred to as "fineness of grind") of pigment in vehicle systems such as paint, enamel, etc.

2. APPARATUS^{1/}

2.1 The apparatus shall consist of a device having two parts shown in figures 1 and 2 and made in accordance with the following requirements.

2.1.1 Gage. A hardened steel block (see figure 1) approximately 7 inches in length, 2-1/2 inches in width, and 1/2 inch in thickness. The top surface of the block shall be ground smooth and flat and shall contain a groove 5-1/4 inches in length and 1/2 inch in width centered in the top of the block. The groove shall be tapered uniformly in depth lengthwise from 4 mils at one end to zero depth at the other, calibrated in accordance with its depth. The groove shall have the dimensional requirements corresponding to the Hegman Standard Graduations as shown in table I.

TABLE I. Dimensional requirements of groove for determination of fineness of grind.

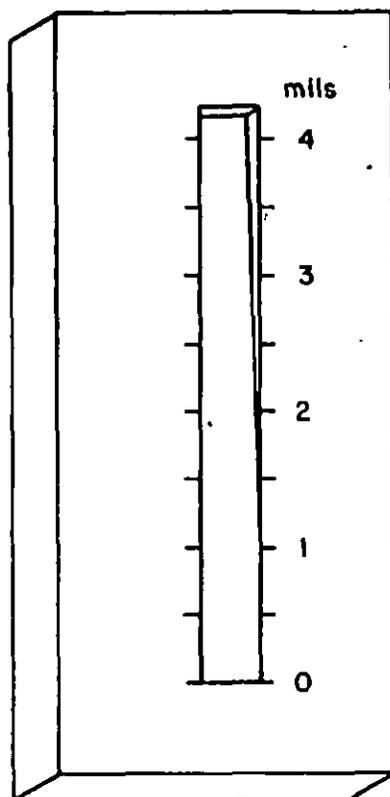
Distance from zero end-inches	Depth mils	Hegman std. graduations
5-1/4	4+	Well for sample.
5	4.0	0
4-3/8	3.5	1
3-3/4	3.0	2
3-1/8	2.5	3
2-1/2	2.0	4
1-7/8	1.5	5
1-1/4	1.0	6
5/8	0.5	7
0	0	8

2.1.2 Scraper. A double-wedge steel blade (see figure 2) 3-1/2 inches long, 1-1/2 inches wide, and 1/4 inch thick. The two edges on the long sides shall be rounded to a radius of approximately 0.01 inch.

^{1/} A device known as the Hegman gage.

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METHOD 4411.1
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NOTE:
A DEVICE KNOWN AS THE HEGMAN GAGE.

FIGURE 1. Fineness gage.

X-4466

METHOD 4411.1
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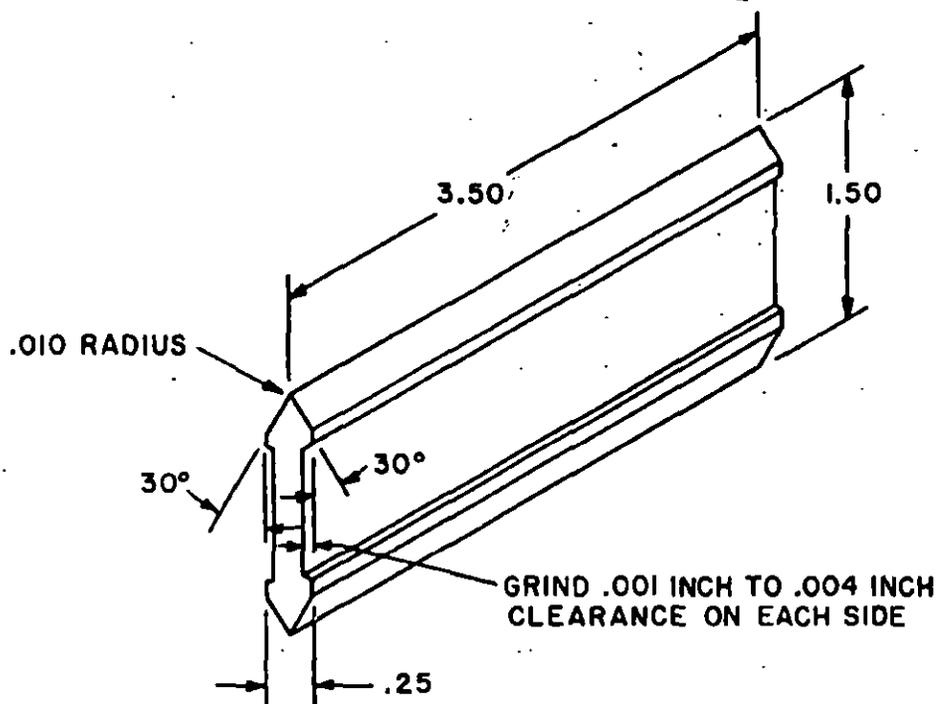


FIGURE 2. Scraper.

X-4467

FED. TEST METHOD STD. NO. 141C

METHOD 4411.1
January 24, 1986

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of sample.

4.1.1 Thoroughly mix and strain an appropriate amount of the material to be tested. Allow to stand for at least one hour in a partially filled closed container to permit all entrapped air to escape.

4.2 Performance of test. Place the thoroughly cleaned and dried gage on a flat, nonslippery surface. Pour a sufficient amount of the prepared sample (see 4.1) into the deep end (well) of the groove so that it overflows the groove slightly. Grasp the scraper between the thumbs and fingers of both hands and place it edgewise in contact with the surface of the gage at the extreme deep ends of the grooves with the long dimension of the scraper across the width and parallel to the short dimension of the gage. While holding the scraper perpendicular to the surface of the gage and at right angles to the length of the grooves, draw it at a uniform moderate rate over the surface of the gage to a point beyond the zero ends of the grooves (graduation 8). Sufficient downward pressure should be exerted on the scraper to just fill the grooves and clean the level surface of the gage. Immediately (see 5.1) determine the fineness value of the material by viewing the gage from the side in such a manner that the line of vision is at right angles to the long dimension of the grooves and not more than 30 degrees nor less than 20 degrees to the face of the gage while it is held in a light that will make the pattern of the material in the grooves readily visible. Observe the point along the groove where the material first shows a predominantly speckled appearance and note the number between the 0 and 8 graduations that most nearly corresponds to that point. Pictorial examples are shown in 5.2. Report this number as the fineness of grind. Disregard any scattered specks which appear prior to the point where the predominantly speckled appearance begins.

5. NOTES

5.1 No reading shall be considered for reporting the fineness when the time lapse exceeds 10 seconds. For this reason it is often advisable to make a preliminary determination to establish the approximate position of the first appearance of a predominantly speckled surface. A second and more accurate reading can then be made very rapidly.

5.2 Pictorial standards.

6. PRECISION

6.1 Readings should be reproducible within $\pm 1/2$ mil division if visual standards are used properly.

FED. TEST METHOD STD. NO. 141C

METHOD 4411.1
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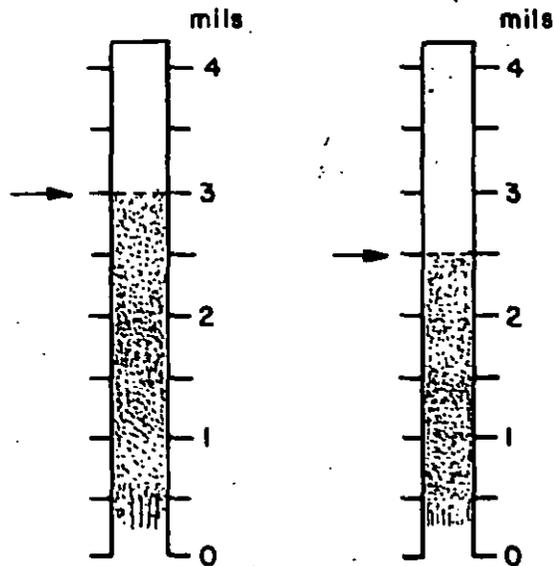


FIGURE 3. Fineness of grind values, 3.0 and 2.5.

X-4468

FED. TEST METHOD STD. NO. 141C

METHOD 4411.1
January 24, 1986

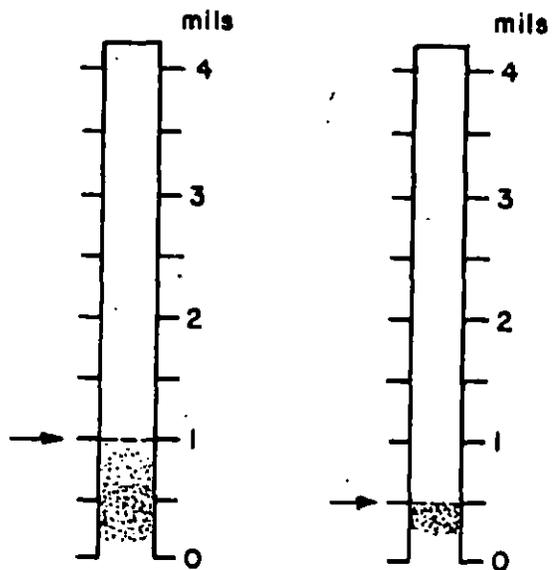


FIGURE 4. Fineness of grind values, 1.0 and 0.5.

X-4469

METHOD 4411.1
January 24, 1986

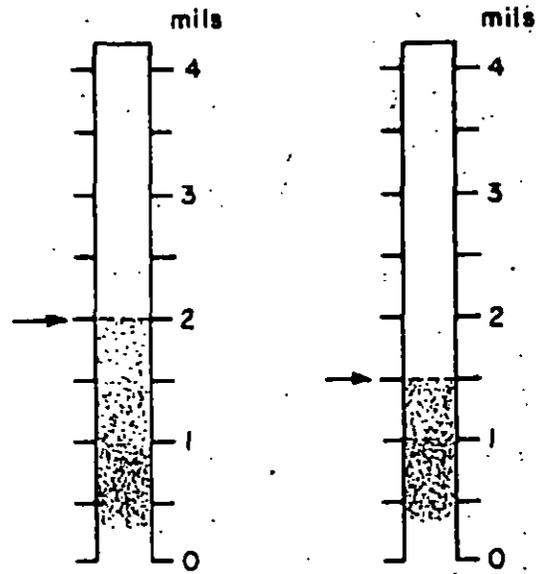


FIGURE 5. Fineness of grind values, 2.0 and 1.5.

X-4470

FED. TEST METHOD STD. NO. 141C

METHOD 4421
January 24, 1986

ABSORPTION TEST

1. SCOPE

1.1 This method gives a rapid but subjective determination of the penetrative or wetting ability of various liquid materials.

2. APPARATUS

2.1 One-half pint friction-top can plug (5-1/2 cm in diameter).

2.2 No. 2 Whatman filter paper or equal.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Fill a one-half pint friction-top can plug (5-1/2 cm in diameter) level full with a portion of the thoroughly mixed sample and then place a No. 2 Whatman filter paper (12.5 cm is a convenient size) flat on the surface of the liquid with the center of the filter paper approximately over the center of the liquid. Allow to remain at 21° C to 32° C (70° F to 90° F) for 3 hours and then measure the average radial distance on the filter paper from the edge of the area originally "wetted" with the liquid to the farthest extent of the absorption of the liquid.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 4491
January 24, 1986

SPOT TEST (THINNERS AND SOLVENTS)

1. SCOPE

1.1 This procedure is an indicative test for chromatic impurities and heavy ends in solvents, both aromatic and aliphatic.

2. APPARATUS

2.1 Filter paper. No. 2 Whatman or equal-12.5 cm in diameter.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Transfer five drops of the sample by means of a small pipette or burette to the center of a No. 2 Whatman or equal filter paper, 12.5 cm in diameter, supported on a 7-cm crystallizing dish and allow the liquid to evaporate at 21° C to 32° C (69.8° F to 89.6° F), away from direct sunlight and strong drafts. Examine the paper visually for the presence of any stain or residual oiliness after 30 minutes or other specified period.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 4493
January 24, 1986

SAG RESISTANCE (BAKER METHOD)

1. SCOPE

1.1 This method provides a procedure for determining the sagging properties of a surface coating.

2. APPARATUS

2.1 Sag liner (see figure 1).

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of test surface. The test surface may be any vertical surface which is being coated and about which there is question of sag resistance (see 5.1).

4.2 Conditions for test.

4.2.1 The area of test shall have the test material applied at the rate specified in the product specification.

4.2.2 The test must be run within two minutes after the product under test is applied, unless otherwise specified.

4.3 Performance of test.

4.3.1 Draw two sets of horizontal lines, each set of which must be continuous for at least 8 inches.

4.3.2 The sag liner shall be held and moved so that contact points fall on a vertical line at all times.

4.3.3 The product specification shall state the time period between drawing the lines and the evaluation of the sagging characteristics.

4.4 Evaluation of results.

4.4.1 The sagging characteristics shall be rated as follows (see 5.2).

4.4.1.1 No sag. Rate as "no sag" those films which reveal no evidence whatsoever of paint movement during the 16-hour aging period for either sag line. The "sag-lines" shall show no evidence of paint sag nor shall there be any signs of paint movement along the paint edges.

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4.4.1.2 Very slight sag. Rate as "very slight sag" those films where the paint runs part way across either sag line, but in both cases fails to contact the other side.

4.4.1.3 Slight sag (unsatisfactory). Rate as "slight sag" those films where the paint runs completely across the narrower sag line at some point to become continuous with the lower side, but only part way across in the case of the border sag line.

4.4.1.4 Sag (failure). Rate as "sag or failure" those films where some portion of the paint in the case of both sag lines is found to run down and across the sag line to touch and become continuous with the other side. A serious sag condition is manifested by a complete running of the paint across both sag lines, obliterating them completely.

5. NOTES

5.1 If the surface being coated is very irregular, a coat of the material under test may be applied to a smooth surface which is then placed in a vertical position.

5.2 When this test is to be run "on the job" there will be considerable variation of temperature. Any time there is disagreement as to the sagging characteristics, a referee test using method 4494 should be run.

6. PRECISION

6.1 No data.

METHOD 4493
January 24, 1986

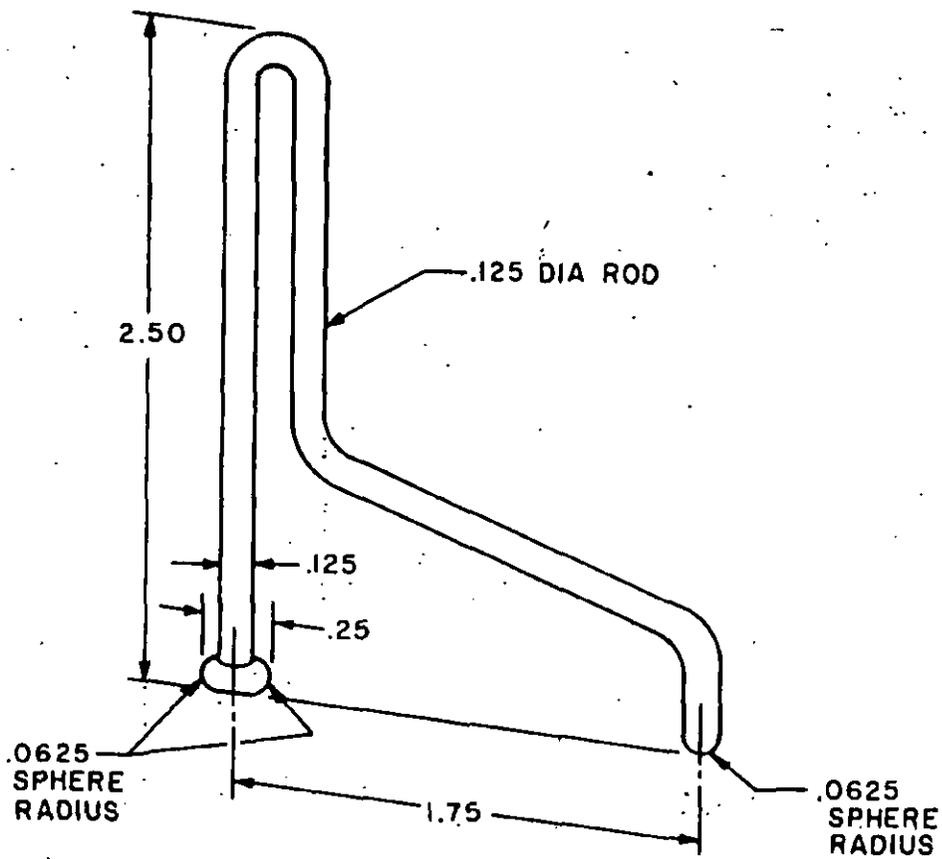


FIGURE 1. Sag lines dimensions.

X-4471

FED. TEST METHOD STD. NO. 141C

METHOD 4494.1
January 24, 1986

SAG TEST (MULTINOTCH BLADE)

1. SCOPE

1.1 This method provides a procedure for determining the sag characteristics of surface coatings. It is intended that this method be used primarily in the laboratory since the physical performance of this test does not lend itself to normal "on the job" conditions.

1.2 This method is intended for use as a referee test in cases of dispute with reference to sagging properties of a coating.

2. APPARATUS

2.1 Multinotch applicator.

2.1.1 An applicator similar to figure 1 except that it shall be designed to lay down at least eight strips of coating of graduated thickness. The space between strips shall be 1/16 inch apart.

2.1.2 The applicator must have a notch of 3 mils or less for minimum clearance and 8 mils more for maximum clearance.

2.1.3 For the clearance interval between 3 mils and 8 mils, inclusive, the notches shall have 1 mil difference. The clearance shall be arranged progressively from the shallowest to the deepest.

2.1.4 It is not necessary for the notches between 3 mils and 8 mils clearance to be on the same side of the applicator; however, the "break" clearance shall be repeated on both series.

2.2 Test surface. A test chart which is smooth surfaced and lacquered. It shall have both black and white areas.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Application of film.

4.1.1 Sufficient coating will be used in the drawdown to give approximately 1 fluid ounce in excess of that required for the drawdown.

4.1.2 The drawdown shall be made in one smooth continuous movement. The drawdown shall be straight with no waves. It is best if some test guide is used.

FED. TEST METHOD STD. NO. 141C

METHOD 4494.1
January 24, 1986

4.2 Performance of test.

4.2.1 The completed drawdown shall immediately be placed in a vertical position, with the stripes horizontal, and the thinnest stripe at the top.

4.2.2 The coating shall be left in this position until it is dry.

4.2.3 The temperature at which the test shall be performed is $23^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

4.3 Evaluation of results. The strip of paint which sags sufficiently to cross into the next thicker strip of paint below it shall be considered the strip thickness at which failure occurs.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 4494.1
January 24, 1986

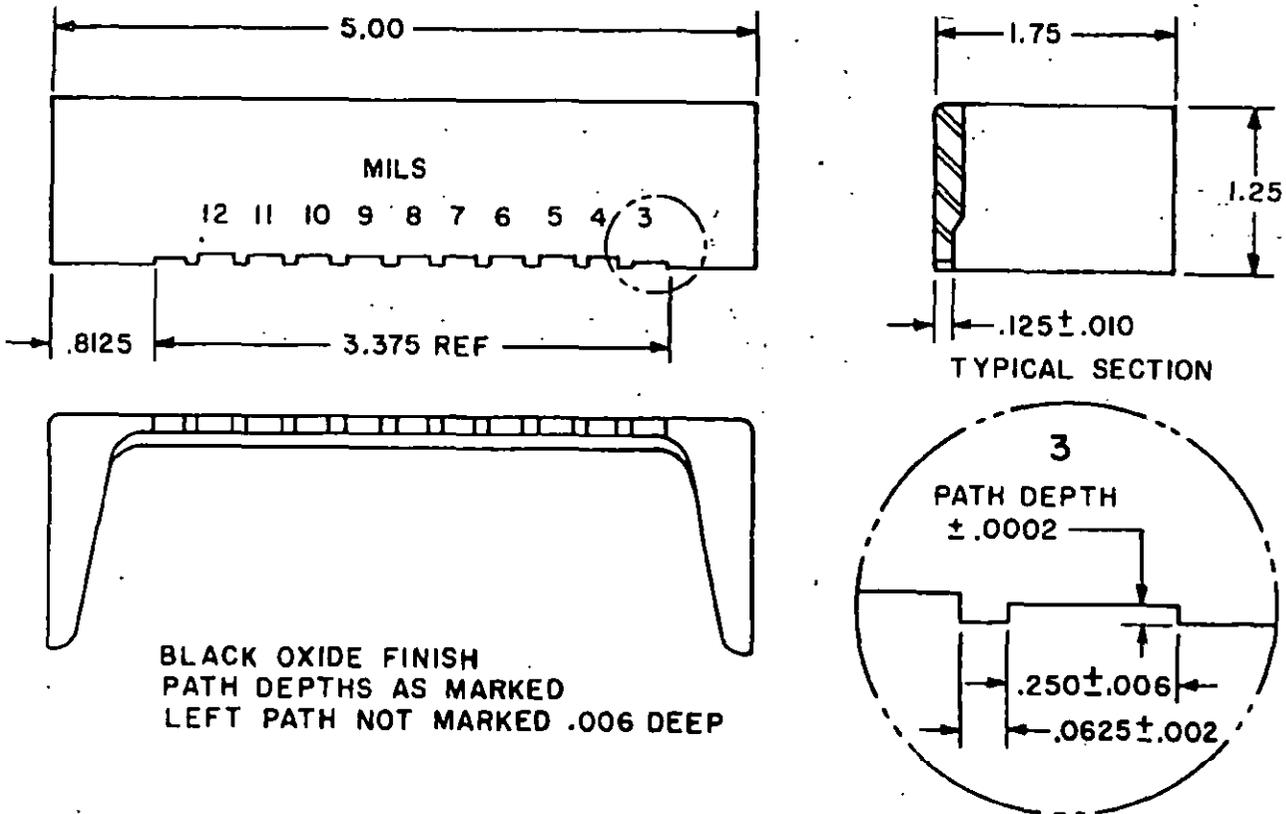


FIGURE 1. Engineering diagram of the anti-sag meter.

X-4472

FED. TEST METHOD STD. NO. 141C

METHOD 4541
January 24, 1986

WORKING PROPERTIES AND APPEARANCE OF DRIED FILM

1. SCOPE

1.1 This method covers procedures for determining the working properties of coating materials and the inspection of the dried films produced therefrom.

2. APPARATUS

2.1 None required.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Working properties. Apply the coating material by brushing, spraying, or other means as specified in the product specification to a wood, metal, or other specified kind of test panel. During and immediately after the application, observe whether the material conforms to the particular requirements for working properties given in the product specification.

4.2 Appearance of dried films. Dry the film as prescribed in the applicable specification, and visually determine whether the appearance of the film is in accordance with the requirements.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 4561.2
January 24, 1986

LIGHT FASTNESS OF PIGMENTS

1. SCOPE

1.1 This method provides a procedure for determining the resistance of pigments to fading upon exposure to sunlight. This is done by producing a pigment paste and then exposing the paste to artificial light.

1.2 The pigment paste must be prepared using the same vehicle as that required in the product specification since the fading characteristics of pigments are affected by the vehicle.

2. APPARATUS

2.1 Weighing device. An analytical balance or a torsion balance, sensitive to 1 division on the indicator per mg.

2.2 Sheets for weighing. Sheets or counterpoised balance pans of glass, metal, or other material impervious to the sample to be weighed.

2.3 Mixing slab. A lithographer's stone or a ground plate glass slab, the surface of which shall be kept sharp by lightly grinding with No. 303 optical emery or its equivalent and turpentine.

2.4 Panels. The panels used in observing color matches shall be either tin or glass, both specified as follows.

2.4.1 Tin. The finished tin plate shall conform to the requirements of QQ-T-425 for type I, grade I, class A2 (best coke), 31 gage, having a base weight per base box of 100 pounds and equivalent weight per square foot of 0.459 pound.

2.4.2 Glass. Clear plate glass not less than 3/16 inch thick.

2.5 Scraper. A French or body-glazing scraper, having a 3- or 4-inch-wide blade.

2.6 Spatula. A flexible steel (not nickel plated) spatula having a 3- or 4-inch blade.

2.7 Weathering machine.^{1/} The weathering machine shall be a self-contained electrically operated unit, having a carbon arc as the light source and adequately equipped to measure and control the current, voltage, temperature of

^{1/} Weathering machines meeting these requirements are manufactured by the Atlas Electrical Devices Co., 4114 North Ravenswood Avenue, Chicago, IL.

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test specimens, water spray, and operating schedule or cycle. The light source shall operate on alternating current at 50 volts ± 2 percent and 60 amperes ± 20 percent at the arc.

2.8 Carbons.^{2/} The carbons shall be copper plated and cored with salts of metals to produce radiated energy, after passing through a Correx D glass filter, approximately that of midday June sunlight in intensity and spectral distribution.

2.8.1 Upper carbons. The upper carbons of each pair shall be 7/8 inch in diameter and 12 inches long.

2.8.2 Lower carbons. The lower carbons of each pair shall be 1/2 inch in diameter and 12 inches long.

2.9 Light filters. The light filters shall be made of Correx D glass. They shall be replaced after 2,000 hours of use or when pronounced discoloration or milkiness develops, whichever occurs first (see 5.1).

2.10 Black panel thermometer unit. The thermometer shall be of the bimetallic type and have its stem fastened to a stainless steel panel. The panel and stem shall be finished with two coats of special light resistant black baking enamel. The thermometer and panel shall be mounted on a suitable frame so that the assembled unit will be held in the same position and at the same distance from the arc as the specimens.

2.11 Test panels.

2.11.1 Size. The panels shall be approximately 3 by 5 inches in size unless otherwise specified. All burrs and rough edges shall be removed with fine sandpaper or emery cloth. The panels shall be buffed lightly with fine steel wool.

2.11.2 Cleaning. The panels shall be cleaned by immersing them in either of the solvent mixtures prescribed in 3.1 and scrubbing lightly with a soft bristle brush or pad of cheesecloth. Dry the panels with a clean lintless cloth and use immediately or store in a desiccator over a suitable desiccant until needed.

3. REAGENTS

3.1 Solvent mixture. Prepare a solvent mixture of equal volumes of petroleum naphtha, TT-N-95, or petroleum spirits, TT-T-291, grade 1, and xylene, ASTM D 845

^{2/} Carbons commercially known as No. 22 and No. 13 National Sunshine Carbons manufactured by National Carbon Co., Inc., Cleveland, OH, meet these requirements.

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or D 846, grade B, or of three volumes of petroleum naphtha, TT-N-95 or petroleum spirits, TT-T-291, grade 1, and one volume of ethylene glycol monoethyl ether, TT-E-781.

4. PROCEDURE

4.1 Preparation of test specimens.

4.1.1 Prepare the test pastes of the sample pigments and the reference pigments as prescribed in ASTM D 387, section 6.

4.1.2 Apply a suitable portion of each paste in juxtaposition on each of two tin (see 2.4.1) or on each of two glass (see 2.4.2) panels. Make each of the four daubs of paste about 1 inch in width and 2 inches in length and lightly draw straight edge or French scraper (3 or 4 inches long) over the pastes to present each pair of daubs on an even plane.

4.2 Exposure of the test panels.

4.2.1 Expose one test panel to the light of a carbon arc as prescribed in ASTM D 822 or G 23 except that the machines shall be operated without water spray. Unless otherwise specified, expose the test specimens for 168 hours.

4.3 Observation of results.

4.3.1 Examine, visually, by light substantially equal to that from a lightly overcast northern sky. Compare the color of the exposed sample paste with that of the unexposed sample paste, and similarly compare the colors of the exposed and unexposed reference pastes. Record whether the color change undergone by the sample is greater or not greater than that undergone by the reference paste.

5. NOTES

5.1 The uniformity of operation of the light source should be checked periodically. Light Sensitive Paper No. 1589 obtainable from the National Bureau of Standards is recommended for this purpose. Directions for its use are given in National Bureau of Standards Letter Circular LC 1016.

6. PRECISION

6.1 No data.

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METHOD 4571.2
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BLEEDING OF PIGMENTS

1. SCOPE

1.1 This method covers procedures for determining bleeding of pigments in both cellulose base and oil base vehicles.

2. APPARATUS

- 2.1 Smooth white glass panel.
- 2.2 A sharp-edged steel spatula.
- 2.3 A pure bristle brush.

3. REAGENTS

- 3.1 Dibutyl phthalate. Reagent grade.
- 3.2 Raw linseed oil. Conforming to TT-L-215.
- 3.3 Spar varnish. Conforming to TT-V-121.
- 3.4 Naphthenate driers.

4. PROCEDURE

4.1 Procedure for dry pigments to be used in cellulose ester lacquers.

4.1.1 Mix a suitable amount of the pigment to be tested with a minimum volume of dibutyl phthalate to give a thick paste when rubbed up with a sharp-edged steel spatula on a glass plate. Stir this paste into a clear cellulose ester lacquer of a composition agreed upon by the buyer and the seller and apply the paint evenly by brushing on a clean, smooth, dry metal panel. Apply the paint to only a part of the panel, leaving a margin of at least 2.5 cm (1 inch) of unpainted metal around the painted portion. Allow the panel to dry for 30 minutes in a well-ventilated room at room temperature (70° to 90° F). Flow successive coats of white lacquer of a composition agreed upon by the buyer and the seller over the entire surface of the panel until complete hiding is obtained, allowing the panel to dry for 20 minutes in a well-ventilated room at room temperature (70° to 90° F) after applying each coat. Do not apply any white lacquer after the application of the particular coat that causes complete hiding. Let the completed panel dry for 24 hours and note the color of the two sections of the panel. If the color of the section containing the pigment under test does not match that of the adjoining area containing only the white lacquer, report the pigment as "bleeding."

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4.2 Procedure for dry pigments to be used in oil or oleoresinous paints and enamels.

4.2.1 Mix a suitable amount of the material to be tested with a minimum volume of a mixture of the following:

Raw linseed oil	5 parts by volume.
Spar varnish	4 parts by volume.
Naphthenate driers	As required.

4.2.2 Rub up the sample prepared in accordance with 4.2.1 with a sharp-edged steel spatula on a glass plate to obtain a uniformly smooth, stiff paste. Thin with turpentine to give a paint of brushing consistency, and apply evenly by brushing on a clean, smooth, dry metal panel. Apply the paint to only a part of the panel, leaving a margin of at least 2.5 cm (1 inch) of the unpainted metal around the painted portion. Allow the panel to dry for 18 hours in a well-ventilated room at room temperature (70° to 90° F). Brush a coat of Damar varnish (Damar resin cut in turpentine) over the entire surface of the panel. Report any color, other than that of the Damar varnish on the unpainted portion of the panel, as "bleeding".

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C
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SECTION 5000

CHEMICAL TESTS OF COATING MATERIALS AND THEIR INGREDIENTS

METHOD 5021
January 24, 1986

QUALITATIVE TEST FOR ORGANIC COLORS OR LAKES

1. SCOPE

1.1 This method covers a quick qualitative test procedure to establish the presence of organic colors or lakes in pigments.

2. APPARATUS

2.1 Beaker. A borosilicate glass beaker with a minimum capacity of 50 ml.

2.2 Weighing device. An ordinary laboratory scale or balance capable of weighing to the nearest 0.1 g.

2.3 Graduate cylinder. A 25 or 50 ml borosilicate glass cylinder calibrated TD at 20 degrees with 0.5 ml subdivisions and ± 0.5 ml accuracy.

2.4 Hotplate. An electric hotplate or steam bath.

3. REAGENTS

3.1 Alcohol. Ninety-five percent ethyl alcohol.

3.2 Chloroform. Chloroform, U.S.P. grade.

4. PROCEDURE

4.1 Place about 1 g of the pigment as received or extracted (see 5.1) from the coating material in a beaker and add approximately 25 ml of 95 percent ethyl alcohol. Heat (do not boil) on a steam bath or electric stove and observe whether the alcohol is colored. Repeat the operation with chloroform and distilled water. Development of color in any of these liquids is indicative of the presence of organic colors or lakes. Certain organic colors are insoluble in these liquids and where their presence is suspected, other methods^{1/} should be employed for their detection.

5. NOTES

5.1 Some organic colors may be completely removed by the solvent mixture during the extraction. Any abnormal coloration occurring in the extracted

^{1/} Suitable methods will be found in the following publications: "Analysis of Dyestuffs," Third Edition, by A. G. Green, published by Charles Griffen & Co., Ltd., London, England (1920) and "The Chemistry and Physics of Organic Pigments," by L.S. Pratt, published by John Wiley & Sons, Inc., New York, NY (1947).

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vehicle should be noted and this information used in conjunction with other appropriate tests to determine the presence of organic colors in the coating material.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 5041
January 24, 1986

QUALITATIVE TEST FOR UNSAPONIFIABLE MATTER IN FATTY ACIDS

1. SCOPE

1.1 This method describes a qualitative procedure for the detection of unsaponifiable matter in fatty acids.

2. APPARATUS

2.1 Weighing device. An ordinary laboratory scale or balance capable of weighing to the nearest 0.1 g.

2.2 Volumetric flask. A borosilicate glass volumetric flask calibrated to contain one liter at 20 degrees with an accuracy of ± 0.3 ml. This flask should have a T stopper.

2.3 Filter paper. Whatman No. 2 filter paper or equal of a diameter as determined by the size of the funnel used.

2.4 Funnel. a 60 degrees, short stem, chemical filtering funnel of a chemical resistant material, such as borosilicate glass.

2.5 Storage container. A storage bottle made of alkali resistant glass, and tightly sealed with a T stopper; capacity of about 1 liter.

2.6 Beaker. A borosilicate glass beaker with a minimum capacity of 50 ml.

2.7 Graduate cylinder. A 25 or 50 ml borosilicate glass cylinder calibrated to deliver at 20 degrees with 0.5 ml subdivisions and a ± 0.5 ml accuracy.

2.8 Hotplate. An ordinary laboratory electric hotplate.

3. REAGENTS

3.1 Alcoholic sodium hydroxide solution. Dissolve A.C.S. reagent grade sodium hydroxide in 95 percent ethyl alcohol in the proportion of about 25 g per liter. Let stand overnight and filter off the precipitate of carbonates. Store the solution in a glass stoppered alkali resistant bottle.

3.2 Distilled water.

4. PROCEDURE

4.1 Place 10 drops of the fatty acids (prepared according to method 5051) in a 50-ml beaker, and add 5 ml of the alcoholic sodium hydroxide solution. Bring the contents of the beaker to a boil on a hotplate to insure solution of the

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fatty acids. Remove the beaker and add 40 ml of distilled water. Thoroughly mix the contents of the beaker with a stirring rod. A permanent cloudiness indicates the presence of unsaponifiable matter.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 5051.1
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PREPARATION OF PAINT-OIL FATTY ACIDS

1. SCOPE

1.1 This method covers a procedure for the preparation of fatty acids directly from the materials rather than from the extracted vehicles.

2. APPARATUS

2.1 Weighing device. An ordinary laboratory scale or balance capable of weighing to the nearest 0.1 g.

2.2 Graduate cylinder. A borosilicate glass cylinder graduated to deliver at 20 degrees 100 ml with 1 ml subdivisions and a ± 1 ml accuracy.

2.3 Casserole. A glazed porcelain casserole with a capacity of approximately 250 ml.

2.4 Steam bath. A container suitable for use as a steam bath and a steam source.

2.5 Separatory funnel. A Squibb or cylindrical separatory funnel with a T stopcock and stopper and a capacity of approximately 250 ml.

2.6 Filter paper. Whatman No. 2 filter paper or equal of a diameter as determined by the size of funnel used.

2.7 Funnel. A 60 degrees, short stem, chemical filtering funnel of a chemical resistant material such as borosilicate glass.

2.8 Erlenmeyer flask. A borosilicate Erlenmeyer flask with a capacity of 100 ml.

2.9 Hotplate. An ordinary laboratory electric hotplate.

2.10 Drying tube. A gas drying tube with inlet and outlet fittings filled with the specified desiccant.

2.11 Storage container. A storage bottle made of alkali-resistant glass, tightly sealed with a rubber stopper, and having a capacity of 450 ml minimum.

2.12 Thermometer. A partial immersion thermometer covering the range 50° C to 80° C in 1 degree divisions with a maximum scale error of 1 degree.

3. REAGENTS

3.1 Sodium hydroxide solution. Dissolve 100 g of A.C.S. reagent grade sodium hydroxide in freshly distilled water and dilute to 300 ml. Store in a glass bottle with rubber stopper.

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3.2 Alcohol. Ninety-five percent ethyl alcohol.

3.3 Ether. A.C.S. reagent grade ethyl ether.

3.4 Sodium sulfate. A.C.S. reagent grade anhydrous sodium sulfate.

3.5 Sulfuric acid (sp. gr. 1.2). Mix 100 ml of A.C.S. reagent grade sulfuric acid with 460 ml of freshly distilled water.

3.6 Carbon dioxide. A source of carbon dioxide gas.

4. PROCEDURE

4.1 To about 25 g of the material under test in a porcelain casserole, add 15 ml of the sodium hydroxide solution and 75 ml of the alcohol, mix and heat uncovered on a steam bath with frequent stirring until saponification is complete (about 1 hour). Add 100 ml of distilled water and boil to remove all volatile thinners (nearly to dryness). Acidify with the sulfuric acid adding 8 to 10 ml in excess. Continue heating and stirring until the fatty acids have separated. Decant the fatty acids and other liquids into a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer and any insoluble or precipitated matter. Wash the fatty acids twice with water; then add 50 ml of water and 50 ml of ether. Dissolve the fatty acids in the ether, using a gentle whirling motion so as not to form an emulsion. Draw off the aqueous layer and wash the ether layer with 10 to 20 ml portions of water until the wash water no longer gives a test for acid. Then draw off the water layer completely.

4.2 Transfer the ether solution to a dry flask, add 20 to 30 g of anhydrous sodium sulfate. Stopper the flask and let stand at 21° C to 32° C (70° F to 90° F) with occasional gentle shaking until the water is completely removed and the ether solution is perfectly clear. Decant this clear solution through a dry filter paper into a dry 100-ml Erlenmeyer flask. Pass a current of dry carbon dioxide (see 5.1) into a mouth of the Erlenmeyer flask and heat at a temperature below 75° C on a hotplate until the ether is entirely driven off (see 5.2). The fatty acids prepared as indicated should be kept in a stoppered flask and examined at once (see 5.3).

5. NOTES

5.1 The current of carbon dioxide should be as rapid as possible without causing condensation of moisture inside the flask.

5.2 It is important to follow all of the details, since ether generally contains alcohol and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solutions and subsequent drying with anhydrous sodium

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sulfate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat.

5.3 The method of preparing the fatty acids directly from the materials, rather than from the extracted vehicle, is based upon past experience in some times obtaining too low results by the latter method. Occasionally, trouble is experienced in saponifying the entire material due to interference of pigment. In such an instance it is permissible to use a portion of the vehicle that has been separated from the pigment in a supercentrifuge (ASTM D 2698) or to use the extracted vehicle obtained by method 4021.

6. PRECISION

6.1 No data.

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METHOD 5132
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CHLORINATED COMPOUNDS (PRESENCE OF)

1. SCOPE

1.1 This method is used for determining the presence of chlorinated compounds in products used in the paint industry. In most instances, chlorinated compounds should not be present.

2. APPARATUS

2.1 Copper wire, 18 to 20 gage.

2.2 Bunsen burner.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Form a small loop of copper wire and heat in a small Bunsen burner until it no longer colors the flame. Allow the loop to cool and then dip it in a sample of the solvent obtained by vacuum distillation or the clear liquid under test. Immediately place in the outer part of the flame and when the first luminous flame disappears, examine for the typical green coloration due to chlorinated compounds.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 5133
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TEST FOR PRESENCE OF METHANOL

1. SCOPE

1.1 This method determines if methanol is present in a lacquer or other type solvent.

2. APPARATUS

2.1 Flask. Distilling, 10-ml.

2.2 Berl saddle.

2.3 Condenser. Water cooled.

2.4 Separatory funnel. Fifty ml.

3. REAGENTS

3.1 Phosphoric acid. Fifty percent.

3.2 Saturated aqueous potassium permanganate solution.

3.3 Sodium sulfite.

3.4 Aqueous resorcinol. A 0.5 percent solution.

3.5 Concentrated sulfuric acid.

4. PROCEDURE

4.1 Place 5 ml of the lacquer solvent or other clear liquid obtained by vacuum distillation in a small 10-ml distilling flask containing a Berl saddle. Stopper the flask and attach a short water-cooled condenser with delivery tube which extends under 4 ml of water contained in a small test tube. Place the distilling flask in a water bath at 85° C - 90° C (185° F - 194° F) and collect the distillate for 15 minutes. Detach the condenser and rinse with 2 ml of water. If two layers of solvent are present in the collected distillate, separate the lower layer by means of a separatory funnel and discard the upper layer. Divide the aqueous layer equally between two small test tubes and treat as "sample" and "blank". To the "sample" add 3 drops of 50 percent phosphoric acid and 1 drop of saturated aqueous potassium permanganate. After one minute decolorize the excess permanganate by adding a little sodium sulfite. Add 2 drops of 0.5 percent aqueous resorcinol and layer the solution onto 1 ml of concentrated sulfuric acid contained in a separate test tube. If methanol is present in the original distillate, a red ring will form at the junction of the two layers and increase in intensity on standing. The entire solution will

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appear red if agitated. Treat the "blank" with 3 drops of 50 percent phosphoric acid and 2 drops of the resorcinol solution and also layer onto 1 ml sulfuric acid in a separate test tube. No red color should form.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 5141.1
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QUALITATIVE TESTS FOR PHENOLIC RESINS
(Including Bisphenol Type Epoxies)

1. SCOPE

1.1 This method covers procedures for identifying three types of phenolic resins. The tests are specific for the p-phenyl-phenol-formaldehyde resins and epoxy resins of the bisphenol type. The test for phenol-formaldehyde resins in general will also be positive for the p-phenyl-phenolic resin but negative for epoxy resins.

2. APPARATUS

2.1 No special apparatus required.

3. REAGENTS

3.1 Ferric chloride reagent. Prepare a 2 percent solution of ferric chloride hexahydrate in pyridine.

3.2 Alcoholic potassium hydroxide. Prepare a 0.5 N solution of potassium hydroxide by dissolving 3.3 g of pellets in 100 ml of 95 percent ethyl alcohol.

3.3 Sulfuric acid, 3.6 N. Dilute one volume of concentrated sulfuric acid with nine volumes of distilled water.

3.4 Sodium nitrite solution, 20 percent. Prepare a fresh solution of sodium nitrite by dissolving 2 grams in 10 ml distilled water.

3.5 Sulfuric acid, concentrated.

3.6 Formaldehyde, 37-40 percent.

3.7 Methyl isobutyl ketone. A.C.S. reagent grade.

3.8 Benzene. A.C.S. reagent grade.

3.9 Ethyl alcohol. A.C.S. reagent grade.

4. PROCEDURE

4.1 Detection of para-phenyl-phenol-formaldehyde resins.

4.1.1 Procedure A. Dissolve approximately one ml of the resin solution to be tested in 10 ml of methyl isobutyl ketone in a test tube. Add ten drops of the 2 percent ferric chloride in pyridine reagent and mix. Now add alcoholic potassium hydroxide dropwise, examining the sample after the addition of each drop. If phenyl-phenol-formaldehyde is present, a green color will form by the time ten drops of alkali have been added.

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4.1.2 Procedure B. Dissolve a few drops of resin solution in a few milliliters of any suitable low-boiling solvent in a beaker or flask and dry with a current of air in a warm water bath or in an oven. Cool and add a few milliliters of concentrated sulfuric acid. If appreciable quantities of p-phenyl-phenol-formaldehyde resin are present, an intense green color forms.

4.2 Detection of phenol-formaldehyde resins (including p-phenyl-phenol-formaldehyde).

4.2.1 Procedure. Pour 40 ml of A.C.S. grade benzene in a 50-ml glass-stoppered graduated cylinder. Add exactly 4 drops of liquid resin or weigh out 50 mg of solid resin and mix to dissolve. If the sample is insoluble in benzene, dissolve it first in 20 ml butyl acetate and add 20 ml benzene. Add 10 ml of the 3.6 N sulfuric acid. Add exactly 2 drops of the sodium nitrate solution, stopper and shake vigorously for at least 10 seconds. Allow to stand undisturbed for exactly 5 minutes and examine the benzene layer. A strong, yellow color indicates phenol-formaldehyde resins. Color that develops after 5 minutes has no significance. If the sample being tested is strongly colored initially, run a blank for comparison, omitting only the sodium nitrite treatment.

4.3 Detection of epoxy resins.

4.3.1 Procedure. Place 2 drops of the resin solution, or an equivalent amount of dry resin, in a test tube. Add about 10 drops of benzene and 10 drops of ethyl alcohol, mix and dry in an oven at 105° C. After cooling, add one ml of concentrated sulfuric acid and warm to about 60° C in a water bath for 10 minutes. Cool and add 2 drops of formaldehyde and allow the sample to stand for a few minutes. Dilute with 10 ml of water added rapidly from a graduate. A green or blue color will form almost immediately if epoxy resins of the bisphenol type are present.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 5151
January 24, 1986

ACETONE EXTRACT

1. SCOPE

1.1 This method describes a procedure for the determination of acetone extract in materials.

2. APPARATUS

2.1 Weighing device. An analytical or torsion balance having a sensitivity of 1 mg at full load. The weights should be class P or better.

2.2 Volumetric flask. A 200 ml borosilicate glass volumetric flask calibrated to contain at 20 degrees with a ± 0.10 ml accuracy.

2.3 Pipette. A 50 ml borosilicate glass transfer pipette calibrated to deliver at 20 degrees with a ± 0.05 ml accuracy.

2.4 Water bath. A container of suitable size and shape to be used as a hot water bath and an electric hotplate.

2.5 Filter paper. Whatman No. 2 filter paper or equal of a size as determined by diameter of the funnel used.

2.6 Funnel. A 60°, short stem, chemical filtering funnel made of chemical resistant material such as a borosilicate glass.

2.7 Evaporating dish. A porcelain evaporating dish with a glazed inside, and approximate capacity of 200 ml and diameter at top of 110 mm.

2.8 Oven. A gravity-convection oven ventilated such that there are a minimum of 10 air changes per hour and thermostatically controlled at the specified temperature $\pm 2^\circ$ C.

2.9 Desiccator. A desiccator of suitable dimensions to permit storage of several of the described evaporating dishes; it should have a sample plate and ground rims between base and lid.

3. REAGENT

3.1 Acetone. A.C.S. reagent grade.

4. PROCEDURE

4.1 Weigh to the nearest mg about 2 g of the sample, transfer to a 200-ml volumetric flask and add 50 ml of the acetone. Heat to boiling, cool, fill to the mark with acetone, and thoroughly mix by shaking. Let settle, draw off

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about 125 ml of the supernatant liquid and filter through a dry paper. Discard the first 10 ml and transfer 100 ml of the clear filtrate to a weighed dish, evaporate the acetone at a temperature not above 75° C, heat the dish for 15 minutes in an oven at 105° C \pm 2° C (221° F \pm 3.6° F), cool in a desiccator, weigh, and compute percentage of acetone extract.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 5172.1
January 24, 1986

ESTER AND KETONE CONTENT (DOPES AND LACQUERS)

1. SCOPE

1.1 This method covers procedures for the separation of the volatile from the nonvolatile portion of dopes and lacquers and the determination of the percentages of esters and ketones in the recovered volatile portion.

2. APPARATUS

2.1 Distillation apparatus. A distillation apparatus consisting of a 2-liter distillation flask with vapor tube at 75 degree angle to neck, two hole stopper fitted with thermometer and vacuum bubbling tube, water cooled condenser of the West or Liebig type with a 400 mm jacket, adapter suitable for connecting to vacuum receiver, hotplate and oil bath, ice water bath, manometer, vacuum pump, and necessary clamps, stands, etc.

2.2 Weighing device. An analytical balance having a sensitivity of 0.1 mg at full load, case enclosed, and of the rider, chain or equally accurate type. The weights should be NBS class S-1 or calibrated against S-1 weights.

2.3 Volumetric flasks. A 100 ml (accuracy of ± 0.08 ml) and a 1 liter (accuracy of ± 0.3 ml) borosilicate glass volumetric flask fitted with a T_s stopper and calibrated to contain above volumes at 20° C.

2.4 Burette. A 50 ml borosilicate glass burette, with 0.1 ml subdivision, stopcock controlled, calibrated to deliver at 20° with an accuracy of ± 0.05 ml.

2.5 Reflux apparatus. A 200 ml Erlenmeyer flask and reflux condenser, made of alkali resistant glass and having a T_s connection, steam bath and steam source, and necessary clamps, stands, etc.

2.6 Pipette. A 10 ml borosilicate glass transfer pipette calibrated to deliver at 20 degrees with an accuracy of ± 0.02 ml.

3. REAGENTS

3.1 Tricresyl phosphate. Tricresyl phosphate, technical grade.

3.2 0.5 N alcoholic potassium hydroxide solution. Dissolve 33 g of KOH, A.C.S. reagent grade, in 95 percent ethyl alcohol and dilute to 1 liter with alcohol. If insoluble matter is present, filter.

3.3 Phenolphthalein indicator. Dissolve 1 g of A.C.S. standard grade phenolphthalein in 100 ml of 95 percent ethyl alcohol.

3.4 0.5 N hydrochloric acid. Dilute 41.7 ml of 12 N A.C.S. reagent grade HCl to 1 liter with distilled water.

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3.5 Indicator solution for ketone determination. Prepare a liter of indicator solution by mixing 20 ml of pyridine with 10 ml of 0.04 percent bromphenol blue (in 95 percent ethanol) and 3 ml of 0.1 percent aqueous congo red. Dilute to one liter with 95 percent ethanol (see 5.3).

3.6 Hydroxylamine hydrochloride solution. Prepare by dissolving 3.5 g of hydroxylamine hydrochloride in 16 ml of water and diluting to 100 ml with 95 percent ethanol (see 5.1).

3.7 Standard methanolic sodium hydroxide solution. Dissolve 20 g of sodium hydroxide in one liter of absolute methanol by refluxing. If solution is cloudy, filter through fritted glass just before using. Standardize this solution with standard acid, using phenolphthalein as indicator.

4. PROCEDURE

4.1 Separation of volatile portion. Weigh to the nearest 0.1 g between 300 and 400 g of the sample under test into a 2-liter round bottom flask. Add an amount of tricresyl phosphate equal to 1-1/2 to 2 times the weight of nonvolatile material in the sample. Fit a two-hole stopper into the neck of the flask to accommodate the thermometer and a glass tube extending to the bottom of the flask through which air may be bubbled, as an aid in the distillation. Place the flask in an oil bath, and attach a suitably cooled condenser. Keep the receiving flask in an ice water bath during the entire distillation. Heat the oil bath in such a manner that the temperature of the contents of the flask will be slowly raised to the initial boiling point of the volatile constituent. Continue the distillation at a rate of 4 to 5 ml per minute, while bubbling air slowly through the contents of the flask, until a vapor temperature of 100° C (230° F) has been reached. At this point quickly replace the receiver with a second tared flask and connect to a vacuum pump through a manometer. Start the pump and slowly reduce the pressure in the distillation flask. After distillation has practically stopped at the reduced pressure, increase the heat gradually until at least 95 percent of the total volatile portion has been recovered. This point is generally reached at 120° C (248° F) or lower in the evacuated system. When temperatures above 120° C are necessary because of the presence of high boiling thinners, special care must be taken to avoid decomposition of the cellulosic derivations (see caution note). Stopper both receiving flasks immediately upon their disconnection. Weigh the flasks and their contents and calculate the percentage by weight of the total distillate recovered. Combine the distillates and determine the ester and ketone contents of the mixture.

Caution: When nitrocellulose begins to decompose, brown nitrous oxide fumes are formed in the flask and the lacquer residue will begin to effervesce. If this condition develops, remove the distillation flask immediately to a hood and thoroughly ventilate the room. Avoid breathing the fumes, since they are quite poisonous.

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4.2 Determination of ester contents (see 5.1).

4.2.1 Saponification number. Weigh to the nearest 0.1 mg between 1 and 2 g of the mixed distillate in a suitable weighing device. (Alkali-free glass ampoule, weighing bottle, Nicol tube pipette or hypodermic syringe of appropriate size may be used at the convenience of the analyst. However, all necessary precautions peculiar to the particular weighing device used must be taken to insure against any loss by volatilization of the sample during the weighing and introduction of the sample into the solution within the flask.) Place one and one-half times the theoretical quantity of 0.5 N alcoholic potassium hydroxide required for complete saponification in a 200-ml Erlenmeyer flask. Introduce the weighed sample or container and sample as the case may be into the flask and attach a reflux condenser to the flask. If the sample is in a sealed container, such as an ampoule, break the container before attaching the condenser. Two blanks of the equivalent amount of alcoholic potassium hydroxide shall be run with the sample and the blanks shall check to within 0.1 ml. Heat the flasks on a steam bath for at least 3 hours (4 hours may be necessary in some cases). Gently swirl the contents of the flasks frequently. Cool to room temperature, wash down the condensers with a small amount of distilled water. Remove the condensers, add three drops of phenolphthalein indicator to the contents of each flask and titrate with 0.5 N hydrochloric acid. Compute the saponification number using the following equation:

$$\text{Saponification number} = \frac{(B - A) N \times 56.1}{S}$$

where:

A = Milliliters of standard acid required to titrate sample.

B = Milliliters of standard acid required to titrate blank.

N = Normality of acid.

S = Grams of sample used.

4.2.2 Acid number. Determine the acid number of the combined distillate as prescribed in ASTM D 1613.

4.2.3 Calculation. Calculate the ester content using the following equation:

$$\text{Percent of ester} = \frac{X - Y}{56.1} \times 0.116 \times 100$$

where: X = Saponification number.

Y = Acid number.

4.3 Determination of ketone content (see 5.2).

4.3.1 Weigh a 100 ml volumetric glass stoppered flask which contains from 20 to 30 ml of absolute ethanol. By means of a pipette, add exactly 10 ml of the

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volatile distillate. Stopper, mix, and reweigh to determine sample weight. Dilute to mark with absolute ethanol and mix. Pipette 25 ml of the hydroxylamine hydrochloride reagent into a glass stoppered 250-ml Erlenmeyer flask and add from pipette exactly 15 ml of the prepared sample solution. Mix and allow to stand at room temperature for one hour. Add 5 ml of absolute ethanol and 15 ml of the indicator solution. Titrate before a brightly lighted background with 0.2 N methanolic alkali from a micro burette. Agitate vigorously near the end point color change from red to yellow and add the alkali in 0.1 ml portions near the end point until no further change takes place. Record the burette reading, but stopper the flask and allow to stand 30 minutes. Retitrate to the permanent yellow-orange end point and use the total titration for the calculation.

4.3.2 Calculation. Calculate the percentage of ketone (computed as methyl isobutyl ketone) using the following equation:

$$\text{Percent ketone} = \frac{(V_1 - V_2) \times N \times 0.1002 \times 6.66 \times 100}{W \times 0.98}$$

where:

- V₁ = Milliliters of alkali for titration of sample.
- V₂ = Milliliters of alkali for titration of blank.
- N = Normality of alkali solution.
- W = Weight of sample (10 ml of volatile distillate).
- 6.66 = Aliquot correction for the 15 ml aliquot.
- 0.98 = Correction factor.

5. NOTES

5.1 Unless otherwise specified, the esters present shall be calculated as butyl acetate.

5.2 Unless otherwise specified, the ketones present shall be calculated as methyl isobutyl ketone.

5.3 The same source of supply hydroxylamine hydrochloride solution and indicator solution must be used with both the sample and blank.

6. PRECISION

6.1 No data.

METHOD 5205.2
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NITROCELLULOSE IN LACQUERS

1. SCOPE

1.1 This method covers procedures for the determination of the nitrocellulose content of the nonvolatile vehicle portion of clear and pigmented nitrocellulose lacquers.

2. APPARATUS

2.1 Flask. Twenty-five ml volumetric glass-stoppered flask.

2.2 Desiccator. Using anhydrous sodium sulfate.

2.3 Infrared spectrometer. Commercial laboratory instrument.

3. REAGENT

3.1 Acetone. A.C.S. reagent grade.

4. PROCEDURE

4.1 Separation and determination of nonvolatile vehicle content.

4.1.1 Pigmented lacquer. Separate the vehicle from the pigment as prescribed in ASTM D 2698. If the pigment is not completely removed in the first centrifuging, add a quantity (1 to 3 parts by volume) of lacquer thinner to the separated vehicle and centrifuge to obtain a clear liquid.

4.1.1.1 Determine the nonvolatile content of the separated vehicle as prescribed in method 4044.

4.1.2 Clear lacquer. Determine the nonvolatile content of the lacquer as prescribed in method 4044.

4.2 Performance of test.

4.2.1 Nitrocellulose.

4.2.1.1 Weigh 2 to 3 g of the lacquer vehicle into a weighed aluminum dish and record as W_1 . At the same time, prepare a standard by pouring a solution of nitrocellulose in acetone into an aluminum dish that is equivalent to 0.2 to 0.4 g of dry nitrocellulose (see 5.1). Thin both sample and standard with about 3 ml of acetone and allow to air dry 18 to 24 hours. Reweigh the dish containing the vehicle sample and record as W_2 . Dry the dish containing the standard nitrocellulose in an oven at 105° C for two hours but do not weigh.

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4.2.1.2 Transfer strips of the air-dried sample film and the oven-dried standard film to separate 25 ml volumetric flasks (weighed). Determine the weight of sample and standard by reweighing the flasks, recording the sample weight as W_3 and the standard weight as W_n . Calculate the weight of the nonvolatile lacquer sample as follows:

$$W_s = W_1 \cdot x \frac{W_3}{W_2} \times \frac{\text{Percent nonvolatile}}{100}$$

4.2.1.3 Dissolve both films in acetone that has been dried over anhydrous sodium sulfate and dilute to the mark with dry acetone (see 5.2). Filter the sample solution (but not the standard) through dry rapid paper into a dry 25-ml glass-stoppered flask and protect the entire sample from evaporation by covering the funnel with a watch glass and wrapping the funnel stem with cotton or foil. Place the sample and dry acetone in separate fixed cells of about 0.15 mm cell length.

4.2.1.4 Using quantitative instrument settings, and with nothing in either beam of the infrared spectrophotometer, set zero with up scale signal at $\lambda = 11.85$ microns, and set 100 percent transmission. Scan the spectrum from 11.3 to 12.45 with sample and reference cells in place, and determine the absorbance from the difference between A_{max} at 12.45 microns and A_{min} at 11.85 microns and record as A_s . Repeat this scanning, rechecking zero and 100 percent settings at 11.85 microns until good reproducibility is obtained. Replace the sample solution with standard solution, repeat the readings, recording the absorbance as A_n . Calculate the absorptivity of the standard as follows:

$$\text{Absorptivity} = a = \frac{A_n}{W_n}$$

Calculate the percent nitrocellulose in the sample as follows:

$$\text{Percent nitrocellulose} = \frac{A_s \times 100}{a \times W_s}$$

5. NOTES

5.1 A standard working curve may be prepared and used in place of the standard sample, if preferred.

5.2 The sample film may not always dissolve completely but after standing for one hour, the nitrocellulose in the film will have dissolved.

6. PRECISION

6.1 No data.

METHOD 5211.2
January 24, 1986

EXTRACTION OF RUBBER BASE PRECIPITATE (CHLORINATED RUBBER AND S-A TYPES)

1. SCOPE

1.1 This method provides a procedure for the determination of precipitate which can be obtained from rubber base paints. Two types are covered in this method, natural rubber (chlorinated) base and a styrene-acrylate copolymer base. Different procedures will be given for each.

2. APPARATUS

2.1 Crucible. Porcelain, glazed.

2.2 Centrifuge tubes. Ninety ml.

2.3 Centrifuge. Capable of 2,000 r.p.m.

2.4 Beakers. Volumetric, 100, 150, and 800 ml.

2.5 Crucible. Gooch, 25 ml.

2.6 Evaporating dish.

3. REAGENTS

3.1 Benzene. A.C.S. reagent grade.

3.2 Petroleum ether. O-E-751.

3.3 Concentrate H_2SO_4 .

4. PROCEDURE

4.1 Rubber-base precipitate, natural rubber-base (chlorinated rubber). Weigh approximately 5 g of the paint sample into a tared 150-ml beaker and dilute with 75 ml of benzene. Add this diluted sample drop by drop to 500 ml of petroleum ether in an 800-ml beaker. Stir the petroleum ether rapidly with an air-driven stirrer during the addition of the diluted sample. Continue stirring, and slowly add 100 ml of anhydrous methanol. Allow the precipitate to settle for at least 1 hour, then decant as much of the supernatant liquid as possible (ignore any slight turbidity). Transfer the precipitate to the original 150-ml beaker and dissolve the organic material with 75 ml of benzene. Repeat the precipitation step using the 500 ml of petroleum ether, followed by 100 ml of anhydrous methanol. Allow the precipitate to settle for 1 hour, then decant as above and transfer the precipitate to a tared evaporating dish. Dry the precipitate under a gentle stream of air until solvent odor is gone, and then dry in an oven at 105° C to 110° C to a constant weight. The dry solids contain the rubber-base precipitate and most of the pigment. Wet the dry solids with concentrated

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H₂SO₄; fume off the acid and organic matter under a hood using a low flame; then heat strongly to drive out all acid; and finally ignite at 900° C to a constant weight. Consider the loss in weight as rubber-base precipitate. The ignited residue will contain most of the pigment, but if the supernatant liquids contain more than a trace of turbidity, the pigment should be determined by sulfate ashing an equal portion of the same sample at 900° C.

Calculate the percentage rubber-base precipitate for type I paint as follows:

$$\text{Percent rubber base precipitate} = \frac{A \times 100}{W}$$

where:

A = loss of weight of dry solids on sulfate ashing.

W = weight of paint sample.

Percent rubber base precipitate,

$$\text{total vehicle bases} = \frac{B \times 100}{100 - \text{percent pigment}}$$

where:

B = percent rubber base (total paint).

4.2 Rubber-base precipitate, styrene-acrylate-copolymer. Weigh accurately about 2 g of the paint into a weighed 90 ml centrifuge tube. Add 25 ml of the extraction mixture "C", 1:1-toluene:acetone, specified in method 4021 and mix thoroughly with a glass stirring rod. Remove the stirring rod; wash any adhering material back into the tube with a little of the extraction mixture. Place the tube, with contents, in the container of a centrifuge and centrifuge for 10 minutes at about 2,000 r.p.m. Decant the vehicle solution portion of the tube into a 100-ml volumetric flask. Repeat the extraction two more times and bring the vehicle solution in the 100-ml volumetric flask to the 100 ml volume mark by adding benzene, and mixing thoroughly. Set the vehicle solution aside for further tests. Dry the tube and contents in an oven at 105° C; cool in a desiccator, and weigh. Record the difference in weight of the tube and the content as the weight of pigment I.

4.2.2 Prepare a 25 ml Gooch crucible with a heavy mat of pure well-washed asbestos or with 3 or 4 fiber glass disks; wash thoroughly with petroleum ether; dry in an oven at 105° C; cool in a desiccator, and weigh. Wash the prepared Gooch crucible thoroughly with benzene, and filter a 25 ml aliquot of the vehicle solution through the Gooch crucible. Wash the crucible and contents with petroleum ether. Dry the crucible and content in an oven at 105° C; cool in a desiccator, and weigh. Record the difference in weight of the crucible and the content as the weight of pigment II.

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4.2.3 Add 20 ml of the extraction mixture "D", 1:3-ethyl ether:petroleum ether, specified in method 4021 to a weighed 90-ml centrifuge tube. Add a 50-ml aliquot of the vehicle solution and mix thoroughly by whipping the side of the tube for about 30 seconds. Then add an additional 20 ml of the extraction mixture and centrifuge for 10 minutes at about 2,000 r.p.m. Add 5 ml of the extraction mixture. If a cloud forms, add more extraction mixture and recentrifuge. Repeat the extraction until no cloud is formed by the addition of 5 ml of the extraction mixture after centrifuging. Decant the supernatant liquid into a tared 250-ml beaker; evaporate to dryness on a steam bath; dry in oven at 105° C; cool in a desiccator, and weigh. Set the beaker and contents aside for further testing. Dry the contents in the centrifuge in an oven at 105° C; cool in a desiccator, and weigh. Record the difference in weight of the centrifuge tube and the contents as the weight of rubber base I.

4.2.4 Dissolve the residue in the beaker in a little benzene and transfer into a 90-ml centrifuge tube. Add 40 ml of extraction mixture "D". Repeat the extraction procedure as performed in c, and record the weight of the contents of the tube as the weight of rubber base II.

4.2.5 Calculate the percentage rubber-base precipitate for type II paint as follows:

Grams vehicle =
 (grams paint - [grams pigment I + grams pigment II x aliquot factor])

Percent rubber base =

$$\frac{\text{grams rubber base I} + \text{grams rubber base II} \times \text{aliquot factor} \times 100}{\text{grams vehicle}}$$

5. NOTES

5.1 This method should be applicable to pigments containing TiO₂, ZnO, silicates, and Fe₂O₃. Other pigments would be handled best by centrifugation or ashing at 500° C.

6. PRECISION

6.1 No data.

METHOD 5212
January 24, 1986

EXTRACTION OF RUBBER BASE PRECIPITATE (S-B TYPE)

1. SCOPE

1.1 This method provides a procedure for the determination of precipitate which can be obtained from styrene butadiene base paints.

2. APPARATUS

2.1 No special requirement.

3. REAGENTS.

3.1 Benzene.

3.2 Normal pentane.

4. PROCEDURE

4.1 Rubber base determination.

4.1.1 Weigh a 2-g sample of paint into a tared 250 ml Erlenmeyer flask containing a glass stirrer. Add 20 ml of benzene and warm on a steam bath for 1/2 hour, then allow to cool. Attach stirrer to motor driven stirring apparatus and slowly add (5 ml/min.) normal pentane (95 percent) from a buret until the resin begins to coalesce. At this critical point add normal pentane dropwise (0.5 ml/min.) until the rubber-base has precipitated or coagulated. When the solution appears to be clear, add normal pentane at 5 ml/min until a total of 200 ml of normal pentane has been introduced into the flask. Continue stirring for 30 minutes, allow to precipitate or settle and decant supernatant liquid. Redissolve the precipitate in 20 ml of benzene and repeat precipitation and decantation as above. Wash precipitate with 100 ml of normal pentane. Evaporate the residual solvent on a steam bath and dry to constant weight in an oven at 105° C to constant weight, cool and weigh. The residue in the flask consists of rubber-base precipitate plus pigment. Weigh a 2-g sample of paint into a tared 30 ml porcelain crucible, place in an oven at 105° C for 1/2 hour to evaporate the solvents, ignite at 550° C to constant weight, cool and weigh. The crucible contains total pigment and the loss on ignition is considered total vehicle. Calculate rubber-base precipitate based on the total vehicle.

5. NOTES.

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 5221.1
January 24, 1986

MATTER INSOLUBLE IN HOT SOLVENTS

1. SCOPE

1.1 This method determines the insoluble matter in dry shellac, varnish, or other products.

2. APPARATUS

2.1 A Jena glass-filter crucible having a capacity of 30 ml, and a fritted glass filter disk having a thickness of 3 mm and a porosity of 5 to 7 (average diameter of pores $35\mu\text{m}$ to $40\mu\text{m}$).

2.2 A carbon filter funnel, made of pyrex glass.

2.3 A heating coil, of 3-mm copper tubing of such size and shape that the filter crucible and the large part of the carbon tube will fit snugly within it.

2.4 Filter paper, quantitative grade, coarse, with an ash content not to exceed 0.01 percent.

3. REAGENTS

3.1 Normal Butyl alcohol.

3.2 Ethylene glycol monoethyl ether.

3.3 Ethyl alcohol, 95 percent.

3.4 Methanol.

4. PROCEDURE

4.1 Preparation of the apparatus.

4.1.1 Cut a disc of filter paper to fit inside the crucible, and place it on top of the glass filter. Introduce upon the filter paper an asbestos mat approximately 3 mm thick. Dry the crucible to constant weight at $105^{\circ} \pm 2^{\circ} \text{C}$ and cool in a desiccator. Weigh the prepared crucible and place it within the carbon filter funnel using thin rubber tubing to form an air-tight connection. Place the combined filtering unit within the heating unit which is insulated on the outside with sheet asbestos, attach to a suction flask, and pass steam through the coil.

4.2 Performance of test. Weigh into a 200-ml beaker to the nearest 0.1 mg sufficient material to contain about 5 g of nonvolatile matter, or about 5 g of dry resin. Add 75 ml of the specified solvent and bring the solution to a boil. Keep the solution boiling slowly for 5 minutes to ensure complete solution. Pour about 10 ml of boiling solvent from a wash bottle into the

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heated crucible. Apply gentle suction to the crucible, and in such a manner as to retain as much as possible of the insoluble residue in the beaker. Wash the insoluble residue successively with three 20-ml portions of the solvent, boiling the solution for about 1 minute before each filtration. Finally, transfer the residue from the beaker to the crucible with a stream of boiling solvent from a wash bottle, using a policeman when necessary. Wash down the inner walls of the crucible with boiling solvent. The total volume of the solvent used should be approximately 175 ml. Keep the crucible covered with a small watchglass at all times, except when actually transferring the solution from the beaker to the crucible, or when washing down the inside walls of the crucible. By this procedure, a higher temperature is maintained within the crucible. Allow the crucible to remain within the heating coil with the suction on for a few minutes, so as to suck the residue as dry as possible. Remove the crucible, wash the outside with boiling solvent, dry it in an oven at $105^{\circ} \pm 2^{\circ}$ C for 1 hour, cool in a desiccator, and weigh. From the weight of the residue and the weight of the sample, calculate the percentage of insoluble matter based on the nonvolatile matter in the sample taken.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 5231
January 24, 1986

WAX IN SHELLAC

1. SCOPE

1.1 This method describes a procedure for the determination of percent wax content in shellac.

2. APPARATUS

2.1 Weighing device. An analytical or torsion balance having a sensitivity of 1 mg at full load and a set of NBS class P weights or better.

2.2 Beaker. A 200 ml tall form beaker made of borosilicate glass and a watchglass suitable for use as cover.

2.3 Hot water bath. A container suitable for use as a hot water bath and heat source or a steam bath and steam source.

2.4 Büchner funnel. A porcelain Büchner filtering funnel with a diameter of 2-1/4 to 3-1/2 inches.

2.5 Oven. A gravity-convection type oven ventilated so that there is a minimum of ten air changes per hour and thermostatically controlled at the specified temperature $\pm 2^{\circ}$ C.

2.6 Extraction apparatus. A continuous extraction type apparatus of the Soxhlet, Underwriter's Lab, or Wiley-Bailey type.

3. REAGENTS

3.1 Alcohol. Specially denatured alcohol (190 proof), formula No. 1 or No. 30 of the United States Bureau of Internal Revenue.

3.2 Chloroform. A.C.S. reagent grade chloroform, which has been redistilled to remove nonvolatile matter.

3.3 Filter cel. Filter cel which has been extracted with chloroform.

3.4 Sodium carbonate solution. Dissolve 2.50 g of Na_2CO_3 , A.C.S. reagent grade, in 150 ml of hot distilled water. Make this solution up just prior to use and do not cool.

4. PROCEDURE

4.1 Weigh to the nearest mg from a stoppered flask about 10 g of the dry lac resin or a sufficient amount of the shellac varnish to contain about 10 g of nonvolatile matter into a 200-ml tall form beaker. Dissolve 2.5 ± 0.01 g of A.C.S reagent grade sodium carbonate in 150 ml of hot distilled water and add

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the solution to the beaker. Immerse the beaker in a steam or hot water bath and stir until solution of the lac resin is complete. Cover the beaker with a watch glass and allow to remain in the bath for 2 to 3 hours without agitation. Remove the beaker from the hot bath and place it in a cold water bath. Cover the bottom of a Büchner funnel (2-1/4 or 3-1/2 inches in diameter) with a disk of filter paper and pour onto it in a fairly uniform layer, 1 g of filter cel mixed with distilled water. Add 0.5 g of the filter cel to the solution and filter with the aid of suction. Transfer all the wax, which may occur as a cake or small hard particles to the funnel using a stream of distilled water from a wash bottle and a policeman if necessary. Wash the wax and filter paper to remove all soluble matter with water and finally pour over the filter paper and the precipitate a few ml of the alcohol to facilitate drying. Dry the funnel and its contents at 40° +2° C (104° +3.6° F). Carefully wrap the contents of the funnel in a clean filter paper and tie or bind with a thin copper wire. Place in a suitable continuous extraction apparatus and extract with the chloroform solution to a clean weighed beaker or flask. Evaporate most of the chloroform on a steam bath and then dry the wax to within 10 mg of constant weight in an oven at 105° +2° C (221° +3.6° F).

4.2 Calculation.

4.2.1 Calculate the percentage of wax using one of the following equations as required:

4.2.1.1 For lac resin.

$$\text{Percent wax} = \frac{W}{S} \times 100$$

4.2.1.2 For shellac varnish.

$$\text{Percent wax} = \frac{W}{SXD} \times 100$$

where: W = Grams of wax obtained.
 S = Grams of sample used.
 D = Percentage of nonvolatile matter in shellac varnish, expressed as a decimal fraction.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 5264
January 24, 1986

ASH (CELLULOSES)

1. SCOPE

1.1 This method covers procedures for the determination of ash in nitrocellulose, cellulose acetate, and ethyl cellulose.

2. APPARATUS

2.1 Oven. A gravity-convection type oven ventilated so that there is a minimum of ten air changes per hour and thermostatically controlled at the specified temperature $\pm 2^{\circ}$ C.

2.2 Crucible. A porcelain ignition crucible and cover.

2.3 Steam bath. A container suitable for use as a steam bath and steam source.

2.4 Bunsen burner.

2.5 Furnace. An electric muffle furnace covering a temperature range to 1880° F with a thermoregulator and pyrometer. Interior dimensions should be such that ignition crucible is not in contact with either walls or heating element.

3. REAGENTS

3.1 Nitric acid. Nitric acid, A.C.S. reagent grade.

4. PROCEDURE

4.1 Dry between 5 and 10 g of the sample for 2 hours in an oven at $105^{\circ} \pm 2^{\circ}$ C ($221^{\circ} \pm 3.6^{\circ}$ F) (see 5.1) and proceed as follows:

4.2 Nitrocellulose. Weigh to the nearest 0.1 mg approximately 2 g of the dried sample into a previously ignited and weighed porcelain crucible. Moisten with 20 to 30 drops of A.C.S. reagent grade nitric acid and heat on a steam bath until the sample is decomposed to a gummy mass. If necessary to complete decomposition, add several additional drops of the acid at intervals. Then heat the crucible slowly over a Bunsen burner or in a muffle furnace until all volatile matter is driven off. Finally ignite at red heat to constant weight. Cool, weigh, and calculate the percentage of ash.

4.3 Cellulose acetate. Weigh to the nearest 0.1 mg between 5 and 10 g of the dried sample into an ignited and weighed porcelain crucible. Heat the crucible and its contents over a flame from a Bunsen burner in such a manner that the sample will fume off slowly without igniting. When no further fumes are evolved

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and the bottom of the crucible is dull red, transfer the crucible to a muffle furnace and heat at 500° to 600° C (932° to 1112° F) for 1 hour or longer if required to burn off all the carbon. Cool in a desiccator, weigh, and calculate the percentage of ash.

4.4 Ethyl cellulose. Weigh to the nearest 0.1 mg between 2 and 5 g of the dried sample into an ignited and weighed porcelain crucible. Burn off most of the organic matter over a flame from a Bunsen burner. Finally heat the crucible in a muffle furnace at 900° \pm 50° C (1652° \pm 90° F) for 1 hour or longer if required to burn off all the carbon. Cool in a desiccator, weigh, and calculate the percentage of ash.

5. NOTES

5.1 Danger from flammable vapor may be avoided by a preliminary air drying of the sample in the event that the number of samples being tested or the construction of the oven are such as to warrant this precaution.

6. PRECISION

6.1 No data.

METHOD 5271
January 24, 1986

MATTER INSOLUBLE IN ACID

1. SCOPE

1.1 This method describes a procedure for the determination of acid insoluble matter in extracted pigments.

2. APPARATUS

2.1 Weighing device. A case enclosed analytical balance having a sensitivity of 0.1 mg at full load and a set of NBS class S-1 weights or equal.

2.2 Beaker. A 200 ml borosilicate glass beaker.

2.3 Graduated cylinder. A 10 ml graduated cylinder made of borosilicate glass, calibrated to deliver at 20 degrees with 0.1 ml subdivisions and an accuracy of ± 0.1 ml.

2.4 Hotplate. An electric hotplate.

2.5 Filter. A filter consisting of a 60 degrees short stem chemical filtering funnel and Whatman No. 41 filter paper or equal.

2.6 Bunsen burner.

2.7 Steam bath. A container suitable for use as a steam bath and steam source.

3. REAGENTS

3.1 Alcohol. Ninety-five percent ethyl alcohol.

3.2 Hydrochloric acid. One to one or 6 N HCl prepared by diluting concentrated HCl, A.C.S. reagent grade 12 N, with an equal volume of distilled water.

3.3 Hydrochloric acid. HCl, A.C.S. reagent grade.

3.4 Sulfuric acid. H_2SO_4 , A.C.S. reagent grade.

3.5 Ammonium sulfate. $(NH_4)_2SO_4$, A.C.S. reagent grade.

4. PROCEDURE

4.1 Weigh to the nearest 0.1 mg about 1 g of the extracted pigment, as obtained by method 4021, into a 200-ml beaker. Moisten the pigment with a few drops of 95 percent ethyl alcohol, add 40 ml of 1 to 1 hydrochloric acid, cover with a watch glass and boil gently for 5 to 10 minutes. Remove and wash off the

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watchglass. Evaporate the contents of the beaker to dryness and heat at about 150° C (302° F) for 1/2 to 1 hour to dehydrate the residue. Moisten the residue with 4 ml of A.C.S reagent grade hydrochloric acid, and allow to stand a few minutes. Dilute with 100 ml of hot distilled water and boil for a few minutes. Filter while hot through an ashless filter paper, washing all the insoluble matter from the beaker onto the filter paper with a stream of hot water from a wash bottle. Wash the residue on the filter paper until the washings no longer give a test for lead and chlorine. Ignite the paper and residue in a previously ignited and weighed crucible until all carbonaceous matter is destroyed. Cool in a desiccator, weigh, and calculate the percentage of insoluble matter.

4.2 If the amount of insoluble matter is found to be in excess of that allowed in the product specification, transfer it from the crucible to a 400-ml beaker. Add 10 g of A.C.S. reagent grade ammonium sulfate and 20 ml of A.C.S. reagent grade sulfuric acid. Heat to boiling and boil for 10 minutes. Cool, dilute to 200 ml and again boil for 20 minutes. Let settle on a steam bath. Filter while hot through an ashless filter paper and wash the paper and residue with 5 percent sulfuric acid. Ignite, cool, weigh, and calculate the percentage of acid insoluble matter as described above.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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Section 6000

PHYSICAL AND CHEMICAL TESTS OF DRIED FILMS

METHOD 6051
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HEAT RESISTANCE

1. SCOPE

1.1 This method covers a general procedure for determining the resistance to heat of certain types of organic coating materials.

2. REAGENTS

2.1 None.

3. REQUIRED INFORMATION

The following information shall be given in the product specification by inserting the details in the following list.

3.1 Preparation of panel.

3.1.1 Nature of panel.

3.1.2. Size of panel.

3.1.3 Pretreatment of panel (if required).

3.2 Number of coats.

3.3 Thickness of each coat.

3.4 Time of drying or aging.

3.5 Manner of drying or aging.

3.6 Relative position (vertical or horizontal).

3.7 Duration of exposure (hrs).

3.8 Temperature of exposure.

3.9 Comparative test coating, if any.

3.10 Diameter of rod for bend test (if required).

4. PROCEDURE

4.1 Arrange the test coating in a well-ventilated electric oven or thermostatically controlled muffle furnace and heat at the specified temperature for the specified exposure period. Remove and examine the test coating as specified for conformance to the requirements of the product specification.

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4.2 When a flexibility determination is specified, proceed as follows: After removal from the oven, condition the test coating for one-half hour at $23^{\circ} \pm 1.1^{\circ} \text{C}$ ($73.4^{\circ} \pm 2^{\circ} \text{F}$) and a relative humidity of 50 ± 4 percent. Place the panel with the test coating uppermost over a rod of the specified diameter, held firmly in suitable supports, at a point equally distant from the top and bottom edge of the panel. Bend the panel double in 1 second. Examine the coating over the bent area, using a 7-power lens, for conformance to the requirements of the product specification. Cracks extending no more than one-quarter inch from either end shall be disregarded.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 6091.1
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BLUSH RESISTANCE

1. SCOPE

1.1 This method determines the resistance of a coating of blushing.

2. APPARATUS

2.1 None.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Application. Prepare a film as specified in the product specification and immediately place the panel in a conditioning chamber maintained at 23° to 27° C and 60 to 70 percent relative humidity. Air in the chamber shall be completely changed every 10 minutes.

4.2 Examination. Examine the film while it is drying at intervals not exceeding 5 minutes during the first hour of drying, or after it has been dried and rubbed as specified in the product specification.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 6101.1
January 24, 1986

60-DEGREE SPECULAR GLOSS

1. SCOPE

1.1 This method is used for the determination of specular gloss of most nonmetallic specimens and for determining when methods 6103 and 6104 are applicable. This is the primary method for determining the gloss of materials usually described as semigloss (60° values 30 to 70).

1.2 Definitions.

1.2.1 Specular gloss. The luminous fractional reflectance of a specimen at the specular direction.

1.2.2 Luminous fractional reflectance. The ratio of the luminous flux reflected from, to that incident on, a specimen for specified solid angles.

1.3 Standards.

1.3.1 Primary standards may be high polished, plane, black-glass surfaces. Polished black glass with refractive index of 1.567 shall be assigned a specular gloss value of 100.

1.3.2 Secondary working standards^{1/} shall be of materials having hard, uniform surfaces, such as ceramic tile or depolished opaque glass. Secondary standards shall be calibrated against primary standards on a glossmeter conforming to the geometric requirements of this method.

2. APPARATUS

2.1 Instrumental components. The apparatus shall consist of an incandescent light source, means for locating the specimen surface, and a receptor located to receive the required pyramid of rays reflected by the specimen.

2.2 Geometric conditions. The axis of the incident beam shall be 60 degrees ± 0.1 degree from the perpendicular to the test surface. The axis of the receptor shall be at the mirror reflection of the axis of the incident beam. The angular dimensions (apertures) and tolerances of the source and receptor shall be as indicated.

2.3 Measurement mechanism. The receptor-measurement mechanism shall give a numerical indication proportional to the light flux passing the receptor field stop within ± 1 percent of full scale.

^{1/} Suitable working standards for this gloss test are available from the Gardner Laboratory, Inc., 2431 Linden Lane, Silver Spring, MD. and the Hunter Associated Laboratory, Inc., 11495 Sunset Road, Reston, VA.

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TABLE I. Angular dimensions and tolerances of receptor and source.

Relation to measurement plane	Source	Receptor
In plane	0.75 \pm 0.25 deg	4.4 \pm 0.1 deg
Perpendicular to plane.	3.00 deg max	11.7 \pm 0.2 deg

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Apparatus adjustment.

4.1.1 Test aperture ratio and image centering by adjusting the instrument to read correctly the gloss of a polished glass standard and then reading the gloss of one or more standards having poor image-forming characteristics. If the instrument readings of the latter standards do not agree closely with the calibration values for these standards, readjust the instrument according to instructions of the manufacturer or return the instrument to him for readjustment.

4.2 Preparation of test specimens.

4.2.1 Unless otherwise specified, prepare films of the finish to be tested by drawing down with a doctor blade to yield a wet film thickness of approximately 0.003 inch on plane opaque white glass panels as prescribed in 2.1.5 of method 2021. Dry panels for 48 hours at a temperature of 23° \pm 1.1° C (73.4° \pm 2° F), a relative humidity of 50 \pm 4 percent, and under dust-free conditions.

4.3 Calibration. If the apparatus is properly adjusted, calibrate the glossmeter at the start and completion of every period of operation, and during operation at sufficiently frequent intervals to assure constancy of instrument response. To calibrate, adjust the instrument to read correctly the gloss of a standard whose assigned value is approximately that of the glossiest specimens to be measured.

4.3.1 Measurement. Measure at least three portions of the specimen surface to obtain an indication of uniformity.

4.4 Specular gloss components.

4.4.1 Evaluation of the specular gloss components, surface-reflected flux and body-reflected flux, may be required when comparing low-gloss specimens or

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widely differing body reflectance. When required, evaluate approximately the body-reflected flux and subtract it from the specular gloss to obtain an approximate value for the surface-reflected flux.

4.4.2 Body reflected flux. Obtain an approximation of the body-reflected flux by using either of the following techniques:

4.4.2.1 Adjust and calibrate a goniophotometer to read correctly 60 degrees specular gloss, then illuminate the specimen perpendicularly and view at 60 degrees without change of apertures. Read the photometer to obtain the approximate value for the body reflected flux.

4.4.2.2 Alternatively express as a decimal fraction the 45 degrees, 0 degree luminous directional reflectance measured according to ASTM E 97 and multiply by 2.1 to obtain an approximate value for the body-reflected flux.

4.5 Report of results.

4.5.1 Report the average of the specular gloss readings.

4.5.2 Report the presence of any specimen having portions of the test surface differing in gloss by more than 5 percent of the average gloss of that specimen.

4.5.3 Report the method of specimen preparation if different from that specified in 4.2.

5. NOTES

5.1 Results obtained may be uncertain due to the cumulative effect of several sources of error: that is, difference between the geometric distribution of flux reflected from standards and specimens may bring about uncertainties in the measured gloss even though the source and receiver apertures are within the tolerances specified; inaccuracy of reading may result even though the precision of the measurement mechanism is held within the tolerance specified, and lens arrangement and stray reflections from the interior may cause errors in gloss readings.

6. PRECISIONS

6.1 Readings obtained on the same instrument should be repeatable to within 1 percent of the magnitude of the readings. Readings obtained on different instruments should be reproducible to within 5 percent of the magnitude of the readings (see 5.1).

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METHOD 6103
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85-DEGREE SPECULAR GLOSS (SHEEN)

1. SCOPE

1.1 This method is used for the determination of specular gloss of nonmetallic specimens having a 60 degree specular gloss lower than 30 as measured by method 6101. Such materials are usually described as flat or lusterless (85° values less than 15), eggshell flat (60° values 10 to 15), and eggshell gloss (60° values 15 to 30).

1.2 Definitions.

1.2.1 Specular gloss. The luminous fractional reflectance of a specimen at the specular direction.

1.2.2 Luminous fractional reflectance. The ratio of the luminous flux reflected from, to that incident on, a specimen for specified solid angles.

1.3 Standards.

1.3.1 Primary working standards may be highly polished, plane, black-glass surfaces. Polished black glass with refractive index of 1.567 shall be assigned a specular gloss value of 100.

1.3.2 Secondary working standards^{1/} shall be of materials having hard, uniform surface, such as ceramic tile or depolished opaque glass. Secondary standards shall be calibrated against primary standards on a glossmeter conforming to the geometric requirements of this method.

2. APPARATUS

2.1 Instrumental components. The apparatus shall consist of an incandescent light source, means for locating the specimen surface, and a receptor located to receive the required pyramid of rays reflected by the specimen.

2.2 Geometric conditions. The axis of the incident beam shall be 85 degrees ± 0.1 degree from the perpendicular to the test surface. The axis of reception shall be at the mirror reflection of the axis of the incident beam. The angular dimensions (apertures) and tolerances of the source and receptor shall be as indicated in table I.

^{1/} Suitable working standards for this gloss test are available from Gardner Lab., Inc., 2431 Linden Lane Silver Spring, MD and the Hunter Associates Laboratory, Inc., 11495 Sunset Rd, Reston, VA.

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TABLE I. Angular dimensions and tolerances of receptor and source.

Relation to measurement plane	Source	Receptor
In plane	0.75 \pm 0.25 deg.	4.0 \pm 0.3 deg
Perpendicular to plane.	3.00 deg max	6.0 \pm 0.3 deg

2.3 Measurement mechanism. The receptor-measurement mechanism shall give a numerical indication proportional to the light flux passing the receptor field stop within ± 1 percent of full scale.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Apparatus adjustment.

4.1.1 Test aperture ratio and image centering by adjusting the instrument to read correctly the gloss of a polished glass standard and then reading the gloss of one or more standards having poor image-forming characteristics. If the instrument readings of the latter standards do not agree closely with the calibration values for these standards, readjust the instrument according to instructions of the manufacturer or return the instrument to him for readjustment.

4.2 Preparation of test specimens.

4.2.1 Unless otherwise specified, prepare films of the finish to be tested by drawing down with a doctor blade of 0.006 inch gap clearance to yield a wet film thickness of approximately 0.003 inch on plane opaque white glass panels as prescribed in 2.1.5 of method 2021. Dry panels for 48 hours at a temperature of $23^{\circ} \pm 1.1^{\circ} \text{C}$ ($73.4^{\circ} \pm 2^{\circ} \text{F}$), a relative humidity of 50 ± 4 percent, and under dust-free conditions.

4.3 Calibration. If the apparatus is properly adjusted, calibrate the glossmeter at the start and completion of every period of operation and during operation at sufficiently frequent intervals to assure constancy of instrument response. To calibrate, adjust the instrument to read correctly the gloss of a standard whose assigned value is approximately that of the glossiest specimens to be measured.

4.3.1 Measurement. Measure at least three portions of the specimen surface to obtain an indication of uniformity.

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4.4 Reporting of results.

4.4.1 Report the average of the specular gloss readings.

4.4.2 Report the presence of any specimen having portions of the test surface differing in gloss by more than 5 percent, of the average gloss of that specimen.

4.4.3 Report the method of specimen preparation if different from that specified in 4.2.1.

5. NOTES

5.1 Results obtained may be uncertain due to the cumulative effect of several sources of error: that is, difference between the geometric distribution of flux reflected from standards and specimens may bring about uncertainties in the measured gloss even though the source and receiver apertures are within the tolerances specified; inaccuracy of reading may result even though the precision of the measurement mechanism is held within the tolerance specified, and lens arrangement and stray reflections from the interior may cause errors in gloss readings.

6. PRECISION

6.1 Readings obtained on the same instrument should be repeatable to within 1 percent of the magnitude of the readings. Readings obtained on different instruments should be reproducible to within 5 percent of the magnitude of the readings (see 5.1).

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METHOD 6104
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20-DEGREE SPECULAR GLOSS

1. SCOPE

1.1 This method is used for measuring the gloss of nonmetallic specimens having a 60 degree specular gloss higher than 70, as measured by method 6101. Such materials are usually described as full gloss.

1.2 Definitions.

1.2.1 Specular gloss. The luminous fractional reflectance of a specimen at the specular direction.

1.2.2 Luminous fractional reflectance. The ratio of the luminous flux reflected from, to that incident on, a specimen for specified solid angles.

1.3 Standards.

1.3.1 Primary working standards may be highly polished, plane, black glass surfaces. Polished black glass with refractive index of 1.567 shall be assigned a specular gloss value of 100.

1.3.2 Secondary working standards^{1/} shall be of materials having hard, uniform surface, such as ceramic tile or depolished opaque glass. Secondary standards shall be calibrated against primary standards on a glossmeter conforming to the geometric requirements of this method.

2. APPARATUS

2.1 Instrumental components. The apparatus shall consist of an incandescent light source, means for locating the specimen surface, and a receptor located to receive the required pyramid of rays reflected by the specimen.

2.2 Geometric conditions. The axis of the incident beam shall be 20 degrees ± 0.1 degree from the perpendicular to the test surface. The axis of reception shall be at the mirror reflection of the axis of the incident beam. The angular dimensions (apertures) and tolerances of the source and receptor shall be as indicated in table I.

TABLE I. Angular dimensions and tolerances of source and receptor.

Relation to measurement plane	Source	Receptor
In plane	0.75 ± 0.25 deg	1.80 ± 0.05 deg
Perpendicular to plane.	3.00 deg max	3.60 ± 0.10 deg

^{1/} Suitable working standards for this gloss test are available from Gardner Lab., Inc., 2431 Linden Lane, Silver Spring, MD and the Hunter Associates Laboratory, Inc., 11495 Sunset Rd, Reston, VA.

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2.3 Measurement mechanism. The receptor-measurement mechanism shall give a numerical indication proportional to the light flux passing the receptor field stop within ± 1 percent of full scale.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Apparatus adjustment.

4.1.1 Test aperture ratio and image centering by adjusting the instrument to read correctly the gloss of a polished glass standard and then reading the gloss of one or more standards having poor image-forming characteristics. If the instrument readings of the latter standards do not agree closely with the calibration values for these standards, readjust the instrument according to instructions of the manufacturer or return the instrument to him for readjustment.

4.2 Preparation of test specimens.

4.2.1 Unless otherwise specified, prepare films of the finish to be tested by drawing down with a doctor blade of 0.006 inch gap clearance to yield a wet-film thickness of approximately 0.003 inch on-plane opaque white glass panels as prescribed in 2.1.5 of method 2021. Dry panels for 48 hours at a temperature of $23^{\circ} \pm 1.1^{\circ} \text{C}$ ($73.4^{\circ} \pm 2^{\circ} \text{F}$), a relative humidity of 50 ± 4 percent, and under dust-free conditions.

4.3 Calibration. If the apparatus is properly adjusted, calibrate the glossmeter at the start and completion of every period of operation, and during operation at sufficiently frequent intervals to assure constancy of instrument response. To calibrate, adjust the instrument to read correctly the gloss of standard whose assigned value is approximately that of the glossiest specimens to be measured.

4.3.1 Measurement. Measure at least three portions of the specimen surface to obtain an indication of uniformity.

4.4 Reporting of results.

4.4.1 Report the average of the specular gloss readings.

4.4.2 Report the presence of any specimen having portions of the test surface differing in gloss by more than 5 percent of the average gloss of that specimen.

4.4.3 Report the method of specimen preparation if different from that specified in 4.2.

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5. NOTES

5.1 Results obtained may be uncertain due to the cumulative effect of several sources of error: that is, difference between the geometric distribution of flux reflected from standards and specimens may bring about uncertainties in the measured gloss even though the source and receiver apertures are within the tolerances specified; inaccuracy of reading may result even though the precision of the measurement mechanism is held within the tolerance specified, and lens arrangement and stray reflections from the interior may cause errors in gloss readings.

6. PRECISION

6.1 Readings obtained on the same instrument should be repeatable to within 1 percent of the magnitude of the readings. Readings obtained on different instruments should be reproducible to within 5 percent of the magnitude of the readings (see 5.1).

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METHOD 6122.1
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LIGHTNESS INDEX DIFFERENCE

1. SCOPE

1.1 This method is used for color specimens whose color difference from a standard is predominantly a lightness difference.

1.2 Definitions.

1.2.1 Lightness index. Lightness index, L , is defined by the equation

$$L = 10 \sqrt{Y}$$

Where Y is the luminous directional reflectance expressed in percent relative to magnesium oxide.

1.2.2 Lightness index difference. Lightness index difference, ΔL , is the difference in the lightness index before and after exposure.

2. APPARATUS

2.1 Use the apparatus described in method 4252.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of test specimen. Prepare the test specimen as prescribed in ASTM E 97, or as specified in the product specification.

4.2 Exposure of test specimen. Expose the test specimen under the conditions and for time period as specified in the product specification.

4.3 Measurement. Using the apparatus and standards specified in method 4252, measure the directional reflectance of the test specimen before exposure and record as Y_0 . Measure the directional reflectance over the same area of the test specimen after exposure and record as Y_1 .

4.4 Computation. Compute the lightness index, L_0 , for the test specimen before exposure and the lightness index, L_1 , after exposure by substituting the Y_0 and Y_1 , values respectively in the equation given in 1.2.1. Obtain the lightness index difference, ΔL , by the equation

$$\Delta L = L_1 - L_0$$

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5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 6123
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COLOR DIFFERENCE OF OPAQUE MATERIALS
INSTRUMENTAL MEASUREMENT

1. SCOPE

1.1 This method describes procedures for instrumentally determining small color differences, observable in daylight illumination, between nonfluorescent, nonmetameric, opaque surfaces such as paint specimens. The color differences determined by these procedures are expressed in units of approximate uniform visual color perception in the modified Adams color space.

1.2 For product specification, the permissible color difference between test specimen and reference specimen shall be agreed upon by the purchaser and seller. Specific color tolerances may be required for each material and condition of use since other appearance factors (e.g. proximity, gloss, and texture) may affect the correlation between the measured color difference and its commercial acceptability.

2. OUTLINE OF METHOD

2.1 Differences in color between a reference specimen and each test specimen are determined with a spectrophotometer or a colorimeter. Reflectance readings are made which in turn are converted by computations to L, a, and b color scale values, or these color scale values may be read directly from an instrument that automatically makes the computations. Color difference magnitudes are computed from ΔL , Δa , and Δb values, which represent the color scale differences between the reference specimen and the test specimen.

3. DEFINITIONS

3.1 Color space. The daylight colors of opaque specimens for the purpose of this method are represented by points in a space formed by three mutually perpendicular unit vectors, \bar{L} , \bar{a} , and \bar{b} , having scales, L, a, and b, respectively. These scales were chosen so that color differences in this space correlate with perceived color differences.

3.2 Color differences. E, is the resultant vector of three component differences, lightness-index difference, ΔL , and chromaticity differences, Δa and Δb , and may be expressed thus:

$$E = (\Delta L)\bar{L} + (\Delta a)\bar{a} + (\Delta b)\bar{b}$$

The above vector notation indicates that ΔL , Δa , and Δb are magnitudes of differences reacting in the direction of L, a, and b. The latter are considered as unity. The magnitude of this resultant, E, called the color difference, is a scalar quantity and may be computed from the following:

$$E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

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The magnitude, E, of course, gives no indication of the character of the difference since it does not indicate the relative quantity and direction of hue, saturation, and lightness differences.

The direction of the color difference is described by the magnitude and algebraic signs of the components ΔL , Δa , and Δb .

$$\begin{aligned}\Delta L &= L_1 - L_0 \\ \Delta a &= a_1 - a_0 \\ \Delta b &= b_1 - b_0\end{aligned}$$

Where: L_0 , a_0 , and b_0 refer to the reference specimen, and L_1 , a_1 , and b_1 refer to the test specimen. The sign of the components ΔL , Δa , and Δb have the following approximate meanings:

- + ΔL = lighter
- ΔL = darker
- + Δa = redder (less green)
- Δa = greener (less red)
- + Δb = yellower (less blue)
- Δb = bluer (less yellow)

3.3 Color scales. The L, a, and b color scales shall be based on any one of the following color scale systems. Although each of these systems will provide color difference values in approximate agreement with average visual judgments of color difference, one of the systems may be more suitable for use with a specific color measuring instrument than the others.

The color scale values obtained with different color measuring instruments generally cannot be expected to agree exactly because of differences in the characteristics of the instruments and differences in the equations used.

Color scale system A_1 is based on the Adams chromatic value diagrams combined with the Munsell value scale. In this system,

$$\begin{aligned}L_A &= 9.2V_y \\ a_A &= 40 (V_x - V_y) \\ b_A &= 16 (V_y - V_z)\end{aligned}$$

where: V_x , V_y , and V_z are Munsell values related to tristimulus values X, Y, and Z for the CIE standard observer and source C. V_y is defined as follows:

$$\begin{aligned}Y_s/Y_{M80} &= 1.2219 V_y - 0.23111 V_y^2 \\ &+ 0.23951 V_y^3 - 0.021009 V_y^4 + 0.0008404 V_y^5\end{aligned}$$

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where:

Y_S = reflectance of a specimen,
 Y_{MgO} = reflectance of a magnesium oxide primary standard, and
 V_X and V_Z are defined similarly by substituting the tristimulus values, X and Z, respectively, for the reflectance Y.

Note 1. The conversion of tristimulus values to the values V_X , V_Y , and V_Z may be made by using tables prepared for this purpose, or by using a suitably programmed computer.

Note 2. Color system A_1 is preferred for determining color differences with the General Electric Recording Spectrophotometer and the Color Eye Colorimeter.

Color scale A_2 is designed for three filter colorimeters. In this system,

$$\begin{aligned} L_C &= 10.0 V (G) \\ a_C &= a_{R'} - a_G \\ b_C &= b_G - b_B \end{aligned}$$

where:

$$\begin{aligned} a_G &= 41.86 V (G) \\ a_{R'} &= 41.86 V (R') \\ b_B &= 16.74 V (B) \\ b_G &= 16.74 V (G) \end{aligned}$$

where: $R' = 0.8R + 0.2B$, V refers to the Munsell value function, and R , G , and B are colorimeter reflectance readings for red, green, and blue filters, respectively. These quantities are related to the tristimulus values for the CIE standard observer and source C as follows:

$$\begin{aligned} R' &= 1.020 X \\ G &= Y \\ B &= 0.847Z \end{aligned}$$

Note 3. The conversion of R' , G , and B values to the values of L , a_G , b_G , $a_{R'}$, and b_B may be made by means of tables prepared for this purpose.

Note 4. Color system A_2 is preferred for determining color differences with the Colormaster Differential Colorimeter.

Color scale system B_1 is designed for direct reading colorimeters. In this system,

$$\begin{aligned} L_R &= 10 Y \\ a_R &= 1.75 f_y (1.02X - Y) \\ b_R &= 70 f_y (Y - 0.847Z) \end{aligned}$$

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where

$$f_y = \frac{0.51 (21 + .20Y)}{(1 + .20Y)}$$

and X, Y, and Z are tristimulus values for the CIE standard observer and source C.

Color scale system B₂, is based on an alternate color system designed for direct reading colorimeters. In this system,

$$L_L = 10\sqrt{Y}$$

$$a_L = \frac{17.5(1.02X - Y)}{\sqrt{Y}}$$

$$b_L = \frac{7.0(Y - 0.847Z)}{\sqrt{Y}}$$

where X, Y, and Z are tristimulus values for the CIE standard observer and source B.

Note 5: Color systems B₁ and B₂ are preferred for determining color differences with color difference meters of the Hunterlab or Gardner types.

Color scale system C, is designed for three filter colorimeters. In this system,

$$L_G = 10\sqrt{G}$$

$$a_M = 70(G)^{1/2}(A - G)/(B + A + 2G)$$

$$b_G = \frac{28\sqrt{G}(G - B)}{(B + A + 2G)}$$

where G, A, and B are colorimeter reflectance readings for green, amber, and blue filters, respectively. These quantities are related to the tristimulus values for the CIE standard observer and source C as follows:

$$A = 1.277 X - 0.213Z$$

$$G = Y$$

$$B = 0.847Z$$

Note 6: Color system C is preferred for determining color differences with the Gardner (Hunter) Multi-purpose Reflectometer.

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4. APPARATUS

4.1 Instrumental components. The apparatus shall consist of means for locating the surface of the specimen, a light source furnishing a beam incident on the specimen, and a receptor located to receive the required rays reflected by the specimen. The receptor shall be a photosensitive device responding to visible radiation. Means shall be provided for controlling the spectral energy distribution incident on the receptor to meet the requirements of 4.3.

4.2 Geometric characteristics. The angle of viewing shall be separated from the angle of illumination in such a manner that only rays reflected diffusely from the test specimen surface enter the receptor. Provision shall be made to exclude specular reflection and stray light from the receptor.

Note 7: ASTM E 179 "Recommended Practice for Selection of Geometric Conditions for Measurement of Reflectance and Transmittance" may be used as a guide. Conditions of illumination and viewing in which interlaboratory tests have given color difference measurements in approximate agreement with average visual judgments of color difference are:

Angle of Illumination	Angle of viewing
Diffuse	40 degrees from perpendicular
45 degrees from perpendicular	0 degrees from perpendicular
6 degrees from perpendicular	Diffuse

4.3 Spectral conditions - Instrument response characteristics shall approximate the tristimulus specifications $E_c X$, $E_c Y$, and $E_c Z$ of the CIE standard observer for source C or provide ready computation of them.

Note 8: The accuracy with which a filter colorimeter's response characteristics match the tristimulus specifications of the CIE standard observer may be indicated by determining the tristimulus values X, Y, and Z of plaques of various colors and comparing these values with those computed from spectral reflectance measurements. Ideally, these plaques should be so selected as to permit the determination of the spectral centroids of each of the filter-phototube combinations and their cut-off points.

Note 9: Because of variation in the spectral sensitivity among instruments, the color scale values and color differences obtained with different instruments may not agree exactly. Therefore, to obtain the greatest precision in interlaboratory tests, comparisons of results should be made only for those instruments that possess closely similar spectral sensitivity characteristics. Of course, identical conditions of illumination and viewing and identical means of computing color scales also are required for maximum precision.

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Note 10: The following instruments meet the above apparatus requirements and are believed to provide color difference measurements in approximate agreement with average visual judgments of color difference:

Color Eye Colorimeter
G.E. Recording Spectrophotometer
Hunterlab Color Difference Meter
Colormaster Differential Colorimeter
Gardner (Hunter) Color Difference Meter
Gardner (Hunter) Multipurpose-Reflectometer

5. CALIBRATION STANDARDS

5.1 Primary standard. The primary standard for reflectance measurements is a fresh layer of magnesium oxide prepared according to ASTM D 986. This standard is assigned a percent reflectance of 100.0 at all wavelengths in the visible spectrum. Thus, its R, G, B values are each equal to 100.0 and its L, a, and b values are 100.0, 0.0, and 0.0, respectively. The assigned tristimulus values, X, Y, and Z, for CIE source C shall be 98.0, 100.0, and 118.1, respectively.

5.2 Working standards. To facilitate instrument calibration, pieces of white structural glass, known as Vitrolite, or opaque porcelain enameled plaques may be used when calibrated in terms of the primary standard and the instrument geometry with which they are to be used.

Note 11: Working standards usually can be obtained from the manufacturers of color measuring instruments. These standards shall be calibrated with the color measuring instrument with which they are to be used.

6. CALIBRATIONS

6.1 The calibration of photometer scales shall be carefully checked at reasonable time intervals in a manner to insure accuracy over all ranges.

Note 12: Calibration may be accomplished by placing a series of neutral filters of known transmittance in the incident beam or by measuring the reflectance of calibrated opaque specimens.

7. TEST SPECIMENS

7.1 These test procedures do not cover preparation techniques. Whenever a test for color difference requires the preparation of a specimen, the users of this method shall agree in advance on the technique of preparation.

Note 13: Greatest precision in color difference measurements of coatings is obtained when they are applied at a thickness that provides complete hiding of the substrate. If measurements must be made on coatings at incomplete hiding,

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then the optical hiding of all specimens should be maintained within close tolerances and color of substrate held constant. Best results are also obtained when the surfaces of specimens are smooth and uniform.

7.2 One of the specimens to be tested shall be designated as the reference specimen. The others shall be designated as test specimens.

8. PROCEDURE

8.1 Measurements.

8.1.1 Operate the instruments according to the manufacturer's instructions.

8.1.2 Standardize the instrument with either a primary or working standard. Restandardize at sufficient intervals to minimize instrument drift.

8.1.3 When a spectrophotometer is used, obtain the reflectance values of the reference specimen and test specimens, in turn, at a sufficient number of wavelength intervals to permit accurate calculation of CIE tristimulus values.

8.1.4 When a reflectance reading filter colorimeter is used, obtain the reflectance values of the reference specimen and test specimens, in turn, with each of the filters.

8.1.5 When a color scale reading colorimeter is used, obtain the color scale values of the reference specimen and test specimens, in turn.

8.1.6 For any instrument used, remeasure the working standard to determine whether instrumental drift has occurred during measurement. Compensate for instrumental drift, if observed, or remeasure specimens.

Note 14: For maximum accuracy and precision of measurements, the instrument should be adjusted to read the reflectance values, or color scale values, assigned to a working standard whose spectral reflectance characteristics are similar to those of the specimens to be measured.

8.1.7 Measure at least three portions of each specimen surface to obtain an indication of uniformity. Mark the location of these measurements on the specimen.

8.2 Calculations.

8.2.1 If reflectance measurements were obtained for the specimens, convert these readings to L, a, and b color scale values by means of equations, tables, or other appropriate computational procedures for one of the color scale systems outlined in 3.2.

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Note 15: The computation of tristimulus values from spectrophotometric reflectance values is greatly facilitated by using an automatic integrator or by introducing reflectance data into a programmed digital computer.

8.2.2 Calculate the lightness and chromaticity index differences, ΔL , Δa , Δb for each test specimen relative to the reference specimen. Indicate whether the index differences are positive or negative.

8.2.3 Calculate the color difference magnitude, E , for each test specimen relative to the reference specimen by using the equation:

$$E = \sqrt{(\Delta L^2) + (\Delta a^2) + (\Delta b^2)}$$

9. REPORT

9.1 Report the color difference, E , of each test specimen from the reference specimen. Indicate the direction of color difference by reporting the lightness and chromaticity differences, ΔL , Δa , and Δb , together with their signs. For nonuniform specimens, report the range of color difference magnitudes obtained for different areas. Report the L , a , and b values for the reference specimen.

9.2 Describe or otherwise identify the method of preparing the specimens.

9.3 Identify the instrument used by the manufacturer's name and model number.

9.4 Identify the color scale system used by the designation in 3.3.

10. PRECISION

10.1 Limited interlaboratory tests were conducted to determine measurement precision for color difference magnitudes ranging from about 0.5 to 9.5. The samples used in these tests were high gloss enamels with systematic variation in purity and lightness in three hue groups. Five different types of color measuring instruments were used and the resulting measurements indicated the following precision may be expected from this method, depending on the type of instrument used:

Color difference, E		Component differences $\Delta L, \Delta a, \Delta b$	
Repeatability	Reproducibility	Repeatability	Reproducibility
0.1 to 0.2	0.2 to 0.6	0.1	0.2 to 0.3

Note 16: Repeatability, is defined as the standard deviation in results obtained by one operator using one instrument in one laboratory.

Reproducibility, is defined as the standard deviation in results obtained by different operators using the same type instrument in different laboratories.

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METHOD 6124
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UNDERWATER REFLECTANCE OF CAMOUFLAGE COATINGS

1. SCOPE

1.1 This method determines the absolute underwater reflectance R_0 , for camouflage coatings in the range of R_0 from 0 to 6.5 percent.

2. APPARATUS

2.1 As described in ASTM E 97.

3. REAGENTS

3.1 Water containing 0.009 \pm 0.001 percent of an anionic surfactant (see 5.1) shall be used to wet the specimen.

4. PROCEDURE

4.1 Preparation of specimen.

4.1.1 Application of the coating, selection of the substrate, and drying time shall be as specified in the material specification.

4.2 Reflectance measurement.

4.2.1 Operate the reflectometer in accordance with the operating manual. Zero the galvanometer with the reflectometer scale set at mid-range, the light on, the light control window closed, and a 0 percent reflectance standard in the sample window.

4.2.2 Calculation. Underwater reflectance, R_0 , is calculated as follows:

$$R_0(\text{percent}) = \frac{R_w}{0.42 R_w + 0.564} \times 100$$

Where R_w is the reflectance determined in 4.3, through a green filter and at a 45-degree, 0-degree geometry.

4.3 Underwater reflectance.

4.3.1 For coatings having R_0 up to 2.6 percent. Standardize the instrument with the green filter using the $G = 2.2$ percent standard (in a dry condition) set on the instrument scale at 0.88. Cover the specimen to be tested with a thin film of water and obtain the green reflectance reading. Calculate the wet reflectance in percent: $R_w = 1/4$ reading $\times 10$. Using R_w , determine the underwater reflectance, R_0 , from table I.

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4.3.2 For coatings having R_0 from 2.6 to 4.0 percent. Standardize the instrument with the green filter using the $G = 2.2$ percent standard (in a dry condition) set on the instrument scale at 0.66. Cover the specimen to be tested with a thin film of water and obtain the green reflectance reading. Calculate the wet reflectance in percent: $R_w = 1/3$ reading $\times 10$. Using R_w , determine the underwater reflectance, R_0 , from table I. If the scale reading is less than 0.45, determine R_w in accordance with the procedure for R_0 up to 2.6.

4.3.3 For coatings having R_0 from 4.0 to 6.5 percent. Standardize the instrument with the green filter using the $G = 2.2$ percent standard (in a dry condition) set on the instrument scale at 0.44. Cover the specimen to be tested with a thin film of water and obtain the green reflectance reading. Calculate the wet reflectance in percent $R_w = 1/2$ reading $\times 10$. Using R_w , determine the underwater reflectance R_0 from table I. If the scale reading is less than 0.46, determine R_w in accordance with the procedure for R_0 from 2.6 to 4.0. If the scale reading is more than 0.74, this procedure does not apply.

5. NOTES

5.1 A suitable anionic surfactant, Aerosol OT, is available from American Cyanamid Company, Bound Brock, New Jersey 08805.

5.2 The expanded scale is used so that readings are obtained in the middle of the instrument scale. This eliminates the error introduced when the sensor is close to the panel on the low end of the scale. This error is considerable; values of $R_0 = 1.8$ on the expanded scale read $R_0 = 2.2$ on the unexpanded scale.

5.3 Coatings having an R_w of up to 1.48 (R_0 up to 2.6 percent) will be determined on a scale of $2.2 = 0.88$ using the $G = 2.2$ percent standard. Coatings having an R_w from 1.48 to 2.3 (R_0 from 2.6 up to 4.0 percent) will be determined on a scale of $2.2 = 0.66$ using the $G = 2.2$ percent standard. Coatings having an R_w from 2.3 to 3.8 (R_0 from 4.0 to 6.5 percent) will be determined on a scale of $2.2 = 0.44$ using the $G = 2.2$ percent standard. Coatings having an R_0 above 6.5 percent are not currently considered to be camouflage coatings.

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TABLE 1. Determination of percent underwater reflectance (R_0)
from percent wet reflectance (R_w).

R_w	R_0	R_w	R_0	R_w	R_0
0.50	0.88	2.00	3.49	3.50	6.05
0.55	0.97	2.05	3.58	3.55	6.13
0.60	1.06	2.10	3.67	3.60	6.22
0.65	1.15	2.15	3.75	3.65	6.30
0.70	1.24	2.20	3.84	3.70	6.38
0.75	1.32	2.25	3.92	3.75	6.47
0.80	1.41	2.30	4.01	3.80	6.55
0.85	1.50	2.35	4.10	3.85	6.64
0.90	1.59	2.40	4.18	3.90	6.72
0.95	1.67	2.45	4.27	3.95	6.80
1.00	1.76	2.50	4.35	4.00	6.89
1.05	1.85	2.55	4.44	4.05	6.97
1.10	1.93	2.60	4.52	4.10	7.05
1.15	2.02	2.65	4.61	4.15	7.14
1.20	2.11	2.70	4.69	4.20	7.22
1.25	2.20	2.75	4.78	4.25	7.30
1.30	2.28	2.80	4.86	4.30	7.39
1.35	2.37	2.85	4.95	4.35	7.47
1.40	2.46	2.90	5.03	4.40	7.55
1.45	2.54	2.95	5.12	4.45	7.64
1.50	2.63	3.00	5.20	4.50	7.72
1.55	2.72	3.05	5.29	4.55	7.80
1.60	2.80	3.10	5.37	4.60	7.89
1.65	2.89	3.15	5.46	4.65	7.97
1.70	2.98	3.20	5.54	4.70	8.05
1.75	3.06	3.25	5.63	4.75	8.13
1.80	3.15	3.30	5.71	4.80	8.22
1.85	3.24	3.35	5.80	4.85	8.30
1.90	3.32	3.40	5.88	4.90	8.38
1.95	3.41	3.45	5.96	4.95	8.47

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METHOD 6131
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YELLOWNESS INDEX

1. SCOPE

1.1 This method is used for white or near-white specimens to determine color departure from white toward yellow or color change towards yellow on exposure or treatment.

1.2 Definitions

1.2.1 Yellowness index. Yellowness index, N, is defined by equation (1) or alternatively by equation (2).

$$(1) N = \frac{(1.250X - 1.038Z)}{Y}$$

$$(2) N = \frac{(A - B)}{G}$$

1.2.1.1 In these equations X, Y, and Z are the CIE tristimulus values and A, B, and G are tristimulus values read directly by some colorimeters. A, B, and G are related to X, Y, and Z by the following equations:

$$A = 1.250X - 0.191Z$$

$$B = 0.847Z$$

$$G = Y$$

1.2.2 Yellowness index difference. Yellowness index difference, ΔN , is the difference in yellowness index of a specimen before and after exposure or treatment.

2. APPARATUS

2.1 As described in method 4252.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of test specimen. The test specimen shall be prepared as specified in the product specification.

4.2 Exposure or treatment of test film. When required, the test specimen shall be exposed, or subjected to the treatment as specified in the product specification.

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4.3 Measurement.

4.3.1 For colorimeters reading X, Y, and Z directly, determine the tristimulus values by using method 4252. For colorimeters reading A, B, and G directly, follow a similar procedure to obtain specimen values in these terms.

4.3.2 When yellowness index difference is required, determine tristimulus values before and after exposure or treatment.

4.4 Computation.

4.4.1 Yellowness index. Compute the yellowness index of test specimen by substituting the tristimulus values obtained in either equation (1) or (2) as applicable.

4.4.2 Yellowness index difference. Determine the yellowness index difference by subtracting the yellowness index of the test specimen before exposure or treatment from its yellowness index after it has been exposed or treated.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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ACCELERATED YELLOWNESS

1. SCOPE

1.1 The purpose of this method is to provide a standard procedure for accelerating the tendency of a coating to yellow.

2. APPARATUS

2.1 Panel. A 3- by 5-inch, opaque, white glass.

2.2 Desiccator. An 8-inch, in diameter, desiccator.

3. REAGENTS

3.1 Potassium sulfate. Saturated solution with excess potassium sulfate salt.

4. PROCEDURE

4.1 Preparation of panel.

4.1.1 Brush two normal coats of the coating material on a 3- by 5-inch opaque white glass as prescribed in method 2021, or white porcelain enamel panel. (This should give a dry film thickness of from 2 to 3 mils). Unless otherwise specified, allow the first coat to dry for 48 hours, before applying the second coat. Allow the second coat to dry 48 hours, or other specified time.

4.2 Performance of test.

4.2.1 Place the panel on edge in a rack or other suitable support in an 8-inch diameter desiccator, the lower portion of which has been filled with an aqueous solution of potassium sulfate. The solution shall be saturated and contain an excess of the potassium sulfate salt at $49^{\circ} \pm 1^{\circ} \text{C}$ ($120.2^{\circ} \pm 1.8^{\circ} \text{F}$). Cover the desiccator and place in the dark in an oven maintained at $49^{\circ} \pm 1^{\circ} \text{C}$. At the end of three days remove the panel, allow to cool to room temperature.

4.3 Measure the yellowness index of the coating as described in method 6131.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 6141.1
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WASHABILITY

1. SCOPE

1.1 This method determines the washability of a paint by subjecting a soiled film of the dried paint to the cleaning action of a wet sponge and cake grit soap. Measurements of reflectance and gloss of the paint film prior to and after washing provide an indication of the completeness of soil removal and of the change in gloss brought about by cleaning.

2. APPARATUS

2.1 The apparatus (see 5.1) shall consist of an electric or air-operated motor mounted on a flat metal plate, and mechanism through which the motor imparts a reciprocating motion to a sponge held in a metal box, which moves the sponge lengthwise across a test panel clamped to the plate.

2.2 Sponge. The sponge shall conform to L-S-626, type II, porosity B, and measure 95 by 73 by 38 mm.

2.3 Sponge holder. The holder shall weigh 454 g.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Preparation of test film.

4.1.1. Draw down a film of the paint on a 152 by 432 by 6 mm plate glass panel which has a plane ground surface, and which has been cleaned as specified in method 2021. The drawdown shall be 90 mm or more in width and shall be made parallel to the long dimension of the panel with an even motion. Age the panel in a ventilated room for 96 \pm 4 hours at a temperature of 23° \pm 1° C and a relative humidity of 50 \pm 5 percent; place in an oven for 24 hours at 60° \pm 2° C, and cool to room temperature.

4.2 Soiling medium (see 5.2).

4.2.1 The soiling medium shall consist of either of the following:

4.2.1.1 Carbon black (see 5.3)	8 grams
Mineral oil, USP, heavy	60 grams
Mineral spirits, odorless	32 grams
(TT-T-291, type III)	
	<hr/>
	100 grams

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Mix and disperse components to a Hagnen fineness of 7 (13 μ m) or finer, and test in accordance with ASTM D 1210.

4.2.1.2 Raw umber	35 grams
White petrolatum (U.S.P. Petroleum album)	6 grams
Mineral spirits, odorless (TT-T-295)	40 milliliters

4.3 Application of soil.

4.3.1 Soil the panel by drawing down a 0.076 mm (0.003 inch) film, 90 mm wide, of thoroughly mixed soiling medium across the middle portion of the paint film. Allow to air-dry for one-half hour, and then bake the soiled panel for 1/2 hour at 105° \pm 2° C in a well-ventilated oven, remove, cool to room temperature, and carefully brush off the excess pigment.

4.4 Performance of test.

4.4.1 Measure the specular gloss (ASTM D 523) and daylight directional reflectance (ASTM E 97) on the unsoiled portions of the film. Record the average of two readings for each property.

4.4.2 Clamp the panel on the washability apparatus so that the soiled portion of the panel is approximately in the center of the cleaning stroke. Soak the sponge in distilled water at room temperature until thoroughly wet, remove from water, and squeeze repeatedly with one hand until no water drips from the sponge, and then pour 50 ml of distilled water, measured in a graduate, uniformly over the sponge. Rub cake grit soap, conforming to P-S-571, type A (see 5.4), 25 times back and forth (i.e., 25 times in each direction) across the sponge using moderate pressure. Be sure to completely cover the entire side of the sponge with each stroke of the grit soap. Do not squeeze out any water. Insert the sponge in the holder immediately and start the motor. The sponge shall travel at a rate of 37 \pm 2 cycles (74 \pm 4 separate strokes) per minute. After 25 cycles (50 separate strokes), remove the sponge, clean in running water, squeeze out, and rewet with 50 ml of distilled water, rub with cake grit soap as before, and run another 10 cycles (20 separate strokes). This makes a total of 35 cycles or 70 separate strokes. If additional cycles should be required to remove the soils, the product specification should so state. Remove the test panel from the apparatus, rinse with running water and clean sponge, and allow to dry at room temperature. When thoroughly dry, determine its daylight directional reflectance at 60 degrees specular gloss over the previously soiled area.

4.4.3 Express the results as percentages, or units difference in the case of gloss if so specified, of the respective average values obtained just before washing.

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5. NOTES

5.1 The Gardner Washability Machine Model M-105A and accessories, available from the Gardner Laboratories, Inc., Bethesda, MD 20014, has been found satisfactory for this purpose.

5.2 A prepared staining medium, manufactured by the Leneta Co., P.O. Box 576, Ho-Ho-Kus, New Jersey 07423, has been found satisfactory.

5.3 Printer U Beads from DeGussa, Inc., 2 Pennsylvania Plaza, New York, NY 10001, have been found satisfactory.

5.4 Cake Bon Ami and Day and Frick's Spar Polish have been found suitable for this purpose.

6. PRECISION

6.1 No data.

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METHOD 6142
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SCRUB RESISTANCE

1. SCOPE

1.1 This method determines the scrub resistance of a paint applied to a primed panel by subjecting the paint system to the abrasive action of a bristle brush wetted with soap solution.

2. APPARATUS

2.1 The apparatus shall consist essentially of an electric motor mounted on a flat metal plate and a mechanism through which the motor will impart a reciprocating motion to a brush lengthwise across a test panel clamped to the plate. A satisfactory arrangement is described in method 6141, except that a brush and a brush holder are substituted for the sponge and sponge-box.

2.2 Brush. The block of the brush shall be aluminum, 3-1/2 inches long, 1-1/2 inches wide, and 1/2 inch thick. The brush stock shall be stiff, black, butt-cut Chinese hog bristle. There shall be 60 holes in the block, 5/32 inch in diameter, solidly filled with bristle. The bristles shall extend 3/4 of an inch beyond the block. If necessary, the bristle ends shall be dressed down with sandpaper, or burned on a hotplate, so that the abrading surface of the brush will be as nearly plane as possible. The total weight of the brush and brush holder shall be 16 ± 1/2 ounce; sheet lead may be fastened to the top of the brush holder to attain this weight.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Preparation of panels and accelerated aging of film.

4.1.1 Prepare two specimens for testing by using 6- by 17- by 1/4-inch plate glass panels with a plane ground surface which has been cleaned as specified in method 2021. Using the white enamel undercoat formulated as shown in table I, apply the undercoat with a 7-mil clearance film caster to obtain a dry film of 2.2 ± 0.3 mil, and a width of 5-1/4 inches. The drawdown shall be made parallel to the long dimension of the panel. If a tinted undercoat is required for contrast, tint the undercoat by adding 40 g of regular raw sienna color-in-oil and 60 g of dark chrome green color-in-oil per gallon of white undercoat. Allow the undercoat to dry in a horizontal position for not less than 24 hours prior to the application of the test sample. Apply the test sample with a 10-mil clearance caster to obtain a dry film of 1.8 ± 0.3 mil, and a width of 5-1/2 inches over the undercoat. Allow the test samples to dry in a ventilated room 96 ± 4 hours in a horizontal position at 73.5° ± 2° F and a relative humidity of 50 ± 4 percent or as otherwise specified.

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TABLE I. White enamel undercoat.

Ingredient	Weight per 100 gallons Pounds
Titanium-calcium pigment (30 percent TiO ₂) (RCHI). Titanium dioxide, rutile (TiPure R-610). China clay (ASP-400) Celite (281) Aluminum stearate (Metasap) Alkyd resin sol'n (60 percent solids) conforming to TT-R-266, type I, class B). Mineral spirits, conforming to TT-T-291, grade 1. Cobalt naphthenate drier (6 percent). Lead naphthenate drier (24 percent).	470 120 150 50 2 386 156 1.6 5.0
Antiskinning agent Fineness of grind (minimum).	As required. 5

4.2 Performance of test.

4.2.1 Immerse the brush bristles in water at 25° to 30° C (77° to 86° F) for 30 minutes to a depth of one-half inch. Shake the brush vigorously several times to remove "free" water and then soak an additional 5 minutes in a 0.5 percent Ivory flake soap solution made with distilled water. Mount the test panel firmly on the washability apparatus; place the soap-saturated brush on the paint film and start the motor. The brush shall travel at a rate of 37 \pm 2 cycles (74 separate strokes \pm 4 strokes) per minutes.

4.2.2 During the test permit additional soap solution to drop or run into the path of the brush at a rate of about 12 drops per minute, or just sufficient to keep the panel wet. Remove the panel at the end of number of cycles required in the product specification; wash immediately in water at moderate temperature, and inspect the film within the middle 6 inches of brush travel by both reflected and transmitted light for definite breaks, wear, or detachment of the film down to the undercoat. A few small pin-point size areas shall be disregarded.

4.2.3 If the first panel passes this test, the material shall be reported as satisfactory as regards scrub resistance, and the second panel need not be tested. If, however, the first panel fails the test, a second one shall be run and if it passes the test, the material shall be reported as satisfactory. If neither panel passes the test, it shall be reported as unsatisfactory as regards scrub resistance.

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5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 6192.1
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ABRASION RESISTANCE (TABER ABRASER)

1. SCOPE

1.1 This method covers a procedure for determining the abrasion resistance of films of organic coating materials in terms of "wear index" or "wear cycles" using the Taber Abraser (see 5.1).

1.2 Definitions.

1.2.1 Wear index. The wear index (rate of wear) is the loss in weight in milligrams per 1,000 cycles of abrasion under a specific set of test conditions.

1.2.2 Wear cycles. Wear cycles (amount of wear) is the number of cycles of abrasion required to wear a film of a specified thickness through to the test plate under a specific set of test conditions.

2. APPARATUS

2.1 The apparatus shall consist of a Taber Abraser and accessories as follows:

2.1.1 Abraser. The abramer shall be so constructed that wheels of several degrees of abrasiveness may be readily used. Loads of 250, 500, and 1,000 grams on each wheel shall be obtained by the use of changeable weights and counter weights.

2.1.2 Abraser turntable. The turntable of the abramer shall rotate substantially in a plane with a deviation at a distance of 1/16 inch from its periphery of not greater than ± 0.002 inch.

2.1.3 Abrasive wheels. Resilient calibrase wheels No. CS-10 or No. CS-17, as required, shall be used. Because of the slow hardening of the rubber bonding material in this type of wheel, the wheel shall not be used after the date marked on it or one year after its purchase, if wheel is not dated.

2.1.4 Refacing medium. The medium used in refacing the abrasion wheel shall be an S-11 abrasive disc.

2.1.5 Vacuum pick-up assembly. The assembly shall consist of a vacuum unit, a variable transformer suction regulator, a nozzle with bracket attachment and a connecting hose with adapter.

2.1.6 Specimen plates. Unless otherwise specified, the test plates shall be 4- by 4-inch steel plates cut from 20 gage auto body steel. The plates shall have all corners rounded and a one-quarter inch center hole. They shall be uniform and level, and have a dull finish to insure good adhesion of the coating to the metal.

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2.2 Maintenance.

2.2.1 The abraser shall be lubricated, kept free of dust and dirt, assembled for test, operated and stored, and the abrasive wheels shall be faced and kept round in accordance with the directions given in the Instruction Manual furnished with the instrument.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Conditions of test. Details, as to the following conditions of test, shall be as specified in the product specification.

4.1.1 Type of test plate other than steel (wood, masonite, etc.) to which test film is to be applied.

4.1.2 Method of application of test film (spraying, dipping or film applicator).

4.1.3 Thickness of coating to be tested when wear cycles are specified.

4.1.4 Method of drying test film (air dry or bake) and number of days film is to be aged before testing.

4.1.5 Kind of calibrase wheels to be used (CS-17 or CS-10).

4.1.6 Pressure of each wheel against test film (250, 500, or 1,000 grams).

4.1.7 Number of wear cycles to be employed (200, 500, 1,000, etc.).

4.2 Performance of test.

4.2.1 Apply the material to be tested to 3 or 5 clean, dry test plates in such a manner that uniform films of the required thickness are obtained; dry and age the test films (see 4.1.4). Weigh the test specimens to the nearest 0.1 milligram and record their respective weights, if the wear index is to be reported. If the results are to be reported in wear cycles, measure (ASTM D 1186 or D 1005) the thickness of each test film along the path the abrasion will be made in at least 10 places. Record the average thickness of each test film. Unless otherwise specified, condition the test film to 23° +1.1° C (73.4° +2° F) and 50 +4 percent relative humidity. Mount the abrasive wheels (see 4.1.5) and adjust the loading to the specified wheel pressure (see 4.1.6). Fasten the test specimen on the turntable. Lower the abrading heads carefully until the wheels rest squarely on the test film. Place the vacuum pickup nozzle in position and adjust it to a distance of 1/32-inch above the test film. Set the counter to

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"zero". Set the suction regulator (variac) to approximately 50 points on the dial. The setting may be increased to 90 if more effective removal of the abrasings appear necessary. Start the vacuum pickup and then the motor of the abraser. Continue the abrasion until the specified number of wear cycles have been registered on the counter or the film has worn through to the test plate (see 4.1.1). Record the number of wear cycles registered. If the index of wear is required, remove any loose abrasings remaining on the test specimen by light brushing and reweigh the test specimen. Repeat the operation on each of the remaining test specimens. Calculate and report the average of the results obtained by whichever of the following ways is required in the product specification.

4.3 Calculation and reporting of results.

4.3.1 Wear index. Compute the wear index of each test specimen using the following equation:

$$\text{Wear index} = \frac{(A - B) 1000}{C}$$

where: A = Weight of test specimen before abrasion, milligrams.
 B = Weight of test specimen after abrasion, milligrams.
 C = Number of cycles of abrasion recorded.

Report the average of the calculated indices as the wear index of the material.

4.3.2 Wear cycles. From the number of cycles recorded when the test film was abraded, through to the test plate, calculate the average number of cycles of abrasion and report this number as the wear cycles of the material.

5. NOTES

5.1 The abraser and its accessories are manufactured by the Taber Instrument Company, North Tonawanda, N.Y.

6. PRECISION

6.1 No data.

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ABRASION RESISTANCE (JET ABRADER)

1. SCOPE

1.1 This method covers a procedure for measuring the abrasion resistance of organic coating materials in terms of the time required for a controlled jet of fine abrasive particles to abrade through the coating to the substrate. The method is also applicable to film or tape when these are bonded or taped to a suitable rigid substrate.

2. APPARATUS

2.1 Jet abrader.

2.1.1 The jet abrader shall consist of a suitable, lighted enclosure having a transparent top and a transparent front door, in which are mounted a specimen support table and a jet nozzle assembly equipped with a means for accurately setting nozzle angle and the nozzle-to-coating distance. The test enclosure shall be equipped with a vacuum exhaust to remove spent abrasive and coating debris. The abrasive powder shall be stored in a cylindrical chamber atop a vibrator. The abrasive storage chamber shall have a row of holes (0.022-inch in diameter) in its bottom through which the abrasive will sift, under the impetus of vibration, into a lower tubular compartment from which the particles are swept toward the nozzle by a stream of carbon dioxide gas under closely controlled pressure. A pressure-equalizing standpipe shall connect the upper and lower compartments of the storage chamber. Close control of abrasive flow shall be provided by controlling the voltage which controls the amplitude of vibration. Flexible, pressure tubing shall interconnect the various parts of the system. A solenoid valve between the abrasive storage chamber and the nozzle shall provide positive on-off control of the abrasive flow. A timer shall permit reading to the nearest half-second. The vibrator, solenoid, and timer shall be operated from a single on-off switch.

2.1.2 A tube for collecting and weighing the quantity of abrasive leaving the nozzle per unit time shall be provided. The tube shall be made of brass with threaded end collars, to hold a rubber diaphragm with a pinhole for accommodating the nozzle tip at one end, and a diaphragm of filter paper at the other end to trap the abrasive while permitting escape of gas.

2.2 Abrasive. The abrasive shall be an aluminum oxide powder having an average particle size of 27 microns and containing only a small proportion of particles smaller than 10 microns or larger than 53 microns (270 mesh).^{1/} The abrasive shall not be reused.

2.3 Reference panel. A reference panel for calibration purposes shall be prepared by bonding a uniform film of known thickness to a metal substrate,

^{1/} Equivalent to S.S. White, Airbrasive Cutting Powder No. 1.

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using a negligibly thin layer of a suitable adhesive [e.g., a 5 percent solution of polyvinyl butyral resin in ethyl alcohol (95 percent)]. The reference panel may be standardized in terms of a specified standard panel when available and indicated in the applicable specification.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Calibration of apparatus.

4.1.1 Standard operating conditions. The standard operating conditions shall be as follows, unless otherwise specified for coatings of unusually low or extremely high abrasion resistance:

Nozzle-to-coating distance	0.40 inch
Abrading angle	90 degrees
Gas pressure	50 p.s.i.
Abrasive flow	5 grams/minute (approx)
Temperature	23° \pm 1.1° C (73.5° \pm 2° F)
Relative humidity	50 \pm 4 percent

Control of temperature and humidity, while desirable for maximum accuracy and precision, may be waived under ordinary room conditions when maximum accuracy is not needed.

4.2 Adjustment of abrasive flow.

4.2.1 For initial adjustment of abrasive flow in the absence of a standard reference panel, the amplitude of vibration as controlled by the voltage to the vibrator shall be adjusted to yield an abrasive flow of approximately 5 grams per minute as measured by collecting and weighing the powder in the tube described in 2.1.2. Before measuring abrasive flow, the apparatus should be turned on for 30 seconds to ensure clear lines and equilibrium conditions.

4.2.2 When a previously calibrated reference or standard panel is available, the apparatus may be quickly calibrated by adjusting the voltage to the vibrator to yield the specified abrasion time on the reference panel.

4.3 Performance of test.

4.3.1 Place (or clamp) the test panel on the specimen support table. Adjust the gas pressure, nozzle-to-coating distance, and the abrading angle for the standard operating conditions specified in 4.1, unless otherwise indicated. (The apparatus shall have been previously calibrated against the reference panel or weighing tube, as indicated in 4.2.1). Start the vacuum exhaust. Turn on

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the operating switch which simultaneously starts the vibrator, opens the solenoid valve, and starts the timer. Continue the test until the first show of bare substrate signals the end point, as evidenced by an abrupt change in color gloss of pinhole-sized abrasion pit. Record the time required to abrade through the coating to the substrate.

4.3.2 Make five replicate tests per panel, except that if the first three tests agree very closely, no further replicates are necessary.

4.3.3 Make periodic calibration checks against the reference panel at intervals as necessary or desirable to assure reliable results.

4.3.4 Measure the thickness of the coating by a suitable means.

4.4 Reporting of results:

4.4.1 Report the average abrasion time, the number of replicates, the thickness of the coating, and the average abrasion time per unit thickness obtained on the test specimen. Report also the thickness, abrasion time, and abrasion time per unit thickness for the reference panel employed.

5. NOTES

5.1 None.

6. PRECISION

6.1 The reproducibility of measurements on a uniform reference panel with a abrasion time of the order of 50 seconds should agree within ± 1.0 second. Variations on the test specimen, if greater than 3 percent, are usually indicative of non-uniform thickness in the test specimen.

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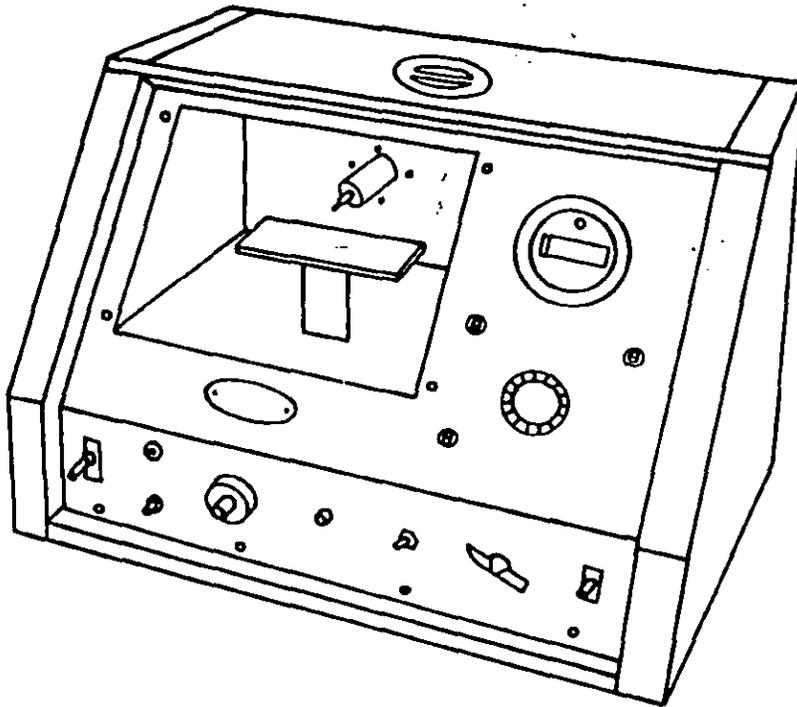


FIGURE 1. Jet abrader.

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METHOD 6201
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HUMIDITY TEST

1. SCOPE

1.1 This method covers a general procedure for exposing films or systems of films of organic coating materials to a moisture-saturated atmosphere at controlled temperature and with continuous condensation on the test films.

1.2 Supplementary information. Definite instructions relative to the following pertinent details shall be given in the product specification:

1.2.1 Substrate.

1.2.1.1 Nature of substrate.

1.2.1.2 Size of substrate.

1.2.2 Method of coating application.

1.2.3 Number of coats to apply.

1.2.4 Manner of drying.

1.2.4.1 Time of aging before testing.

1.2.5 Duration of exposure

1.2.6 Temperature of exposure.

1.2.7 Relative humidity of exposure.

1.2.8 Is air in cabinet to be changed.

1.2.8.1 Rate of air exchange.

1.2.9 When are exposed paints to be inspected.

1.2.9.1 How are exposed paints to be inspected.

1.2.10 List characteristics.

2. REAGENTS

2.1 None required.

3. APPARATUS

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3.1 Humidity cabinet.^{1/} The humidity cabinet shall be of suitable size, construction and design to satisfy the conditions of the test described herein. It shall be equipped with rods or notched supports for properly suspending the test specimens. The cabinet shall be properly vented and so designed that the drops of water, which accumulate on its ceiling or cover will not fall on the test specimens, and no condensate shall drip from one test specimen onto another.

3.2 Temperature indicating or recording devices. Suitable partial immersion thermometers, temperature indicating or continuous recording devices with subdivisions of 0.5° C (0.9° F) and a scale error of not more than 0.5° C (0.9° F) shall be used for measuring the temperature within the cabinet.

3.3 Heating elements. Two thermostatically controlled heating elements of sufficient capacity to satisfactorily heat the water used to humidify the air within the cabinet. One of the elements shall serve as an intermittent heater, the other as an auxiliary heater. The intermittent heater shall be controlled by a thermostat located near the test panels, and the auxiliary heater by a thermostat located in the water tank.

3.4 Water supply. Only distilled water shall be used in the cabinet. It shall be kept in a suitable tank preferably located at one end or in one corner on the floor of the cabinet. The water shall be kept at a constant level in the tank by means of a suitable float-actuated device. If a constant source of distilled water is not available, a glass water storage bottle placed above the water tank in the cabinet, with a glass syphon tube and hydrostatic leveling tube, may be used.

3.5 Air supply. The air supply shall be capable of delivering sufficient air to maintain a constant flow of air through the cabinet when and as specified (see 1.2.8). The air shall be free from industrial fumes such as sulfur dioxide. Any oil or foreign particles from the air compressor shall be removed by passing the air through a tower lightly packed with asbestos or glass wool, before admitting it to the cabinet.

3.6 Air metering device. A suitable air metering device for indicating or continuously recording the air flow.

3.7 Maintenance of equipment.

3.7.1 Care of air supply. The tower packed with asbestos or glass wool for removing oil and foreign matter from the air supply shall be replaced or cleaned whenever necessary.

3.7.2 Care of humidity cabinet. The cabinet shall be cleaned and the distilled water replaced at least once every 60 days to remove any contamination which may be introduced from the test specimens.

^{1/} A cabinet as that described in appendix to ASTM D 1748 or, preferably, appendix to ASTM D 2247 and modified to the condition of test, is suggested.

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3.8 Preparation of test specimens. The test specimens shall be prepared as specified in the product specification (see 1.2.1 through 1.2.4). The back and edges of each test panel shall be given a protective coating of varnish, enamel or melted wax, whichever is applicable.

4. PROCEDURE

4.1 Using glass or plastic hooks or waxed string suspend the test specimens vertically in the cabinet. Arrange the test specimens so that they will be equally spaced from each other and none will be close to and immediately over the water tank. (Generally, condensation will not be noted on the specimens unless they are well above the tank.) When necessary, suspend blank stainless steel panels of the same size and shape from all unoccupied notches or rods so that the cabinet will always be filled with panels at equal distance from each other during the exposure. Adjust the thermostat of the intermittent heater to $49^{\circ} \pm 1^{\circ} \text{C}$ ($120 \pm 1.8^{\circ} \text{F}$) or other temperature (see 1.2.6) specified for the exposure. Set the thermostatic control of the auxiliary heater to break contact a few degrees above the setting of the intermittent thermostat. Adjust the water to the proper level in the tank and start the air bubbling through the water at a rate previously determined to be sufficient to maintain a relative humidity of 93 to 100 or other specified percentage (see 1.2.5 through 1.2.8.1). During the exposure observe and record the cabinet temperatures and rate of air flow at least once during each 24-hour period. Also make periodic observations to determine whether condensation is occurring on the test specimens, since it is of prime importance that condensation be continuous throughout the exposure. At the end of the exposure period (see 1.2.5) examine the test coatings immediately or after the stipulated recovery period whichever is specified (see 1.2.9) and determine whether the test coatings have met the requirements of the product specification (see 1.2.10).

5. NOTES:

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 6221
January 24, 1986

FLEXIBILITY

1. SCOPE

1.1 This method covers a general procedure for determining the flexibility (elasticity) of single films or systems of films of organic coating materials.

2. REAGENTS

2.1 None required.

3. APPARATUS

3.1 Test panels. The test panels shall be steel (method 2011), tinplate (method 2012), aluminum alloy (method 2013) or other material as specified.

3.2 Mandrels. A series of smooth, straight steel rods, ranging from 1/8- to 1-inch in diameter and held firmly by suitable supports.

3.3 Supplementary information. Definite instructions relative to the following pertinent details shall be given in the product specification.

3.3.1 Nature of substrate (see 3.1).

3.3.1.1 Size of substrate.

3.3.1.2 Preparation of substrate.

3.3.2 Method of coating application.

3.3.3 Thickness of coating applied.

3.3.4 Number of coats.

3.3.5 Manner of drying coating.

3.3.6 Temperature of conditions when air dried.

3.3.7 Humidity of conditions when air dried.

3.3.8 Time of drying.

3.3.9 Temperature of conditions baked.

3.3.10 Time of baking.

4. PROCEDURE

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4.1 Apply a film of the coating material, at the required thickness to the test panel or coating system by drawing down with a film applicator (method 2161 or method 2162) or by spraying (method 2131), and dry it in accordance with the instructions given in the product specification (see 3.3.5). Unless otherwise specified, condition the dried coating at $23^{\circ} \pm 1.1^{\circ} \text{ C}$ ($73.4^{\circ} \pm 2^{\circ} \text{ F}$) and a relative humidity of 50 ± 4 percent for at least one-half hour. While at this temperature, place the test panel, with the coated side uppermost on a mandrel of the specified diameter, at a point equally distant from the top and bottom edges of the panel, and bend the panel double in approximately 1 second. Examine the film at the bend under a magnification of 7 diameters, using diffused light having a color temperature of $6,500^{\circ} \text{ K}$ and an intensity of about 165 foot-candles. (For those not equipped with a standard illuminant, viewing the test coating at a north window illuminated by a fairly light overcast sky will approximate these conditions.) Cracks occurring at either end and extending no more than one-quarter inch shall be disregarded.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 6241.1
January 24, 1986

INFRARED REFLECTANCE FROM SPECTROPHOTOMETRIC DATA

1. SCOPE

1.1 This method is designed to provide a measure of infrared reflectance. The effective infrared reflectance of a specimen for a specified wavelength interval and for specified geometric conditions is dependent upon the spectral composition of the incident energy, the spectral sensitivity of the receiver, and the spectral reflectance of the specimen. This method shall be the basic method for the measurement of infrared reflectance and in case of dispute, the results obtained under this method shall govern.

2. APPARATUS

2.1 The apparatus shall consist of a spectrophotometer by means of which the spectral characteristics of the test specimen may be determined within the applicable limits of the infrared spectrum. Such apparatus shall be of the photoelectric type, and shall be suitable for determinations throughout the applicable wavelength interval. The apparatus shall be designed to minimize the effect of stray energy. This may be done by the introduction of stray-energy filters or the use of double dispersion prisms (see 5.1). The direction of irradiation shall be nearly perpendicular to the irradiated face of the test specimen and viewing shall be hemispherical or the converse. The slit widths of the monochromator shall be sufficiently narrow throughout the applicable infrared spectrum to insure that the spectral characteristics of the specimen are not appreciably altered when narrower slit widths are used.

2.2 Standard.

2.2.1 The standard shall be smoked or pressed MgO or pressed BaSO₄ prepared according to Recommended Practice for Preparation of Reference White Reflectance Standards, ASTM E 259.

3. REAGENTS

3.1 None Required.

4. PROCEDURE

4.1 Irradiating and viewing.

4.1.1 If glossy samples are to be measured, the specular components of reflected energy shall be excluded wherever possible.

4.2 The area of the test specimen viewed shall be sufficiently large to be representative of the test specimen, textured specimens in particular.

4.3 Calibrations.

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4.3.1 The calibration of the photometer scale, shall be carefully checked at reasonable time intervals in a manner to insure accuracy over the entire range. Glass filters may be used for this purpose.

4.3.2 The wavelength calibration shall be checked frequently to insure accuracy. Spectrum lamps may be used for this purpose.

4.4 Performance of test.

4.4.1 Preparation of test specimen. The test specimen shall be prepared as indicated in the product specification.

4.4.2 Determination of reflectance. Determine the spectral reflectance relative to the reference standard at the wavelength given in tables I and II or at sufficiently close intervals so that interpolation will permit accurate determination of the spectral reflectance at these wavelengths.

4.5 Calculation of reflectance from spectrophotometric data.

4.5.1 Tabulate spectral reflectance data for the specimen for the 30 wavelengths listed in table I and the 10 wavelengths in table II.

4.5.2 Calculate the average of the 30 spectral reflectance values in table I and the 10 values in table II.

TABLE I. Thirty wavelengths at which spectral reflectance values are to be obtained in red and near infrared.

No.	Wave- lengths microm- eter	No.	Wave- lengths microm- eter
1	0.6975	16	0.7742
2	.7077	17	.7787
3	.7144	18	.7831
4	.7200	19	.7878
5	.7253	20	.7925
6	.7301	21	.7973
7	.7347	22	.8022
8	.7392	23	.8071
9	.7436	24	.8122
10	.7479	25	.8171
11	.7522	26	.8232
12	.7566	27	.8292
13	.7609	28	.8357
14	.7653	29	.8434
15	.7697	30	.8542

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4.6 Report.

4.6.1 Report the average infrared reflectance of the specimen for the wavelength intervals specified in tables I and II.

4.6.2 If required, report the spectral reflectance of the test specimen either as a plotted curve or as tabulated data.

4.6.3 Report the method of specimen preparation, if any.

4.6.4 Report the reference standard and spectrophotometer used for the measurements.

5. NOTES

5.1 The General Electric (Hardy) recording spectrophotometer, the Beckman Model DK-2A spectrophotometer, and Cary Model 14 spectrophotometer may be used. The specular component should be excluded. The Beckman DU spectrophotometer does not use hemispherical viewing and, therefore, does not meet the geometric requirements of the method, but it may be used if no other instrument is available

TABLE II. Ten wavelengths at which spectral reflectance values are to be obtained in the infrared.

No.	Wave-lengths micrometer	No.	Wave-lengths micrometer
1	0.929	6	0.998
2	.945	7	1.012
3	.959	8	1.030
4	.972	9	1.053
5	.985	10	1.099

6. PRECISION

6.1 No data.

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METHOD 6242.1
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INFRARED REFLECTANCE FROM REFLECTOMETER

1. SCOPE

1.1 The reflectometer test method is designed to provide a simple photoelectric means for evaluating the infrared reflectance of a specimen by comparison with a spectrophotometrically calibrated standard.

1.2 This method is supplementary to the spectrophotometric method 6241. When results obtained by those two methods disagree, the results from the spectrophotometric method will govern.

2. APPARATUS

2.1 The apparatus shall provide a means for irradiating the test specimen and of indicating the radiation reflected by the test specimen. The spectral irradiation from the source and the spectral sensitivity of the receiver shall be appropriate to cover the spectral band in which the measurements are made.

2.1.1 The geometry of irradiating and viewing measured from the perpendicular to the irradiated face of the test specimen shall be as follows:

Irradiating	Viewing
0° Hemispherical	Hemispherical 0°

The area irradiated shall be sufficiently large to be representative of the test specimen. Other geometries of irradiating and viewing may be employed provided the results obtained are comparable to the results obtained by specified geometries. Any reflectometer which yields results comparable to those obtained from the spectrophotometric method may be used.

2.1.2 Reflectometer for the spectral region covered by the 30 wavelengths listed in table I, method 6241. Although it does not have the preferred geometry, a Hunter Multipurpose Reflectometer using a Wratten 88 or 89B filter and photovoltaic-type photocell with standards described in 2.2 may be used. Another photometer employing an S-1 phototube has been found to be satisfactory when employed with a suitable source and filter combination. The source shall consist of a tungsten lamp operating at a color temperature between 2,800° and 2,950° Kelvin. An interference-type filter having a peak transmission at 0.79 micrometer in combination with a Wratten 89B filter is placed in front of the phototube which provides hemispherical irradiation and perpendicular viewing.

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2.1.3 Reflectometer for the spectral region covered by the 10 wavelengths listed in table II of method 6241. The photometer described in 2.1.2, employing the S-1 phototube and tungsten light source together with a suitable filter is satisfactory. A glass filter having the following transmission characteristics has been found to be adequate: 3 ± 1 percent at 0.9 micrometer, 29 ± 3 percent at 1.0 micrometer. A Corning glass filter No. 2540 can be obtained to meet these requirements.

2.2 Standards.

2.2.1 Standards shall be prepared by painting or otherwise finishing metal or wood panels in order to obtain a suitable range of infrared reflectance values. The gloss and texture properties of the standards shall be nearly the same as those of the test specimens. The standards shall be calibrated according to method 6241. The standards shall be replaced when they show signs of deterioration.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Calibrations.

4.1.1 The photometer scale shall be carefully calibrated for linearity over the range of reflectances to be measured.

4.1.2 The standards shall be recalibrated at reasonable time intervals.

4.2 Performance of test.

4.2.1 Preparation of test specimen. The test specimen shall be prepared as described in the product specification.

4.2.2 Determination of reflectance. The instrument readings for the test specimen shall be compared with those of the standards and the reflectance value of the test specimen determined. Linear interpolation is permissible if the instrument response is linear or if the scale interval between reflectance standards is small. Upper and lower limit standards may be used for a go-no-go type of measurement.

4.3 Report. The report shall include the following:

4.3.1 The reflectance values of the specimen for the two wavelength regions and their acceptability as required by the governing specification.

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4.3.2 The instruments and standards used shall be designated or described.

4.3.3 Any unusual condition of the test specimen or situation under which the test was conducted. Any deviations from the specifications shall be reported.

5. NOTES

5.1 Other instruments meeting the prescribed irradiating and viewing geometry of the method are: (1) the Hunter Associates Laboratory Color Difference Meter D25P, and (2) the Zeiss Elrepho Photoelectric Reflectance Photometer. These instruments have to be modified to provide detector and filter for measurements according to the method.

5.2 Measurements made in the spectral region covered by 2.1.2 are useful when data applicable to infrared photography are required. Measurements covered in 2.1.3 are intended to be relatable to infrared image tubes.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 6244
January 24, 1986

INSTRUMENTAL MEASUREMENTS OF PHOTOMETRIC PERFORMANCE
OF RETROFLECTIVE BEADS

1. SCOPE

1.1 This method determines the instrumental photometric performance of retro-reflective beads by measuring the luminance factor of a panel prepared by dropping the beads (glass spheres) onto a standard white binder.

2. DEFINITIONS

2.1 Retroreflective bead. A spherical bead made of glass which by refraction or reflection produces the phenomenon of retroreflection.

2.2 Geometric terms. Definitions are covered in section 2.2 of Fed. Test Method Std. 370.

3. APPARATUS

3.1 Photometric assembly. A photoreceptor, light projector source, goniometer sample holder, and a 50-ft. photometric range in accordance with the requirements of section 5 of Fed. Test Method Std. 370 shall be used.

3.2 Motor driven blade film applicator. The applicator shall conform to the description of method D in ASTM D 823.

3.3 Bead dropping device.^{1/} The bead dropping device (figure 1) shall have a series of "V" shaped hoppers tied together mechanically to permit simultaneous opening when triggered. The bead-retaining mechanism shall be attached to the top of a box having inside dimensions of 140 by 380 by 610 mm high. To dispense the beads uniformly, an internal screen shall be provided approximately 25 mm below the bead-retaining mechanism. The base of the structure shall be open so that the paint film can be inserted. Mechanical means shall be provided to lower the structure onto the test plate and to tilt the structure so that the base may be cleaned.

4. STANDARD

4.1 Standard white binder. The standard white binder shall be prepared with the following ingredients:

4.1.1 Barium sulfate - certified laboratory grade.

4.1.2 Polyvinyl alcohol^{2/}.

^{1/} Mechanical drawings of the bead dropper can be obtained from GSA-FSS, Paints Branch (FREC-P) Washington, DC 20406.

^{2/} Gelvatol 20-30 of the Monsanto Company has been found to be satisfactory.

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Hydrolysis, percent - 88.7 to 85.5.
 Viscosity, cps, 10 percent in methanol - 4 to 6*
 Specific gravity, 25/25° C - 1.23.
 Pounds per gallon, average - 10.3.
 * 4 percent in distilled water.

4.1.3 Ethanol.

4.1.4 Distilled water.

4.2 Formula.

Polyvinyl alcohol, (1 percent solution in distilled water), percent by weight.	42.9
Barium sulfate, percent by weight.	28.6
Ethanol, percent by weight.	28.5

4.2.1 Preparation. The polyvinyl alcohol shall be dissolved in distilled water at a temperature of 50° \pm 5° C by hand mixing or mechanical stirring. The barium sulfate shall be added slowly to the polyvinyl alcohol solution at a temperature of 25° \pm 2° C and thoroughly dispersed by hand mixing or mechanical stirring. The ethanol shall be added to the dispersion to release trapped air bubbles.

4.3 Diffuse reflectance.

4.3.1 Apply the standard white binder at a wet film thickness of 0.38 \pm 0.01 mm (0.015 in)^{3/} to a glass panel by means of a mechanical film applicator. Allow to cure for 24 hours at 25 \pm 1° C (77 \pm 2° F) and 50 \pm 5 percent relative humidity.

4.3.2 The standard white binder shall have a diffuse reflectance of 91.5 \pm 0.1 percent as measured by ASTM E 308.

5. PREPARATION OF TEST PANELS

5.1 Apply the standard white binder at a wet film thickness of 0.38 \pm 0.01 mm (0.015 in) to glass panels measuring 165 by 432 by 6.5 mm by means of a mechanical film applicator. The type and amount of beads shall be specified in the product specifications and shall be distributed uniformly in the closed "V" shaped hoppers. The glass plate shall be inserted under the structure, and the structure shall be lowered onto the plate. Exactly 15 seconds after the draw-

^{3/} To obtain the wet film thickness specified use a blade with a gap clearance of 0.508 mm (0.020 in).

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down, the bead retaining device shall be mechanically triggered, allowing the beads to drop uniformly onto the binder. The panels shall be cured to 24 hours at standard conditions. (Note: Beaded panels are fragile and should be handled with care).

6. PROCEDURE

6.1 The luminance factor shall be determined in accordance with procedure I of Fed. Test Method Std. 370 and with the following test requirements:

Observation angle.	1.33°
Entrance angle.	75° and 88°
Presentation angle.	0°
Viewing angle.	73.67° and 86.67°
Test distance between sample and photoreceptor.	50 ft.
Photoreceptor angular aperture.	10 minutes of arc
Angular aperture of the light projector.	10 minutes of arc

6.2 Goniometer calibration. The goniometer shall be calibrated at the 0° entrance angle position in the vertical and horizontal planes of the test sample. All measurements shall be made relative to this point and shall be checked each time the goniometer base or light projector is moved.

6.3 Photo measurements. The same instrument with the same apertures and field of view shall be used to measure both R_1 and R_2 . Therefore, the photoreceptor need not be calibrated.

7. CALCULATION

7.1 Calculate the mean value for the readings on each panel tested.

7.2 Calculate the luminance factor by means of the following equation:

$$LF = \frac{(R_1 - R_s) (D')^2}{R_2 A}$$

- Where:
- R_1 - Reading of the photoreceptor while measuring the illuminance at the observation position.
 - R_2 - Reading of the photoreceptor while measuring the normal illuminance at the face of the retroreflective sample.
 - R_s - Reading of the amount of stray light measured by replacing the sample with a black surface of the same shape and area.
 - D' - Distance between the center of the photoreceptor entrance aperture and the reference center.
 - A - Surface area of the sample.
 - B - Entrance angle.
 - C - Viewing angle.

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8. PRECISION

8.1 Results obtained on the same panel and the same instrument should be repeatable to within 0.5 percent.

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METHOD 6251
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LACQUER LIFTING TEST

1. SCOPE

1.1 This method covers a general procedure for determining the resistance to deformation of a dried film of organic coating material when a coat of lacquer is applied to it.

1.2 Supplementary information. Definite instructions relative to the following pertinent details shall be given in the product specification.

- 1.2.1 Nature of substrate (see 2.1).
- 1.2.2 Method of application.
- 1.2.3 Thickness of coating.
- 1.2.4 Manner of drying applied coating.
- 1.2.5 Time of drying applied coating.
- 1.2.6 Method of its application.
- 1.2.7 Thickness of its application.
- 1.2.8 When to inspect.
- 1.2.9 How to inspect.

2. APPARATUS

2.1 The test panels shall be steel (method 2011), aluminum alloy (method 2013) or other material as specified.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Apply film of the material to be tested, at the required thickness, to the test panels by drawing down with a film applicator (method 2161 or method 2162), or by spraying (method 2131), or by brushing (method 2141) and dry in accordance with the instructions given in the product specification. After the stipulated drying period or periods, apply the test lacquer as specified. Examine each test specimen either immediately or after drying for the period specified for lifting, embrittlement, or other surface irregularities.

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METHOD 6251
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5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 6252.1
January 24, 1986

SELF-LIFTING TEST

1. SCOPE

1.1 This method covers a general procedure for determining the ability of dried films of organic coating materials to resist deformation upon application of an additional coat of the same material.

1.2 Supplementary information. Definite instructions relative to the following pertinent details shall be given in the product specification.

1.2.1 Nature of substrate (see 2.1).

1.2.2 Method of coating application.

1.2.3 Thickness of coating applied.

1.2.4 Manner of drying coating.

1.2.5 Time of drying coating.

1.2.6 Method of applying subsequent coat or coats.

1.2.7 Thickness of applying subsequent coat or coats.

1.2.8 When to inspect.

1.2.9 How to inspect.

2. APPARATUS

2.1 The test panels shall be steel (method 2011), tin (method 2012), glass (method 2021), concrete (method 2051), or other material, as specified.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Apply films of the coating material, at the required thickness, to the test panels by drawing down with a film applicator (method 2161 or method 2162) by spraying (method 2131), or brushing (method 2141), or by dipping (ASTM D 823, method B) and dry in accordance with the instructions given in the product specification. After the films to be tested have been baked or air-dried for the specified time intervals, apply a second coat of the material as specified. Examine each test film either immediately or after drying for the specified period, for film irregularities such as blisters and pinholes, and for smoothness and uniformity by viewing them by reflected light.

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METHOD 6261.1
January 24, 1986

PRIMER ABSORPTION AND TOPCOAT HOLDOUT

1. SCOPE

1.1 The general procedure given herein provides a means of determining the holdout characteristics of top coats or the absorption characteristics of primers. By using a standard primer the top coat can be evaluated and by using a standard top coat a primer can be evaluated.

2. APPARATUS

2.1 Panel. Gypsum board (sheet rock) meeting the requirements of SS-L-30.

2.2 Brush. Four-inch wall brush.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Panels. Use the panel described in method 2081 unless otherwise specified.

4.2 Primer absorption.

4.2.1 Coat the panel with the primer under test with one coat at 450 square feet per gallon using a 4-inch wall brush. Allow to dry for 24 hours.

4.2.2 Coat the primed panel with a TT-E-508 type paint at approximately 500 square feet per gallon. Allow to dry 72 hours and visually check the surface for uniformity of gloss.

4.3 Holdout properties.

4.3.1 Coat the panel with one coat of TT-S-179 sealer at approximately 400 square feet per gallon using a 4-inch wall brush. Allow to dry 24 hours minimum.

4.3.2 Apply the material under test at approximately 500 square feet per gallon with a 4-inch wall brush. Allow to dry 72 hours. Examine visually for uniformity of gloss.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 6252.1
January 24, 1986

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

FED. TEST METHOD STD. NO. 141C

METHOD 6271.2
 January 24, 1986

MILDEW RESISTANCE

1. SCOPE

1.1 This method determines the resistance of films of organic coatings to the degradative action of fungus growth.

2. APPARATUS

2.1 Autoclave. An autoclave capable of maintaining a steam pressure of 15 pounds per square inch at an exhaust temperature of 121° C, for sterilizing the culture medium and glassware.

2.2 Sterile room. A dust-free room in which to inoculate the test specimens. Sterilizing lamps, antiseptic spray, and air filtered under pressure may be used to maintain aseptic conditions.

2.3 Incubator. An incubator (cabinet or chamber) capable of maintaining a temperature of 28° to 30° C and a relative humidity of 85 to 90 percent.

3. REAGENTS

3.1 Culture medium. The culture medium shall have the following composition:

Sodium nitrate (NaNO ₃)	3.0 g
Dipotassium hydrogen phosphate (K ₂ HPO ₄)	1.0 g
Magnesium sulfate heptahydrate (MgSO ₄ · 7H ₂ O)	0.5 g
Potassium chloride (KCl)	0.25 g
Sucrose	30.0 g
Agar	15.0 g
Distilled water to make	1000 ml

The pH shall be between 5.5 and 6.5. If otherwise, adjust to that range with HCl or NaOH. After mixing, the above ingredients shall be sterilized in an autoclave for 15 minutes at 15 psi and 121° C.

3.2 Test organism. Unless otherwise specified the test organism (see 5.1) shall be one of the following:

	American type culture collection number	Quarter- master culture collection number
<i>Aspergillus niger</i>	6275	QM 458
<i>Aspergillus oryzae</i>	10196	QM 1273
<i>Aureobasidium pullulans</i>	9348	QM 3090

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3.3 Maintaining stock cultures. Cultures of fungi shall be maintained separately on an appropriate culture medium such as potato-dextrose agar or that specified in 3.1, and promptly renewed when there is evidence of contamination. The stock cultures may be kept for not more than 4 months under refrigeration at temperatures between 3 and 10° C. Subcultures incubated at between 28° and 30° C for 10 to 21 days shall be used in preparing the inoculum.

4. PROCEDURE

The test procedure must be conducted under aseptic conditions at all times.

4.1 The following information shall be given in the product specification:

4.1.1 The test organism to be used.

4.1.2 Whether or not the test specimens are to be leached with water before inoculation.

4.1.3 The length of the incubation period.

4.1.4 Whether the criterion for acceptance will be (a) the total absence of fungus growth on the test specimen or (b) the permissible extent of fungus growth on the test specimen.

4.2 Preparation of test specimens. Unless otherwise specified, one normal brush coat of the coating material shall be applied to each side of a sheet of NO. 30 Whatman filter paper or equal; the sheet shall be brushed uniformly in all directions to ensure a uniform impregnation of the paper. The first side shall be allowed to become tack free before the reverse side is coated. After the coating has dried for 48 hours, the sheet shall be cut into 32 mm squares. Guide lines shall be marked with black waterproof ink on one side of each square 3 mm from each edge.

4.3 Leaching of test specimens. When leaching of the test specimens is required (see 4.1.2), the following procedure will be followed: submerge the coated sheet of filter paper in a container of water of such size and shape that all surfaces will have free access to the water. Supply a continuous flow of distilled water at 27° to 30° C to the bottom of the container at a rate of five changes per 24 hours. The ratio of the specimen to the water shall be not less than 1 to 100. After 24 hours of submersion, remove the paper and allow it to air dry. When dry, cut the paper into squares and mark them as specified in 4.2.

4.4 Preparation of culture vessels. Pour freshly prepared culture medium into 10 cm Petri dishes, approximately 30 ml per dish, and allow it to harden undisturbed, taking care to avoid bubbles in the medium.

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4.5 Preparation of inoculum. To a subculture of the specified test organism in a ripe fruiting condition, add 10 ml of sterile distilled water containing approximately 0.05 percent of a nontoxic wetting agent (such as dioctyl sodium sulfosuccinate). Force the spores into the suspension by shaking the subculture to dislodge the spores, and dilute to 100 ml with sterile water. A fresh spore suspension must be prepared each day.

4.6 Inoculation. Place the square specimen, with guidelines showing, firmly on the center of the solidified culture medium contained in the Petri dish. With a sterile pipette or other suitable means, distribute 1.0 ml of inoculum over the surface of the specimen and surrounding culture medium. A minimum of three squares from each test specimen shall be inoculated in separate Petri dishes.

4.7 Incubation. The period of incubation shall be 7 days or other specified period (see 4.1.3) at a temperature between 28° and 30° C and between 85 and 90 percent relative humidity.

4.8 Viability controls. Two Petri dishes containing only solidified culture medium as specified for the test also shall be inoculated with the specified test organism to determine the viability of the inoculum. At the end of the incubation period, the controls should be well covered with growth of the test organism. If this does not occur, the test shall be discarded and repeated.

4.9 Results. Upon completion of the incubation period, the test specimens shall be examined microscopically (at least 18X magnification) to determine if the requirements for fungus resistance specified in the product specification (see 4.1.4) have been met. If the criterion for acceptance is the total absence of fungus growth on the test specimen, evidence of any fungus growth within the area bounded by the guide lines on the specimen square shall constitute failure of the coating material. However, growth on the specimen up to but not crossing the guide lines shall be disregarded.

4.10 Contamination. When contaminating microorganisms (bacteria or fungi other than the test organism) are present on any of the test specimen squares, the test shall be repeated.

5. NOTES

5.1 Test organisms can be purchased from the American Type Culture Collection, 12301 Parklawn Dr., Rockville, MD 20852, or from Culture Collection Fungi (QM), Department of Botany, University of Massachusetts, Amherst, MA 01003.

6. PRECISION

6.1 No data.

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METHOD 6301.2
January 24, 1986

ADHESION (WET) TAPE TEST

1. SCOPE

1.1 This method covers a procedure suitable for establishing acceptability of intercoat and surface adhesion of an organic coating system immersed in water. This procedure may be utilized as a production test.

2. APPARATUS

2.1 Masking tape. The masking tape used shall be 1-inch wide strip of flatback paper tape having an average adhesion of 60 oz/in width and shall be code no. 250 (see 5.1) or equivalent.

2.2 Roller. A 4-1/2-pound rubber covered roller, having a surface Durometer hardness value within the range of 70 to 80, shall be used. The roller shall be approximately 3-1/2 inches in diameter and 1-3/4 inches in width.

3. REAGENTS

3.1 Distilled water.

4. PROCEDURE

4.1 Apply the coating system to the substrate and allow to dry as specified in the product specification. Immerse the test specimen in distilled water for 24 hours. Remove the test specimen from the water and wipe dry with a soft cloth. Within 1 minute after removal from the water make two parallel scratches, one inch apart, through the coating to the metal with a stylus. Immediately apply a 1-inch wide strip of the masking tape with the adhesive side down across the scratches. Press the tape against the surface of the coating by passing the roller across the tape eight times. Remove the tape with one quick motion and examine for damage to the intercoat or surface adhesion.

5. NOTES

5.1 Minnesota Mining Co.

6. PRECISION

6.1 No data.

METHOD 6304.1
January 24, 1986

KNIFE TEST

1. SCOPE

1.1 This method should be limited to testing the brittleness and quality to ribbon when a film is cut with the knife. This method should not be used as a test for adhesion as other methods are more accurate.

2. APPARATUS

2.1 A craftsman's knife with a curved blade such as is illustrated in the Stores Stock Catalog, Federal Supply Service, as item 5110-00-596-8098.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Apply and dry the film of the coating material to be tested as specified in the product specification. Test for brittleness, toughness and tendency to ribbon by cutting a narrow ribbon of the coating from the test panel with the standard knife while holding the blade at an angle of approximately 30 degrees to the panel. Determine if the cut film conforms to the specified requirements.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 6321
January 24, 1986

SANDING CHARACTERISTICS

1. SCOPE

1.1 This method should be used to determine the quality of coating to be sanded for any purpose.

2. APPARATUS

2.1 Panel preparation. The panels shall be cut from synthetic resin bonded straight grain, 5-ply walnut veneer (faces and backs) having no sizing or other surface pretreatment (see 5.1). They shall be approximately 5/16-inch thick. No side of the panels shall be shorter than 4 inches and the minimum area shall be 32 square inches. The panel shall have a moisture content of not less than 6 percent and not more than 8 percent at the time of testing. Prior to finishing, the panel shall be thoroughly sanded smooth with 5/0 or finer sandpaper. Surfaces shall be thoroughly cleaned immediately before application of the coating under test.

3. REAGENTS

3.1 None required.

4. PROCEDURE

4.1 Application of coating or coating system. The specification shall state:

- (1) The coating that should be used as a first coat.
- (2) The coating required as a second coat, if one is desired.
- (3) The coating required as a third or subsequent coats, if desired.
- (4) The application procedure by reference to the correct method in this Federal Standard.
- (5) The drying time and conditions of drying desired between coats.
- (6) If sanding of a coat is required before application of a subsequent coat.
- (7) Fineness of sandpaper to use if it should be different from 5/0 specified herein.
- (8) Direction and number of cycles coating should be sanded if different from that in 4.2.

4.2 Sanding procedure. Sand test coating with unused No. 5/0 or otherwise specified sandpaper, employing the following technique: attach the finishing paper to one face of a metal or weighted wood sanding block, which shall be approximately 2 inches square, have a total weight of 1,200 \pm 5 g, and have the edges and corner of the bearing face rounded off. The finishing paper should completely cover and fit tightly against the face of the block, so as to avoid the formation of wrinkles or creases in the paper while being used. Any sharp folds or edges in the paper around the edge of the block should be bruised,

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softened, or feathered before using. Place the paper covered end of the block on the coating. Grip the block firmly so that it may be slowly moved back and forth across the coating in a straight line. Care should be taken that no downward pressure is exerted by the hand upon the block as the total vertical force exerted against the coating should be not more than the weight of the block (approximately 2/3 pound per square inch). A completed movement of the block forward and back the full length or width of the panel should be considered one cycle. Move the weight over the coating the full length and width of the panel for a total of 25 cycles, changing the direction of movement lengthwise to crosswise and vice versa of the weight every tenth cycle. Observe the sanded coating for smoothness, and uniformity of appearance. Note whether or not the scuffed-off portion is powder or roll form and if any gumming of the finishing paper has occurred.

5. NOTES

5.1 Hardwood plywood shall conform to Commercial Standard CS-35-61 available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402.

6. PRECISION

6.1 No data.

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Section 7000

ANALYTICAL TESTS OF COATING MATERIALS
AND THEIR INGREDIENTS

METHOD 7014
January 24, 1986

ANALYSIS OF UNSAPONIFIABLE MATTER, OIL ACIDS, AND PHTHALIC
ANHYDRIDE IN ALKYD VEHICLES

1. SCOPE

1.1 This method provides for the simultaneous determination of the three named ingredients. It reduces the total time required for analysis, and allows higher accuracy than other oil acids determinations.

2. APPARATUS

2.1 Erlenmeyer flask, 250 ml, with attached air condenser, both with standard 24/40 ground joints.

2.2 Separatory funnels, 500 ml.

2.3 Vacuum oven.

2.4 Volumetric flask, 1000 ml.

2.5 Ultraviolet spectrophotometer.

3. REAGENTS

3.1 Sodium hydroxide, 50 percent. Dissolve 50 g of sodium hydroxide in 50 ml of water.

3.2 Sodium hydroxide, 0.1 N. Add 4 g of sodium hydroxide to about 900 ml of water and dilute to 1 liter.

3.3 Hydrochloric acid, 0.1 N. Add 9 ml of hydrochloric acid to about 900 ml of water, cool, and dilute to 1 liter. Standardize against freshly standardized sodium hydroxide solution.

3.4 Ethyl alcohol, 95 percent.

4. PROCEDURE

4.1 Unsaponifiable matter. Weigh accurately a 5 g sample of the isolated vehicle into a 250 ml Erlenmeyer flask and dissolve in 10 ml of benzene. Add 50 ml of 95 percent ethyl alcohol, 5 ml of 50 percent sodium hydroxide solution, and 5 ml of water. Mix thoroughly, attach an air condenser, and reflux gently in a water bath for two hours. Cool, transfer to a 500 ml separatory funnel, and dilute to 200 ml with water. Wash the flask with 100 ml of ethyl ether, and transfer to the separatory funnel. Shake the funnel thoroughly, and allow complete separation of the liquid layers. Draw off the lower aqueous layer, and set aside for the oil acids determination. Wash the ether extract with 20 ml

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portions of water until neutral, and add all the washings to the aqueous portion already collected. Filter the ether through a rapid filter paper that is damp with ether into a weighed 250 ml beaker. Place a raised cover glass on the beaker, and allow the ether to evaporate at room temperature (approximately 18 to 24 hours). Finally, dry the sample to constant weight in an oven at 105° C. Cool and weigh the unsaponifiable matter. Dissolve the unsaponifiable matter in 50 ml of 95 percent ethyl alcohol, add a few drops of phenolphthalein indicator solution, and titrate with 0.1 N sodium hydroxide. Calculate the percent unsaponifiable matter from the following formula:

$$\text{Percent unsaponifiable} = \frac{[A - (B \times C \times 0.282)] \times 100}{S \times N}$$

where: A = weight of extracted unsaponifiable matter.
 B = ml of sodium hydroxide used in the titration.
 C = Normality of alkali.
 S = weight of vehicle sample.
 N = vehicle nonvolatile fraction.

4.2 Oil acids. To the water layer and washings collected in the procedure of 4.1 and contained in a separatory funnel, add concentrated hydrochloric acid in one ml portions followed by swirling until a permanent cloud forms, then add 2 additional ml. Add 100 ml of chloroform, and shake vigorously. Upon separation, draw off the lower chloroform layer into another separatory funnel, and extract the aqueous layer once more with 50 ml of chloroform. Combine the two chloroform extracts, and transfer the aqueous layer to a 1 liter volumetric flask to be retained for the phthalic anhydride determination. Wash the combined chloroform twice with 200 ml of water, and retain the water washings in the 1 liter flask. Filter the chloroform through rapid filter paper that is wet with chloroform into a weighed 250 ml beaker. Evaporate the solvent in a water bath at 55° C with the aid of a gentle current of air, removing the beaker as soon as the solvents have evaporated. Dry the beaker containing the oil acids in a vacuum oven at 85° C to constant weight. Calculate the percent oil acids on the nonvolatile vehicle basis. Examine the isolated fatty acids for white crystals that may be isophthalic acid and will cause a high oil-acid yield. If crystals are present, dissolve the oil acids in benzene, filter into a weighed beaker, and dry as before. Weigh and calculate the oil acids on the final weighing only.

4.3 Phthalic anhydride. Dilute the combined aqueous layer and washings retained in paragraph 4.2 to one liter with water, mix thoroughly, and allow to stand a few minutes. Withdraw a 2.00 ml aliquot with a pipet, transfer to a 25 ml volumetric flask, and dilute to the mark with 0.1 N hydrochloric acid. Determine the absorbance at 276 nm with 0.1 N HCl in the reference cell. Apply cell corrections if necessary. If the absorbance falls outside the range of

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0.25 to 0.50, withdraw another aliquot of appropriate size, and obtain a new absorbance reading. Calculate the concentration of phthalic anhydride in the dilution as follows:

$$\text{Concentration (g/liter)} = \frac{\text{absorbance}}{8.77 \times \text{cell length in cm}}$$

Calculate the percent of phthalic anhydride in the nonvolatile vehicle as follows:

$$\text{Percent phthalic anhydride} = \frac{1250 \times \text{concentration (g/liter)}}{\text{nonvolatile sample weight}}$$

(The factor 1250 assumes the use of a 2.00 ml aliquot; if a larger or smaller is used, the factor must be adjusted accordingly.)

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 7016
January 24, 1986

CHLORINE CONTENT OF RESIN VEHICLES

1. SCOPE

1.1 This method describes a quantitative gravimetric procedure for the determination of the total chlorine content of lacquer and enamel vehicles. It may also be substituted for ASTM D 1156 in the analysis of vinyl alkyds for their chlorine content.

2. MATERIALS

2.1 Crucible, of nickel metal, capacity 50 ml, provided with nickel or iron stirring rod.

2.2 Muffle furnace.

3. REAGENTS

3.1 Potassium carbonate, anhydrous, A.C.S reagent grade.

3.2 Silver nitrate solution, 5 percent aqueous.

3.3 Nitric acid, concentrated, specific gravity 1.42.

4. PROCEDURE

4.1 Weigh not more than 1.0 gram of vehicle into a 50 ml nickel crucible. Add powdered, anhydrous potassium carbonate to the crucible until it is approximately one-half filled. Mix the contents thoroughly with the stirring rod, leave the rod in the crucible and dry in an oven for 2 hours at 105° C. Add additional potassium carbonate evenly to the crucible until it is approximately two-thirds filled. Do not stir. Transfer the crucible to a muffle furnace and raise the temperature very slowly to 400° C, then increase temperature rapidly to 800° C and maintain for one hour. Allow the crucible to cool and place in a 600 ml beaker. Add 200 ml of water. Cover and add 35 ml of concentrated nitric acid very slowly from a dropper or pipet, with occasional stirring. Filter the solution into an 800 ml beaker, using paper of medium retention. Transfer and wash with water, scrubbing the crucible and rod if necessary. Add 20 ml of 5 percent silver nitrate solution and stir vigorously until maximum coagulation has occurred. Heat to boiling, cool and filter through a Gooch crucible or fritted glass crucible of medium porosity. Wash with water and dry the crucible for 1 hour at 130° C. Cool in a desiccator and weigh.

4.2 Calculation

4.2.1 Calculate the chlorine content as follows:

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$$\text{Percent Chlorine} = \frac{\text{Weight of precipitate} \times 0.2474 \times 100}{\text{Sample weight} \times \text{nonvolatile fraction}}$$

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 7024
January 24, 1986

PHTHALIC ANHYDRIDE IN LACQUER VEHICLES
(ULTRAVIOLET SPECTROPHOTOMETRIC METHOD)

1. SCOPE

1.1 This method is an adaptation of the spectrophotometric determination of phthalic anhydride to the analysis of nitrocellulose lacquers.

2. APPARATUS

2.1 Spectrophotometer. Suitable for use in the ultraviolet region of the spectrum.

2.2 Flask. A 500 ml, 24/40 glass-stoppered Erlenmeyer flask.

2.3 Filter. Fritted glass crucible of coarse porosity.

2.4 Air condenser with 24/40 stem.

2.5 Volumetric flasks. One hundred ml and 1 liter.

3. REAGENTS

3.1 Alcoholic potassium hydroxide 0.125 N. Prepare by dissolving 8.3 grams of potassium hydroxide pellets in one liter of absolute ethyl alcohol by refluxing.

4. PROCEDURE

4.1 Weigh a sample of lacquer vehicle (usually 1 gram) that is estimated to contain 0.1 g of phthalic acid into a 500 ml, 24/40 glass-stoppered Erlenmeyer flask.

4.2 Dissolve in 50 ml methyl isobutyl ketone and add 200 ml of the 0.125 N alcohol KOH. Reflux for 3 hours and filter through a dry fritted glass crucible of coarse porosity that was previously prepared with an additional mat of asbestos. Wash with absolute ethyl alcohol, dry at 105° C and cool in a desiccator.

4.3 Dissolve the dried precipitate in water, add 20 ml concentrated hydrochloric acid and dilute to one liter. Filter a portion of this solution through paper of fine porosity, discard the first 25 ml and dilute a 50 ml aliquot of the filtrate to 100 ml with water in a volumetric flask.

4.4 Read the absorbance on the spectrophotometer at 270 and 276 millimicrons using a slit width of 0.6 mm (see 5.1).

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4.5 CALCULATION

4.5.1 Calculate the concentration of phthalic acid in the aliquot as follows:

$$C_{pa} = \frac{1}{b} (0.672 A_{276} - 0.626 A_{270})$$

where: C_{pa} = Grams of phthalic acid per liter and
 b = cell length in cm.

4.5.2 Calculate the percent of phthalic anhydride in the nonvolatile vehicle as follows:

$$\text{Percent phthalic anhydride} = \frac{178.3 C_{pa}}{\text{sample weight}}$$

where: C_{pa} = Concentration of phthalic from 4.5.1. Factor 178.3
assumes dilution as prescribed in 4.3. Sample weight
= nonvolatile vehicle weight.

5. NOTES

5.1 The absorbance should fall within the range of 0.25 to 0.50; otherwise the size of the aliquot taken in 4.3 will have to be adjusted accordingly.

6. PRECISION

6.1 No data.

METHOD 7026.1
 January 24, 1986

NITROGENOUS RESINS AND PHTHALIC ANHYDRIDE IN BAKING ENAMELS

1. SCOPE

1.1 This procedure is for the determination of phthalic anhydride, ureaformaldehyde, and melamine-formaldehyde in the vehicle of baking enamels, and includes a method for identification of benzoguanamine-formaldehyde when present.

2. APPARATUS

2.1 A double-beam infrared recording spectrophotometer.

2.2 Rock salt plates for use in the spectrophotometer.

2.3 Vacuum oven.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Vacuum dry a film of the isolated vehicle for 30 minutes at 70° C on a rock salt plate. Make a rapid scan of the spectrum from 2 to 15 microns and identify the resin modification present by comparison with the figures in ASTM D 2621. Ureaformaldehyde is identified by an absorption band at 6.05 microns, melamine-formaldehyde by bands at 6.5 and 12.3 microns, and benzoguanamine-formaldehyde by bands at 6.5, 12.1 and 12.75 microns. In all cases the alkyd is represented by the carbonyl band at 5.8 microns, and phthalic anhydride by the band at 13.5 microns. After this preliminary qualitative study, make adjustments appropriate to the instrument being used, for quantitative operation. With both beams empty, set zero and 100 percent at 5.9 microns and scan the spectrum of the film from 5.2 to 6.6 microns and from 11.8 to 14.1 microns, using air as the reference and a slow scanning speed. Rotate the salt plate 90° and repeat the slow scan. The film thickness should be such that the absorbance maximum of the carbonyl band at 5.8 microns lies between 0.7 and 1.0 absorbance units. Referring to the procedures outlined in the following sections for the calculation of absorbance ratios, determine the percentage composition of the nonvolatile vehicle by relating these absorbance ratios to standard straight line working curves prepared by carrying samples of known composition through the procedure and plotting absorbance ratios against concentration ratios (C_r) where

$$C_r = \frac{\% \text{ ALKYD}}{\% \text{ UREA FORMALDEHYDE}}, \text{ or}$$

$$C_r = \frac{\% \text{ ALKYD}}{\% \text{ MELAMINE FORMALDEHYDE}}$$

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and in the case of phthalic anhydride in known alkyds,

$$C_r = \frac{\% \text{ PHTHALIC ANHYDRIDE}}{100}$$

the percentages calculated on the nonvolatile basis.

4.2 Urea-formaldehyde in alkyd-urea and alkyd-urea-melamine blends. Draw a baseline from 5.5 to 6.2 microns. Calculate the absorbance due to the alkyd (A) as the difference between that at 5.8 microns and the baseline, and the absorbance due to the urea-formaldehyde (U) as the difference between that at 6.05 microns and the baseline. Calculate the absorbance ratio, $A_r = \frac{A}{U}$, and

average the absorbance ratios for the two runs. Determine the concentration ratio, C_r , from the working curve. Calculate: $\% \text{ urea-formaldehyde} = \frac{100}{1 + C_r}$.

4.3 Melamine-formaldehyde in alkyd melamine blends. Calculate the absorbance due to the alkyd (A) as the difference between that at 5.8 and 5.3 microns, and the absorbance due to the melamine-formaldehyde (M) as the difference between that at 6.5 and 5.3 microns. Calculate the absorbance ratios, $A = \frac{A}{M}$, and

average the absorbance ratios for the two runs. Determine the concentration ratio, C_r , from the working curve. Calculate: $\% \text{ Melamine-formaldehyde} = \frac{100}{1 + C_r}$.

4.4 Melamine-formaldehyde in alkyd-melamine-urea blends. In the case of alkyd-melamine-urea mixtures, both the urea-formaldehyde and the melamine-formaldehyde contribute to the absorbance at 6.5 microns. The following procedure makes it possible to calculate that part of the 6.5 micron absorbance due to melamine formaldehyde. Draw a baseline across the top of the 12.3 micron melamine band, and calculate the absorbance (M^1) as the difference between that at 12.3 microns and the baseline. Calculate the absorbance at 6.5 microns due to melamine-formaldehyde (M) by multiplying M^1 by a factor f . The factor f is determined by averaging the ratios of the absorbance M to the absorbance M^1 obtained from several urea-free alkyd-melamine-formaldehyde samples, the absorbance M being calculated as in paragraph 4.3. The absorbance due to the alkyd (A) and the percent melamine-formaldehyde are determined as outlined in paragraph 4.3.

4.5 Phthalic anhydride content. Calculate the absorbance due to the alkyd (A) as the difference between that at 5.8 and 5.3 microns. Draw a baseline across the top of the 13.5 micron absorption band from 13.2 to 14.0 microns, and calculate the absorbance due to phthalic anhydride (P) as the difference between that at 13.5 microns and the baseline. Calculate the absorbance ratio, $A = \frac{P}{A}$.

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and average the absorbance ratios for the two runs. Determine the percent phthalic anhydride (PA) in the alkyd from the working curve. Calculate the percent phthalic anhydride in the nonvolatile sample:

$$\% \text{ PA (sample)} = \% \text{ PA (alkyd)} \times \frac{(100 - \% \text{ Nitrogen Resin[s]})}{100}$$

5. NOTES

5.1 None.

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METHOD 7060
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DETERMINATION OF LEAD BY X-RAY EMISSION
 SPECTROMETRIC ANALYSIS

1. SCOPE

1.1 This method is applicable to the determination of low levels of lead (less than 0.1 percent) in pigmented organic coatings.

2. APPARATUS

2.1 X-ray fluorescence spectrometer.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Test panel preparation. Using 100 grams of a known lead free enamel, prepare standard aliquots containing 0.00, 0.03, 0.06, and 0.09 percent lead metal, based on total nonvolatile paint, by adding calculated amounts of lead naphthenate of a known lead content. Thoroughly mix the aliquots to incorporate the lead and draw down the standards and enamel to be tested on duplicate black and white moresst cards using a 0.0020 inch (0.004 inch gap clearance) film applicator. Dry for 48 hours at a temperature of $23^{\circ} \pm 1.1^{\circ} \text{C}$ ($73.4^{\circ} \pm 2^{\circ} \text{F}$), a relative humidity of 50 ± 4 percent, and under dust free conditions. Cut the drawdowns into a suitable size and shape to fit the sample holder of the X-ray fluorescence spectrometer.

4.2 X-ray analytical procedure. Lead content shall be determined using an X-ray fluorescence spectrometer capable of determining lead content at a minimum level of 0.03 percent by weight of the total nonvolatile paint. The parameters of angle, crystal, pulse height selection, counting time, collimator, X-ray tube, voltage and amperage, shall be established for a wave length dispersive fluorescence spectrometer according to conventional X-ray analytical procedures. The analytical line Pb L-Alpha or Pb L-Beta shall be used. To calibrate, place the known standards in the X-ray unit and measure the count rates of lead, lead background and the Compton scattered background from the X-ray tube. The ratio R, of net lead intensity and Compton scattered background is calculated as follows:

$$R = [2I_{\text{Pb}} - I_{\text{Pb}}(\text{Background I}) - I_{\text{Pb}}(\text{Background II})] / 2I_{\text{Compton line}}$$

Where I = Gross Intensity
 and the background is taken on each side of the Pb line.

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Establish a lead calibration curve using these results. Determine the lead content of the test paint using the above procedure and calibration curve. When using an energy dispersive fluorescence spectrometer, it shall be set up in accordance with the manufacturer's manual.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 7071.1
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RED LEAD IN PIGMENTS.

1. SCOPE

1.1 This procedure is for the quantitative measure of red lead in paint pigments. The only interference is from zinc chromate, the absence of which must first be ascertained. If the qualitative test described is positive, the zinc chromate must first be quantitatively removed as provided in the procedure.

2. APPARATUS

2.1 Cylinder. One hundred ml glass-stoppered.

2.2 Flask. Two hundred-fifty ml Erlenmeyer.

2.3 Magnetic stirrer and bar.

2.4 Ten ml burette.

2.5 Buchner funnel.

2.6 Suction flask.

3. REAGENTS

3.1 Ammonium chloride. Ten percent aqueous, freshly prepared for the test in 4.1.

3.2 Iodine solution, 0.1 N. Dissolve 15 grams of potassium iodide in 25 ml of water and add 12.691 g of resublimed iodine by prolonged agitation at room temperature. When the iodine has completely dissolved, dilute to one liter with water.

3.3 Potassium iodide, crystals. A.C.S. reagent grade, iodate-free.

3.4 Sodium acetate, 20 percent aqueous. Dissolve 200 g of the trihydrate in water and dilute to one liter.

3.5 Sodium acetate, special concentrated. Add 305 ml of water and 85 ml of glacial acetic acid to 500 g of sodium acetate trihydrate crystals. Warm to dissolve and cool to room temperature. Do not remove crystals that form at the bottom of the container.

3.6 Sodium thiosulfate, 0.1 N. Dissolve 25 g of sodium thiosulfate pentahydrate in water and dilute to one liter. Add 0.5 g of sodium carbonate. Standardize against iodine crystals or potassium dichromate. If stored for one month before standardization, the normality will not change appreciably.

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4. PROCEDURE

4.1 Place about 2 g of the pigment to be tested in a 100 ml glass-stoppered graduated cylinder and add 50 ml of the freshly prepared 10 percent ammonium chloride solution. Stopper and shake vigorously for several minutes. Filter through a double thickness of the finest filter paper into a flask and examine for color. If the filtrate is colorless or extremely pale yellow, discard and proceed to the quantitative analysis for red lead in 4.2. If a definite yellow color indicates the presence of zinc chromate, weigh a 1- to 2-g sample of pigment into a centrifuge tube and wash out the chromate by repeated decanting, shaking and centrifuging until the supernatant liquid is colorless. Dry the pigment sample and proceed to the analysis for red lead in 4.2.

4.2 A 1- to 2-g sample of the pigment is accurately weighed into a 250 ml glass-stoppered Erlenmeyer flask with a 24/40 joint. A magnetic stirring bar is added, then 2 g of potassium iodide crystals, exactly 10.0 ml of standard 0.1 N sodium thiosulfate from a pipette or buret, and 45 ml of the special concentrated sodium acetate solution. Stopper the flask and stir magnetically for a minimum of 10 minutes, taking care that the heat from the stirrer does not warm the solution. Add 30 ml of the 20 percent aqueous sodium acetate solution from a graduate. Filter through a Buchner funnel using fine filter paper into a clean suction flask and wash with water. Add exactly 10.0 ml of the 0.1 N iodine solution from a pipette, mix and titrate the excess iodine with the standard thiosulfate, using a 10 ml buret, and starch as indicator. Record the volume of thiosulfate as V_1 .

4.3 Determine the iodine-thiosulfate equivalence by titrating 10.0 ml of the iodine solution in the presence of 45 ml of the special concentrated sodium acetate and record the volume of thiosulfate used as V_2 .

4.4 Calculation.

$$\text{Percent red lead} = \frac{[(10.0 + V_1) - V_2] \times \text{normality of thiosulfate} \times 34.3}{\text{Weight of pigment sample}}$$

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 7073
January 24, 1986

ANALYSIS OF PURE RED LEAD PIGMENT

1. SCOPE

1.1 This method is used for the analysis of dry pigment or pigment extracted from enamels in which the red lead content ranges from 85 percent to 100 percent.

2. MATERIALS

2.1 Buret, 50 ml capacity.

2.2 Magnetic stirrer base.

2.3 Magnetic stirrer bar, cylindrical, straight-sided with no center ridge.

3. REAGENTS

3.1 Acetic acid, glacial.

3.2 Indicator, starch solution.

3.3 Methyl alcohol, absolute.

3.4 Sodium acetate solution, 20 percent

3.5 Sodium thiosulfate, 0.1 N solution, standardized with potassium dichromate.

4. PROCEDURE

4.1 Mix in a small beaker 30 grams of crystallized sodium acetate trihydrate, 2.4 grams of potassium iodide and 25 ml of 25 percent acetic acid. Cover and warm gently and stir until dissolved, then cool to room temperature.

4.2 Weigh accurately a sample of the finely ground red lead pigment of approximately 1.0 gram into a 250 ml Erlenmeyer flask. Insert a cylindrical magnetic stirring bar. Fill a 50 ml buret with 0.1 N standardized sodium thiosulfate solution and prepare to add the following in rapid succession. Add 10 ml of a mixture of 3 ml of glacial acetic acid and 7 ml of methyl alcohol. Swirl the flask about 5 seconds, then add the above mixture of iodide and acetate (paragraph 4.1), swirl about 5 seconds then add 30 ml of 20 percent sodium acetate solution. Swirl continually and start titrating immediately with the thiosulfate and continue until the sample is pale yellow. Stopper the flask and place on the stirrer base and stir vigorously for 3 minutes. Continue titrating to pale yellow again and add starch indicator. Titrate to the disappearance of the blue color, approaching the end point very slowly, with one-drop additions. If over-titrated, iodine solution (0.1 N) may be used for back titration.

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4.3 Calculation.

4.3.1 Calculate the red lead content as follows:

$$\text{Percent red lead} = \frac{\text{ml thiosulfate} \times \text{normality} \times 34.278}{\text{Weight of sample}}$$

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 7111.1
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ANALYSIS OF CHROME GREEN PIGMENT

1. SCOPE

1.1 This method covers procedures for the analysis of chrome green pigment either in the dry form or extracted from paste-in-oil. For the analysis of chrome green pigment extracted from paint, method 7281 should be used.

2. APPARATUS

2.1 Beakers, 400- and 600-ml.

2.2 Porcelain dish.

2.3 Gooch crucible.

2.4 Hotplate.

3. REAGENTS

3.1 Ethyl alcohol, 95 percent. Boil and neutralize to the phenolphthalein end point with alkali just before using.

3.2 Nitric acid (1 to 3). Mix 1 part by volume of A.C.S. reagent grade nitric acid with 3 parts by volume of freshly distilled water.

3.3 Sulfuric acid (1 to 1). Mix equal volumes of A.C.S. reagent grade sulfuric acid and freshly distilled water.

3.4 Standard ferrous ammonium sulfate solution (0.1 N). Dissolve 42 g of A.C.S. reagent grade ferrous ammonium sulfate in a cool mixture of 50 ml of A.C.S. reagent grade sulfuric acid and 950 ml of freshly distilled water. To obtain the ratio of the ferrous ammonium sulfate solution to the potassium permanganate solution (see 3.6), take 25 ml of the former, dilute to 350 ml with cool dilute sulfuric acid (see 3.9), add 2 ml of A.C.S. reagent grade phosphoric acid, and titrate with standard potassium permanganate solution to a faint permanent pink tint. Determine a blank on the same volume of water and acids, deduct, and calculate the volume of potassium permanganate solution which is equivalent to 1 ml of the ferrous ammonium sulfate solution. The ratio of the ferrous ammonium sulfate solution to potassium permanganate solution must be determined each day the solution is used for titrating a test specimen, unless the ferrous ammonium sulfate solution is kept in an inert atmosphere, i.e., nitrogen or other inert gas.

3.5 Silver nitrate solution (2.5 percent). Dissolve 2.5 g of A.C.S. reagent grade silver nitrate in freshly distilled water and make up to 100-ml volume.

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3.6 Standard potassium permanganate solution (0.1 N).

3.6.1 Dissolve 3.2 g of potassium permanganate in a liter of water, let stand 8 to 14 days, and siphon off the clear solution (or filter through an asbestos filter). Standardize as follows: Transfer 0.3 g, weighed to the nearest 0.1 mg, of sodium oxalate, National Bureau of Standards standard 40 (dried for 1 hour at 105° C) to a 600-ml beaker. Add 250 ml of 50 percent sulfuric acid, previously boiled for 10 to 15 minutes and then cooled to 27° +3° C. Stir until the oxalate has dissolved. Add 30 to 40 ml of the potassium permanganate solution (0.3 g of the sodium oxalate requires 44.75 ml of 0.1n potassium permanganate solution) at a rate of 25 to 35 ml per minute, while stirring slowly. Let stand until the pink color disappears (about 45 seconds) (Note 1). Heat to 55° to 60° C and complete the titration by adding the permanganate solution until a faint pink color persists for 30 seconds. Add the last 0.5 to 1 ml dropwise, with particular care to allow each drop to become decolorized before the next is introduced.

3.6.2 Determine the excess of permanganate required to impart a pink color to the solution, by matching the color by adding the permanganate solution to the same volume of the sulfuric acid (see 3.6.1) at 55° to 60° C. (This correction may vary from 0.03 to 0.05 ml.)

3.6.3 Calculate the normality of the permanganate solution using the following equation:

$$N = \frac{S}{0.06701 (A-B)}$$

Where: N = normality of potassium permanganate solution.
 S = g of sodium oxalate used.
 A = ml of potassium permanganate solution required for the sodium oxalate solution.
 B = ml of potassium permanganate solution required for the sulfuric acid blank.

3.6.4 The standard potassium permanganate solution shall be protected from light.

NOTE 1. If the pink color should persist because the permanganate is in excess, discard and begin again.

3.7 Ammonium persulfate solution. A freshly prepared solution of A.C.S. reagent grade ammonium persulfate in freshly distilled water in the proportion of 4 grams per 20 ml of solution.

3.8 Hydrochloric acid (1 to 3). Mix 1 part by volume of A.C.S. reagent grade hydrochloric acid with 3 parts by volume of freshly distilled water.

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3.9. Sulfuric acid (1 to 19). Carefully mix 1 volume of H_2SO_4 (Sp gr 1.84) with 19 volumes of water.

3.10. Sodium hydroxide solution (30 percent). Dissolve 30 g of A.C.S. reagent grade sodium hydroxide in 70 ml of freshly distilled water.

4. PROCEDURE

Examine the pigment in dry form or after its extraction from the paste-in-oil by method 4021 as follows.

4.1. Qualitative analysis. Examine the pigment for adulteration as follows.

4.1.1. Transfer about 1 g of the pigment to a 400-ml beaker, and add 20 ml of 30-percent sodium hydroxide solution. Heat to boiling; a reddish-brown precipitate should form. Add an excess of A.C.S. reagent grade hydrochloric acid and boil, then dilute with water to about 300 ml and boil. A clear greenish-yellow solution should result. Test for calcium.

4.1.2. A clear solution in the above test and negative test for calcium indicates freedom from adulteration. In doubtful cases make qualitative test for organic colors in accordance with method 5021.

4.2. Quantitative analysis.

4.2.1. Total lead as $PbSO_4$.

4.2.1.1. Heat about 0.5 g weighed to the nearest 0.1 milligram of the pigment very gently in a small porcelain dish. The heat must be so regulated by moving the burner that the iron blue is thoroughly decomposed without rendering the iron difficultly soluble (see 5.1). Allow to cool, transfer to a 400-ml beaker, add 25 ml of A.C.S. reagent grade hydrochloric acid and boil for from 5 to 10 minutes in a covered beaker, adding about 6 drops of alcohol to the boiling liquid, one at a time. Dilute to 100 ml with hot water and boil for from 5 to 10 minutes (solution of the chrome green should be complete). Filter the hot solution, if insoluble matter is present, and wash with boiling water until washings are free from lead and chlorine.

4.2.1.2. Add A.C.S. reagent grade ammonium hydroxide to the filtrate (or the original solution) until a faint precipitate begins to form, then add 5 ml of A.C.S. reagent grade hydrochloric acid, and dilute to 500 ml with water. Pass into the clear solution a rapid current of hydrogen sulfide until all the lead is precipitated as lead sulfide. Let the precipitate settle, filter through filter paper, and wash with water containing a little hydrogen sulfide. Boil the filter paper and precipitate with dilute nitric acid (see 3.2) until all of the lead has dissolved, filter, and wash thoroughly with hot water.

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4.2.1.3 To the filtrate, add 10 ml of dilute sulfuric acid (see 3.3), evaporate until copious fumes of sulfur trioxide are evolved. Cool, and add about 75 ml of water and then 75 ml of alcohol (see 3.1). Let stand about 1 hour, filter on a weighed Gooch crucible previously prepared in the usual manner, wash with diluted alcohol, dry, and ignite briefly at a dull red heat. Cool the crucible in a desiccator and weigh. From the weight of the precipitate and the weight of the sample taken compute the total lead as percentage of lead sulfate.

4.2.2 Total chromium as PbCrO₄.

4.2.2.1 Transfer about 0.5 g weighed to the nearest 0.1 milligram of the pigment to a 600-ml heat-resistant glass beaker, add 25 ml of A.C.S. reagent grade sulfuric acid and fume lightly on a hotplate for 4 or 5 minutes (see 5.2). Cool, cautiously dilute with water to about 300 ml, stir, and heat to boiling. To the boiling solution add a small amount of potassium permanganate solution (see 3.6) to insure the presence of some manganese. Then add to the boiling solution 10 ml of the silver nitrate solution (see 3.5) and 20 ml (adding this slowly) of the ammonium persulfate solution (see 3.7). The hot solution should show the usual permanganate color. If this color does not develop, or if it disappears, add more persulfate. When the permanganate color is permanent, boil (best done on a hotplate) for 10 to 15 minutes to destroy excess persulfate. Then add 5 ml of dilute hydrochloric acid (see 3.8) and boil for 5 to 8 minutes to reduce oxidized manganese. The color now should be the usual chromate yellow (see 5.3).

4.2.2.2 Cool to about 20° C, dilute to 400 ml, and add exactly 50 ml of the standard ferrous ammonium sulfate solution (see 3.4) from a burette. Titrate^{1/} the excess of ferrous salt with the standard potassium permanganate solution (see 3.6). The first faint permanent (1 minute of stirring) darkening of the green color is taken as the end point. (The end point is quite sharp and well defined, but requires some practice. Addition of excess permanganate increases the depth of the color which finally becomes purple.)

4.2.2.3 Determine the volume of the potassium permanganate solution that is equivalent to 50 ml of the ferrous ammonium sulfate solution according to the procedure for standardization given in 3.4.

4.2.2.4 Calculate the total chromium as percentage of lead chromate using the following equation:

^{1/} If preferred, the electrometric titration as described by Kelly in J. Ind. & Eng. Chem., vol. 13, p. 1033 (1921) may be used.

$$\text{Percent PbCrO}_4 = \frac{(A - B) \times N \times 0.1077}{S} \times 100$$

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where:

- A = Milliliters of the standard potassium permanganate solution equivalent to 50 milliliters of the ferrous ammonium sulfate solution (see 4.2.2.3).
 B = Milliliters of the standard potassium permanganate solution required to titrate excess of ferrous salt in sample (see 4.2.2.2).
 N = Normality of potassium permanganate solution.
 S = Grams of sample used.

4.2.3 Calculation of the percentage of total lead (Pb) present in the form of PbCrO₄. Compute the percentage of lead (Pb) present in the form of lead chromate of the total lead (Pb) in the pigment as follows.

4.2.3.1 Multiply the percentage of lead sulfate as determined in 4.2.1.3 by the factor 0.683 to obtain the percentage of total lead (Pb) in the pigment. Call this value "b".

4.2.3.2 Multiply the percentage of lead chromate as determined in 4.2.2.4 by the factor 0.641 to obtain the percentage of lead (Pb) present as lead chromate in the pigment. Call this value "A".

4.2.3.3 Then "C", the percentage of total lead (Pb) present in the pigment in the form of PbCrO₄, is obtained by the following equation:

$$C = \frac{A}{B} \times 100$$

5. NOTES

5.1 With some very pure chrome greens it may be advantageous to mix the sample with from two to five times its weight of pure barium sulfate before igniting.

5.2 Excessive fuming or cooking over a hot flame is neither necessary nor desirable, as it may produce the difficultly soluble anhydrous chromium sulfate. Prussian blue color is destroyed in a few minutes.

5.3 After the addition of the ammonium persulfate, the elapsed boiling time should be not less than 15 nor more than 30 minutes.

6. PRECISION

6.1 No data.

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METHOD 7131.1
January 24, 1986

ANALYSIS OF CHROME YELLOW AND CHROME ORANGE PIGMENTS

1. SCOPE

1.1 This method covers procedures for the analysis of chrome yellow and chrome orange pigments either in dry form or after extraction from paste-in-oil. For analysis of pigment extracted from chrome yellow and chrome orange paints, method 7271 should be used.

2. APPARATUS

2.1 Five hundred-ml Erlenmeyer flask.

2.2 Gooch crucible.

2.3 Desiccator.

3. REAGENTS

3.1 Ethyl alcohol, 95 percent. Boil and neutralize to the phenolphthalein end point with alkali just before using.

3.2 Potassium hydroxide solution. Dissolve 50 g of A.C.S. reagent grade potassium hydroxide in freshly distilled water and dilute to 100 ml.

3.3. Standard sodium thiosulfate solution (0.1 N). As prescribed in ASTM D 1959.

3.4 Starch indicator solution. As prescribed in ASTM D 1959.

4. PROCEDURES

Examine the dry pigment or the pigment extracted from the paste-in-oil by method 4021 as follows.

4.1 Qualitative analysis. Test the pigment for base metals other than lead by the regular methods of the qualitative analysis. If they are found to be present, as may happen infrequently, determine the amounts in which they are present by appropriate quantitative methods.

4.2 Quantitative analysis.

4.2.1 Total chromium as $PbCrO_4$ (see note 1).

4.2.1.1 Weigh to the nearest 0.1 milligram about 0.4 g of the pigment and transfer to a 500-ml Erlenmeyer flask. Add 6 ml of the potassium hydroxide solution (see 3.2) and 10 ml of distilled water. Heat until dissolved. Do not

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allow to dry. To the hot solution add 50 g of A.C.S. reagent grade potassium chloride. Dilute with water to 200 ml and swirl until dissolved. Add 16 ml of A.C.S. reagent grade hydrochloric acid slowly. Add 10 g of A.C.S. reagent grade potassium iodide and titrate with the standard sodium thiosulfate solution (see 3.3) using starch solution (see 3.4) as the indicator.

4.2.1.2 Calculation. Calculate the total chromium expressed as percentage of lead chromate, in the pigment using the following equation:

$$\text{Percent PbCrO}_4 = \frac{A \times N \times 0.1077}{S} \times 100$$

where:

A = Milliliters of standard sodium thiosulfate solution required for titration.
 N = Normality of sodium thiosulfate solution.
 S = Grams of sample used.

Note 1: The iodine liberation method is not applicable in the presence of molybdenum.

4.2.2 Substances other than insoluble lead compounds.

4.2.2.1 Insoluble matter. Treat about 1 g, weighed to the nearest 0.1 milligram, of the pigment with 25 ml of A.C.S. reagent grade hydrochloric acid and boil for from 5 to 10 minutes in a covered beaker. Add about 6 drops of alcohol (see 3.1), 1 at a time to the boiling liquid. Dilute to 100 ml with hot water and boil for from 5 to 10 minutes. Filter the hot solution (if insoluble matter is present) through a weighed Gooch crucible, prepared in the usual manner. Wash with boiling water till washings are free from lead and chlorine. Dry, ignite, cool in a dessicator, weigh, and compute percentage of insoluble matter.

4.2.2.2 Matter soluble in water. Determine the percentage of water soluble material in the pigment by ASTM D 1208, section 3.

4.2.2.3 Moisture and other volatile matter. Determine the percentage of moisture and other volatile matter in the pigment by ASTM D 280.

4.2.2.4 Computation. Compute the total percentage of all substances other than insoluble lead compounds in the pigment by adding together the percentages of insoluble matter, matter soluble in water, moisture and other volatile matter, and other impurities, determined as prescribed in 4.1.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 7161.1
January 24, 1986

ANALYSIS OF SYNTHETIC BLACK AND BROWN IRON OXIDE PIGMENTS

1. SCOPE

1.1 This method covers procedures for the analysis of synthetic black and brown iron oxide pigments either in dry form or after extraction from paste-in-oil. For analysis of such pigments extracted from paints, method 7291 should be used.

2. APPARATUS

2.1 Conventional laboratory apparatus and equipment to be used in this test method.

3. REAGENTS

3.1 Sulfuric acid (1 to 3). Mix 1 part A.C.S. reagent grade sulfuric acid with three parts of freshly distilled water by volume.

3.2 Sodium carbonate. A.C.S. reagent grade.

3.3 Sulfuric-phosphoric acid mixture. Mix 150 ml of concentrated sulfuric acid with 150 ml of concentrated phosphoric acid and dilute the mixture with water to 1 liter.

3.4 Sodium diphenylamine sulfonate indicator solution. Dissolve 0.2 g of sodium p-(phenylamine)-phenyl sulfonate ($C_6H_5NHC_6H_4SO_3Na$) in 100 ml of water.

3.5 Standard potassium dichromate solution (0.1 N). As prescribed in ASTM D 521.

4. PROCEDURE

Examine the pigment in dry form or after its extraction from the paste-in-oil by method 4021 as follows.

4.1 Qualitative analysis. Examine the pigment for the presence of adulterations and extender pigments such as calcium carbonate, silica, magnesium silicate, barium sulfate, etc., by the regular methods of qualitative analysis.

4.2 Quantitative analysis.

4.2.1 Total iron oxide.

4.2.1.1 Use about 0.3 g, weighed to the nearest 0.1 mg of the pigment and follow the procedure given in ASTM D 50.

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4.2.1.2 Calculate the percentage of total iron oxide expressed as ferrous iron oxide using the following equation:

$$\text{Percent FeO} = \frac{A \times N \times 0.7185}{S} \times 100$$

where:

- A = Milliliters of standard potassium dichromate solution required for titration.
- N = Normality of potassium dichromate solution.
- S = Grams of sample used.

4.2.2 Ferrous iron oxide.

4.2.2.1 Weigh to the nearest 0.1 mg about 1 g of the pigment and transfer to a 300 ml Erlenmeyer flask. Add 50 ml of the sulfuric acid (see 3.1) that has been previously boiled and cooled, and a pinch of sodium carbonate (see 3.2). Boil the solution gently until all the pigment is dissolved. Cover the solution with a layer of carbon dioxide, stopper the flask and allow to cool to room temperature. Add 100 ml of distilled water, 25 ml of the sulfuric-phosphoric acid mixture (see 3.3), and 6 to 8 drops of the sodium diphenylamine sulfonate indicator solution (see 3.4). Titrate with the potassium dichromate solution (see 3.5) taking the sudden change from the green color to a purple color as the end point.

4.2.2.2 Calculate the percentage of ferrous iron oxide using the equation given in 4.2.1.2.

4.2.3 Total ferro-ferric oxide of iron. Subtract the percentage of FeO obtained in 4.2.2.2 from that obtained in 4.2.1.2 and convert this difference to percentage of Fe₂O₃ by multiplying by the factor 1.113. Add the percentage of Fe₂O₃ thus obtained to the percentage of FeO obtained in 4.2.2.2 to obtain the percentage of total ferro-ferric oxide of iron in the pigment.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 7171.1
January 24, 1986

ANALYSIS OF COPPER PHTHALOCYANINE BLUE PIGMENTS

1. SCOPE

1.1 This method covers procedures for the analysis of copper phthalocyanine blue pigment in dry powder form for use as a pigment in paints.

2. APPARATUS

2.1 Beaker. A 50-ml beaker.

2.2 Platinum wire.

2.3 Bunsen burner.

2.4 Beaker. A 200-ml beaker.

2.5 Filter paper. Medium speed.

2.6 Beaker. A 100-ml beaker.

2.7 Distilling head. A distilling head with a 24/40 standard taper in connection with a 24/40 standard taper flat bottomed boiling flask.

3. REAGENTS

3.1 Standard sodium thiosulfate solution (0.1 N). This solution may be prepared and standardized by either of the following methods (see 5.1).

3.1.1 Method A - standardization with potassium dichromate. Prepare and standardize the sodium thiosulfate solution in accordance with the following procedure: dissolve 24.83 g of A.C.S. reagent grade sodium thiosulfate in distilled water and dilute to 1 liter. Let the solution stand 2 weeks before standardizing (one g of sodium carbonate per liter may be added as a preservative). Standardize against A.C.S. reagent grade potassium dichromate^{1/} as follows. Weigh to the nearest 0.1 mg by difference from a weighing bottle 0.16 to 0.22 gram of potassium dichromate that has been ground and then dried to constant weight at 110° C (230° F) prior to use. Place the potassium dichromate in a 500-ml flask or bottle and dissolve in 25 ml of distilled water. Add 5 ml of A.C.S. reagent grade hydrochloric acid and 20 ml of the potassium iodide solution, and swirl to mix. Allow to stand for 5 minutes and then add 100 ml of water. Titrate with the sodium thiosulfate solution, while shaking constantly, until the yellow color has almost disappeared. Add 1 to 2 ml of the starch

^{1/} NBS Std. sample No. 136A of potassium dichromate is recommended for this purpose and should be treated as directed in the certificate of analysis, accompanying the sample.

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indicator solution and continue the titration, adding the sodium thiosulfate solution slowly until the blue color has just disappeared. Calculate the normality of the sodium thiosulfate as follows:

$$\text{Normality} = \frac{20.39 \times C}{A}$$

where:

C = Grams of potassium dichromate used.

A = Milliliters of sodium thiosulfate solution required for titration of the potassium dichromate.

3.1.2 Method B - standardization with metallic copper. Prepare the sodium thiosulfate solution as in method A. Weigh to the nearest 0.1 mg between 0.1 and 0.2 g of A.C.S. reagent grade copper into a flask similar to that used for the determination of copper in the pigment. Add 20 ml of nitric acid and boil with trap in place until all the copper is dissolved. Cool and add 2 ml of A.C.S. reagent grade sulfuric acid and 3 ml of A.C.S. reagent grade 70-percent perchloric acid. Evaporate to almost dryness and continue in accordance with the procedure given in 4.2.1.1 and 4.2.1.2, taking all recommended precautions. Calculate the normality of the thiosulfate solution using the following equation

$$\text{Normality} = \frac{S}{A \times 0.06354}$$

where:

S = Grams of copper used.

A = Milliliters of sodium thiosulfate solution required for titration of the copper.

3.2 Starch indicator solution. Make a homogeneous paste of 10 g of A.C.S. reagent grade soluble starch in cold water. Add 1 liter of boiling water to the paste, stir rapidly and cool to room temperature. Salicylic acid (1.25 g per liter) may be added to preserve the indicator.

3.3 Alcoholic ammonium hydroxide solution (1 to 1). Mix equal volumes of ethyl alcohol (see 3.6) and A.C.S. reagent grade ammonium hydroxide.

3.4 Tartaric acid solution. Dissolve 20 g of A.C.S. reagent grade tartaric acid in freshly distilled water and dilute to 100-ml volume.

3.5 Titanous chloride solution (0.1 N). Titanium trichloride is marketed as a 16-percent solution in hydrochloric acid. Mix 7 ml of this solution with 90 ml of a mixture of 1 part by volume of A.C.S. reagent grade hydrochloric acid and 12 parts by volume of freshly distilled water to obtain an approximately 0.1 N solution of titanous chloride. Protect this solution from oxidation.

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3.6 Ethyl alcohol. Ethyl alcohol (95 percent) or specially denatured alcohol formula 30 or 3A of the United States Bureau of Internal Revenue.

3.7 Hydrochloric acid (1 to 1). Mix equal parts by volume of A.C.S. reagent grade hydrochloric acid and freshly distilled water.

3.8 Sodium hydroxide solution (5 percent). Dissolve 1 part by weight sodium hydroxide, A.C.S. reagent grade, in 19 parts by weight of freshly distilled water.

3.9 Ferric iron solution (2 percent). A solution containing 2 percent of iron as ferric sulfate is required and may be prepared as follows: Dissolve 20 g of pure iron or plain carbon steel in a slight excess of hydrochloric acid, A.C.S. reagent grade, oxidize with A.C.S. reagent grade nitric acid, add approximately 80 ml of sulfuric acid, A.C.S. reagent grade, and heat until fumes of the latter are evolved. Cool, dilute with distilled water to 1,000 ml digest on a steam bath until the sulfates are dissolved, and filter if necessary. Add the potassium permanganate solution until a faint pink color is obtained (to oxidize any ferrous iron that may be present).

3.10 Ammonium chloride solution (5 percent). Dissolve 1 part by weight of ammonium chloride, A.C.S. reagent grade, in 19 parts by weight of freshly distilled water.

3.11 Potassium ferrocyanide solution. Dissolve 0.1 g of A.C.S. reagent grade potassium ferrocyanide in freshly distilled water and dilute to 100-ml volume. This solution must be prepared on the day it is to be used.

4. PROCEDURES

4.1 Qualitative analysis.

4.1.1 Identification. To a small sample (about 0.05 g) in a 50-ml beaker, add about 30 ml of A.C.S. reagent grade sulphuric acid. Stir occasionally for about 15 minutes; the sample shall dissolve, forming a dark greenish-yellow solution (color best seen on the side of the beaker). Pour the sulfuric acid solution into about 250 ml of distilled water and stir. The copper phthalocyanine shall immediately precipitate as a brilliant blue flocculent mass. Filter off the precipitate, and wash once or twice with distilled water. Transfer a small amount of the precipitate to a clean platinum wire moistened with A.C.S. reagent grade hydrochloric acid, and heat over a low flame of a Bunsen burner. As the precipitate burns, a light blue-green flame should be clearly evident. This indicates organically bound copper.

4.1.2 Detection of extraneous material.

4.1.2.1 Basic dye derivatives. Add about 50 ml of the alcoholic ammonium hydroxide (see 3.3) to about 1 g of the pigment in a 200-ml beaker. Warm gently

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and filter through filter paper. Neutralize the filtrate with the tartaric acid solution (see 3.4) until slightly acid to litmus. If the solution is colorless, discounting a slight yellow tint, no basic dye is present. If the solution is colored beyond a slight yellow tint, add about 5 ml of the titanous chloride solution (see 3.5). If a basic dye is present, the color will lighten significantly. If no basic dye is present, no significant color change will occur.

4.1.2.2 Other organic coloring matter. To two small portions of the pigment (about 0.05 gram each) in separate 50-ml beakers, add about 25 milliliters (at room temperature) of the ethyl alcohol (see 3.6) to one, and about 25 ml of A.C.S. reagent grade acetone to the other. Stir each well for a few minutes, and let stand for about 2 hours. Filter through two thicknesses of filter paper. Neither filtrate shall possess more than an extremely slight pink, yellow, or blue cast. The presence of organic colors resistant to the above reagent is unlikely, but may be tested by reference to procedures given in standard reference works.^{2/}

4.1.2.3 Ultramarine blue. Gently warm some of the insoluble portion of the pigment obtained in 4.1.2.1 or some of the original pigment with dilute hydrochloric acid (see 3.7). Decomposition of the ultramarine blue takes place with evolution of hydrogen sulfide gas. This may be detected either by its odor or a brown coloration appearing on a strip of moistened lead acetate paper.

4.1.2.4 Iron blue. Place about 1 g of the pigment in a 100-ml beaker, and add 25 ml of the sodium hydroxide solution (see 3.8). Boil for about 1 minute. Dilute to approximately 40 ml with distilled water and filter. Add the dilute hydrochloric acid to the filtrate until it is faintly acid to litmus. Add 2 ml of the ferric iron solution (see 3.9). The development of a blue color reveals the presence of ferrocyanide and hence iron blue in the original pigment. For small amounts of iron blue (0.05-0.1 percent), the color may take 2 to 3 hours to develop.

4.1.2.5 Ionic copper. Weigh about 1 g of pigment into a 100-ml beaker. Add 15 ml of the ammonium chloride solution (see 3.10). Add about 3 ml of the ethyl alcohol (see 3.6) to aid in wetting the pigment. Boil gently for about 3 minutes. Filter and wash once with the hot ammonium chloride solution. Divide filtrate equally between two test tubes of similar dimensions and optical properties. Add about 1 ml of the potassium ferrocyanide solution (see 3.11) to one of the tubes. Compare tubes after about 1 minute. A rose color as compared with the

2/ "Analysis of Dyestuffs" by A. G. Green, 3rd ed., published by Charles Griffin and Co., Ltd., London, England (1920). "The Chemistry and Physics of Organic Pigments," by L. S. Pratt, published by John Wiley and Sons, Inc., New York, N.Y., (1947).

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untreated solution indicates ionic copper. A positive test close to the threshold value indicates copper of the order of magnitude of 0.002 percent Cu^{++} . A negative test indicates less than 0.002 percent Cu^{++} .

4.2 Quantitative analysis.

4.2.1 Copper content. Considerable hazard may be involved in the solution of the pigment, therefore, the precautionary measures given in 5.3 and 5.4 should be closely followed.

4.2.1.1 Weigh to the nearest 0.1 mg about 1 g of the pigment into a 250-ml flask equipped with an all glass trap (see 5.2 and 5.3). Add 2 ml of A.C.S. reagent grade sulfuric acid, 20 ml of A.C.S. reagent grade fuming nitric acid, and 3 ml of A.C.S. reagent grade 70-percent perchloric acid. Heat (see 5.4) at such a temperature that the solution is almost to dryness in about 15 minutes. Allow to cool somewhat and wash down the trap and flask with water. Add a few ml of A.C.S. reagent grade hydrochloric acid and with the trap in place, again take the volume down almost to dryness. Again wash down trap and flask with water finishing with a volume of about 40 ml. Heat to boiling with the trap off and evaporate to about 20 ml (see 5.5).

4.2.1.2 Cool. Add A.C.S. reagent grade ammonium hydroxide dropwise to just alkaline. Add 6 ml of A.C.S. reagent grade glacial acetic acid. Cool. Add 1 g of A.C.S. reagent grade sodium fluoride and stir until most of it has dissolved. Disregard any small amount that may remain out of solution (see 5.6). Add 0.8 g of A.C.S. reagent grade potassium iodide previously dissolved in a little water. Titrate the free iodine with the standard solution of sodium thiosulfate (see 3.1). Near the end point add 1 ml of the starch indicator solution (see 3.2) and 0.8 g of A.C.S. reagent grade ammonium or potassium thiocyanate (see 5.7). Continue the titration to the disappearance of the blue starch-iodine color (see 5.8).

4.2.1.3 Calculate the percentage of copper in the pigment using the following equation:

$$\text{Percent Cu} = \frac{A \times N \times 0.06354}{S} \times 100$$

where:

A = Milliliters of the standard sodium thiosulfate solution required for titration.

N = Normality of sodium thiosulfate solution.

S = Grams of sample used.

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5. NOTES

5.1 Method A has the advantage of being much faster than method B but it is not based on as sound chemical principles. The two methods give results that are no further apart than 0.5 percent. A small amount of evidence indicates that method B gives slightly higher results.

5.2 For small amounts of copper such as are present in copper phthalocyanine blue lakes (approximately 1 percent Cu) a 2-g sample may be used. It will be necessary then to increase the sulfuric, nitric, and perchloric acids and the sodium fluoride proportionately. The final volume of 20 ml just before addition of ammonium hydroxide may be too small, and should therefore be increased to 35 ml. Also during digestions considerable bumping may be experienced. This can be partly overcome by the addition of glass beads.

5.3 A trap is desirable to prevent loss of copper during the oxidation of the pigment and the fuming of the resulting solution. The trap must be all glass because of the danger of an explosion if hot concentrated perchloric acid comes in contact with rubber or cork. A distilling head with 24/40 standard taper in connection with a 24/40 standard taper flat bottom boiling flask makes a satisfactory combination. A small stirring rod placed in the flask and partially up the trap will tend to lead the condensate back into the flask without dropping directly into the hot solution causing spattering during this initial heating period.

5.4 In the interest of safety, goggles should be worn when working with hot concentrated perchloric acid because of the danger of an explosion if the perchloric acid comes in contact under certain conditions with reducing agents. When used in the manner indicated, no explosion has ever occurred, but it is well to keep the flask shielded so as to offer protection to the operator during this initial heating period. Under these conditions, the organic matter can be completely destroyed in one operation. If, however, it is thought that some organic matter remains, the acid treatment should be repeated.

5.5 The chlorine produced from the perchloric acid is removed by boiling. Its complete removal can be tested with starch-potassium iodide paper or by smelling cautiously.

5.6 The fluoride is added to complex any iron that may be present. The amount suggested should take care of all the iron ever found in a copper phthalocyanine blue pigment.

5.7 The thiocyanate prevents the absorption of iodine by the precipitate and makes the endpoint sharper.

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5.8 The correct observation of the endpoint requires a little experience because the precipitate has a gray to tan color. With experience, however, the endpoint is precise to 1 drop of 0.1 N sodium thiosulfate.

6. PRECISION

6.1 No data.

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ANALYSIS OF IRON BLUE PIGMENT-

1. SCOPE

1.1 This method covers procedures for the detection of impurities in the form of extenders and diluents in iron blue pigment either in the dry form or after extraction from paste-in-oil.

2. APPARATUS

2.1 Conventional laboratory apparatus and equipment shall be used in this test method.

3. REAGENTS

3.1 Sodium hydroxide solution (5 percent). Dissolve 1 part by weight of sodium hydroxide in 19 parts by weight of freshly distilled water.

3.2 Hydrochloric acid (1 to 1). Mix equal parts by volume of A.C.S. reagent grade hydrochloric acid and freshly distilled water.

3.3 Ammonium phosphate solution (10 percent). Dissolve 100 g of A.C.S. reagent grade dibasic ammonium phosphate in freshly distilled water and dilute to 1 liter.

3.4 Ammonium oxalate solution (4 percent). Dissolve 40 g of A.C.S. reagent grade ammonium oxalate, monohydrate, in warm, freshly distilled water and dilute to 1 liter.

3.5 Sodium hydroxide solution (10 percent). Dissolve 100 g of A.C.S. reagent grade sodium hydroxide in freshly distilled water and dilute to 1 liter.

3.6 Ammonium hydroxide solution (1 to 4). Mix 1 part by volume of A.C.S. reagent grade ammonium hydroxide with 4 parts by volume of freshly distilled water.

3.7 Ethyl alcohol. Ethyl alcohol (95 percent) or specially denatured alcohol formula 30 or 3A of the U.S. Bureau of Internal Revenue.

4. PROCEDURES

Examine the pigment in dry form or after its extraction from paste-in-oil by method 4021 as follows.

4.1 Identification. To approximately 0.1 g of the pigment in a 50 ml beaker add 15 ml of sodium hydroxide solution (see 3.1). Heat to boiling. In a few minutes the blue color should be completely destroyed, giving in its place the characteristic reddish brown precipitate of ferric hydroxide. Add hydrochloric

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acid (see 3.2) until the solution is faintly acid to litmus. The iron blue should be reformed, yielding again the characteristic blue color. If the sodium hydroxide does not completely destroy the blue color, the evidence is strong that a foreign pigment is present. In this case make the following tests.

4.2 Detection of acid-insoluble extenders. Acid-insoluble extenders include barium sulfate, silica, and silicates. Alumina may also be found in part, with this group.

4.2.1 Ignite 1 +0.01 g of the pigment in a glass beaker or porcelain dish over a low flame, just enough to decompose the last trace of pigment but not high enough to render the iron difficultly soluble in hydrochloric acid. During the ignition, roll the pigment around in the container so as to expose all of its surface to the oxygen of the air. Ignition is complete when pigment ceases to glow and acquires an uniform brown color. Allow to cool and add 15 ml of A.C.S. reagent grade hydrochloric acid. Add a few drops of A.C.S. reagent grade bromine, cover with a watchglass and evaporate to a syrup. Add 15 ml of water and boil. It may be necessary to add a drop or two of hydrochloric acid to effect complete solution of the ferric iron residue. Filter and wash with hot water. Examine the filtrate for alumina hydrate by the procedure given in 4.3.1.

4.2.2 Ignite the residue, cool and weigh. If the ignited residue weighs appreciably more than 3 mg, the presence of extenders should be suspected. If it is required to know which extenders are present, the residue may be examined for silica, barium sulfate and alumina.

4.3 Detection of acid-soluble extenders. Acid-soluble extenders include alkaline earth carbonates or sulfate, magnesium carbonate and alumina hydrate.

4.3.1 Alumina hydrate. Add sodium hydroxide solution (see 3.5) to the filtrate from 4.2.1 until it is just alkaline and then 5 ml in excess. Boil for about 2 minutes, let stand in a warm place until the hydrous iron oxide is coagulated. Filter through a rapid filter paper, and wash a few times with hot water. Discard the precipitate.

4.3.2 To the filtrate add 7 ml of hydrochloric acid (see 3.2). Make just alkaline with ammonium hydroxide (see 3.6). Boil about 2 minutes. If no precipitate is apparent let stand for about 1/2 hour. If solution is still clear, no alumina hydrate is present. A white gelatinous precipitate will indicate alumina hydrate. Filter and save the filtrate for the detection of alkaline earth and magnesium salts by the procedure given in 4.4.

4.3.3 If a rough estimate of the amount of alumina hydrate is desired, the residue may be washed, dried, ignited, cooled, and weighed as Al_2O_3 .

4.4 Alkaline earth and magnesium salts.

4.4.1 To the filtrate from 4.3.2 add hydrochloric acid (see 3.2) until it is faintly acid. Divide the filtrate into two equal portions.

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4.4.2 To one portion add 15 ml of the ammonium phosphate solution (see 3.3) and neutralize with A.C.S. reagent grade ammonium hydroxide and then add 10 ml in excess. If no precipitate forms immediately, let the solution stand for a short time in a cool place with occasional vigorous stirring and rubbing the inside of the beaker with a glass rod to initiate crystallization. A white microcrystalline precipitate indicates the presence of magnesium salts and possibly some alkaline earth salts as well.

4.4.3 To the other portion of the filtrate add 5 ml of ammonium oxalate solution (see 3.4). Make the solution slightly alkaline with A.C.S. reagent grade ammonium hydroxide. If no precipitate forms immediately, warm on a hotplate and let stand for a short time. A white microcrystalline precipitate indicates the presence of alkaline earth salts. If it is required to know specifically which acid-soluble extenders are present, any of the established tests for these metal salts may be employed.

4.5 Detection of organic colors and lakes. Make examination for these materials in accordance with procedure given in method 5021.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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ANALYSIS OF CEMENT-WATER PAINT POWDER

1. SCOPE

1.1 This method covers procedures for the analysis of both white and tinted powder (pigment) for use in cement-water paint.

2. APPARATUS

2.1 Conventional laboratory apparatus and equipment to be used for the purpose of this method.

3. REAGENTS

3.1 Nitric acid (1 to 5). Mix 1 part by volume of A.C.S. reagent grade nitric acid with 5 parts by volume of freshly distilled water.

3.2 Silver nitrate solution (2.5 percent). Dissolve 2.5 g of A.C.S. reagent grade silver nitrate in freshly distilled water and make up to 100-ml volume.

3.3 Bromine water. Prepared by saturating freshly distilled water with A.C.S. reagent grade bromine.

3.4 Ammonium oxalate solution (saturated). A saturated solution of A.C.S. reagent grade ammonium oxalate in freshly distilled water.

3.5 Sulfuric acid (1 to 5). Mix 1 part by volume of A.C.S. reagent grade sulfuric acid with 5 parts by volume of freshly distilled water.

3.6 Sulfuric acid (1 to 1). Mix equal volumes of A.C.S. reagent grade sulfuric acid and freshly distilled water.

3.7 Standard potassium permanganate solution (0.1 N).

3.7.1 Dissolve 3.161 g of A.C.S. reagent grade potassium permanganate in a liter of freshly distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: Transfer 0.3 g weighed to the nearest 0.1 mg of sodium oxalate, National Bureau of Standards, standard sample 40F, (dried for 1 hour at 105° C) to a 600-ml beaker. Add 250 ml of dilute sulfuric acid (see 3.13), previously boiled for 10 to 15 minutes and then cooled to 27° +3° C. Stir until the oxalate has dissolved. Add 39 to 40 ml of the potassium permanganate (0.3 g of the sodium oxalate requires 44.75 ml of 0.1 N potassium permanganate solution) at a rate of 25 to 35 ml per minute, while stirring slowly. Let stand until the pink color disappears (about 45 seconds) (see 5.1). Heat to 55°-60° C and complete the titration by adding the permanganate solution until a faint pink color persists for 30 seconds. Add the last 0.5 to 1 ml dropwise, with particular care to allow each drop to become decolorized before the next is introduced.

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3.7.2 Determine the excess of permanganate required to impart a pink color to the solution, by matching the color by adding the permanganate solution to the same volume of the sulfuric acid (see 3.13) at 55° C to 60° C. (This correction usually amounts to from 0.03 to 0.05 ml).

3.7.3 Calculate the normality of the permanganate solution using the following equation:

$$N = \frac{S}{0.06701 (A - B)}$$

where:

N = Normality of potassium permanganate solution.

S = Grams of sodium oxalate used.

A = Milliliters of potassium permanganate solution required for the sodium oxalate solution.

B = Milliliters of potassium permanganate solution required for the sulfuric acid blank.

3.7.4 The standard potassium permanganate solution shall be stored in a glass-stoppered bottle painted black to keep out light.

3.8 Neutral glycerol-alcohol solution (1 to 5). Prepare a solution consisting of 1 part by volume of A.C.S. reagent grade glycerol and 5 parts by volume of absolute ethyl alcohol or anhydrous alcohol denatured in accordance with formula 3a or formula 2b of the U.S. Bureau of Internal Revenue. To each liter of this solution add 2 ml of an indicator prepared by dissolving 1 gram of phenolphthalein in 100 ml of the absolute alcohol or anhydrous denatured alcohol. It is essential that the glycerol-alcohol solution be neutral to the indicator. If the solution is colorless, add a dilute alcoholic solution of sodium or potassium hydroxide until the pink color appears, and just remove this by a drop of an alcoholic solution of ammonium acetate. If the initial color is pink, just remove it by the alcoholic solution of ammonium acetate.

3.9 Standard alcoholic ammonium acetate solution (0.2 N). Dissolve 16 g of A.C.S. reagent grade ammonium acetate in 1 liter of absolute ethyl alcohol or anhydrous denatured alcohol (see 3.8). Standardize this solution by titrating against pure freshly prepared calcium oxide as follows.

3.9.1 Pulverize a few grains of calcite or calcium oxalate (ordinary c.p. calcium oxide shall not be used) to an impalpable powder and calcine by heating it in a platinum crucible at 900° C to 1,000° C to constant weight. Weigh to the nearest 0.1 mg about 0.1 g of the calcium oxide into a 200-ml Erlenmeyer flask containing 100 ml of the neutralized glycerol-alcohol solution (see 3.8) and approximately 0.5 g of A.C.S. reagent grade barium chloride. Disperse the calcium oxide in the solution by shaking. Insert a water or air reflux condenser in the top of the flask, and boil the mixture for 18 hours. Then

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remove the condenser and immediately titrate the solution, while near boiling, with the ammonium acetate solution. Replace the condenser and boil the solution again for 2 hours. If, after this boiling, the color does not reappear, the reaction may be considered complete. If the color does appear, titrate as above, adding this value to the first titration. Calculate the value of the solution in terms of grams of lime equivalent to each milliliter of standard ammonium acetate solution (see 3.9).

3.10 Hydrochloric acid (1 to 2). Mix 1 part by volume of hydrochloric acid (A.C.S. reagent grade) with 2 parts by volume of freshly distilled water.

3.11 Standard potassium ferrocyanide solution. Dissolve 22 g of A.C.S. reagent grade potassium ferrocyanide in freshly distilled water and dilute to 1,000 ml. (A.C.S. reagent grade sodium sulfite may be added to stabilize the solution in the proportion of 7 g per liter of solution.) To standardize, transfer about 0.2 g weighed to the nearest 0.1 mg of A.C.S. reagent grade metallic zinc to a 400-ml beaker. Dissolve in 30 ml of hydrochloric acid (see 3.10). Drop in a small piece of litmus paper, add the ammonium hydroxide (see 3.16) until slightly alkaline. Add the hydrochloric acid, until just acid, and then add 3 ml in excess. Dilute to about 250 ml with hot water and heat nearly to boiling. Run in the ferrocyanide solution slowly from a burette with constant stirring until a drop tested on a white porcelain plate with a drop of uranyl indicator (see 3.12) shows a brown tinge after standing about 1/2 minute. Run a blank with the same amounts of reagents and water. Calculate and record for use in procedures the grams of zinc oxide equivalent to 1 milliliter of the solution as follows:

$$F = \frac{S \times 1.2447}{A - B}$$

where:

- F = Zinc oxide equivalent of the standard potassium ferrocyanide solution in grams per milliliter.
- S = Grams of metallic zinc taken.
- A = Milliliters of potassium ferrocyanide solution required for titration of zinc.
- B = Milliliters of potassium ferrocyanide solution required for titration of blank.

3.12 Uranyl indicator solution. A 5 percent solution of A.C.S. reagent grade uranyl nitrate in freshly distilled water or a 5 percent solution of A.C.S. reagent grade uranyl acetate in water and made slightly acid with A.C.S. reagent grade glacial acetic acid.

3.13 Sulfuric acid (1 to 19). Mix 5 parts by volume of A.C.S. reagent grade sulfuric acid with 95 parts by volume of freshly distilled water.

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3.14 Hydrochloric acid (1 to 1). Mix equal parts by volume of A.C.S. reagent grade hydrochloric acid and freshly distilled water.

3.15 Ammonium phosphate (dibasic) solution (saturated). Saturate freshly distilled water with A.C.S. reagent grade dibasic ammonium phosphate.

3.16 Ammonium hydroxide. A.C.S. reagent grade.

3.17 Acetic acid (3 percent). Mix 3 parts by volume of A.C.S. reagent grade of glacial acetic acid with 97 parts by volume of freshly distilled water.

3.18 Sulfuric acid. A.C.S. reagent grade.

3.19 Ammonium sulfate. A.C.S. reagent grade.

3.20 Ferric sulfate solution. A solution containing 2 percent of iron as ferric sulfate is required and may be prepared as follows: Dissolve 20 g of pure iron or plain carbon steel in a slight excess of hydrochloric acid (see 3.22), oxidize with A.C.S. reagent grade nitric acid, add approximately 80 ml of sulfuric acid (see 3.18) and heat until fumes of the latter are evolved. Cool, dilute with distilled water to 1,000 ml, digest on a steam bath until the sulfates are dissolved, and filter if necessary. Add the potassium permanganate solution (see 3.7) until a faint pink color is obtained to oxidize any ferrous iron that may be present.

3.21 Phosphoric acid (85 percent). A.C.S. reagent grade.

3.22 Hydrochloric acid. A.C.S. reagent grade.

4. PROCEDURE

4.1 Qualitative analysis. The following preliminary tests shall be made on the cement-water paint powder.

4.1.1 Organic binder. Test for organic binder by placing a small amount (1 to 3 grams) of the sample in an evaporating dish and gently heating it over an open flame. Only a trace of smoke having an oily odor should be given off. If appreciable smoke is noted, mix 25 ml of the sample with 25 ml of water and let stand for 30 minutes. Stir and brush on a clean tinplate (see method 2012). Allow to dry 1 hour. If the paint bonds to the impervious surface, organic binder is present.

4.1.2 Water repellents. To test for the presence of water repellents (calcium or aluminum stearate) place about 100 ml of cement-water paint powder in a 250-ml beaker, and add an equal volume of water without stirring. The paint powder should repel the wetting action of the water for several seconds. Then mix the powder and water and allow the mixed sample to stand for 10 minutes. A light scum floating on the surface of the water is indicative of the presence of water repellent stearates.

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4.1.3 Hygroscopic salts. Hygroscopic salts (calcium or sodium chloride) may be detected by precipitating the water-soluble chlorides with the silver nitrate solution (see 3.2).

4.1.4 Chlorides. Mix approximately 2 g of the sample with 30 ml of water and allow to stand 30 minutes. Filter the supernatant liquid and make slightly acid with dilute nitric acid (see 3.1). Then add approximately 5 ml of the silver nitrate solution. A white precipitate (silver chloride) indicates the presence of chlorides.

4.1.5 Zinc sulfide and titanium dioxide. Use regular qualitative test procedures to determine whether the paint powder contains zinc sulfide or titanium dioxide or a mixture of these.

4.2 Quantitative tests. The following tests shall be made on class A paint powder of both types as received. In case of class B material of either type the analysis shall be made only on that portion passing a No. 100 sieve (see 4.2.1) and the computations shall also be based on this portion and not on the sample as received.

4.2.1 Siliceous aggregate, types I and II, class B only. Weigh to the nearest 0.1 g approximately 20 g of the sample which has been carefully mixed, and transfer to a weighed No. 100 sieve. Sieve the material dry, by gently tapping the sides of the sieve, collecting the portion passing through in an evaporating dish. When the bulk of the pigment has been separated from the aggregate, agitate the portion remaining on the sieve by brushing with a camel's hair brush until nothing more passes through. Preserve the portion that has passed through for examination under 4.2.2 to 4.2.9 inclusive. Finally, wash the sieve and aggregate with a gentle stream of water, discarding the washings. Dry the sieve in an oven at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ($221^{\circ}\text{F} \pm 3.6^{\circ}\text{F}$) for 1 hour. Cool, weigh, and calculate as percent of siliceous aggregate. The aggregate shall then be transferred from No. 100 sieve to No. 20 sieve and washed with water, using a camel's-hair brush, if necessary. All the aggregate shall pass the No. 20 sieve.

4.2.2 Total calcium oxide.

4.2.2.1 Mix about 0.5 g, weighed to the nearest 0.1 mg, of the sample, with 0.5 g of A.C.S. reagent grade ammonium chloride in a 50-ml beaker. Add 5 ml of A.C.S. reagent grade hydrochloric acid, cover and heat on the steam bath for 15 minutes. Transfer the gel to a filter paper or Gooch crucible and wash the insoluble residue thoroughly with hot water. Discard the residue.

4.2.2.2 To the filtrate add 8 to 10 g of the ammonium chloride. Bring to near boiling, add a few ml of bromine water (see 3.3) and make alkaline with A.C.S. reagent grade ammonium hydroxide. Let the precipitate settle, filter, and wash thoroughly with hot water. Concentrate the filtrate and washings from the iron, aluminum, etc., to about 200 ml adding a few drops of ammonium

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hydroxide. Boil and add while boiling 25 to 30 ml of a hot saturated solution of ammonium oxalate (see 3.4), stir and continue the boiling until the precipitated calcium oxalate becomes granular. Set on a steam bath to settle (about 1 hour). Filter and wash the oxalate thoroughly with small amounts of hot water. Save the filtrate for the magnesium oxide determination (see 4.2.6).

4.2.2.3 Transfer the moist precipitate to a 400-ml beaker by means of a stream of water from the wash bottle, dissolve the part remaining on the filter by washing with warm dilute sulfuric acid (see 3.5). Add to the beaker 20 ml of dilute sulfuric acid (see 3.6), dilute to about 300 ml with hot water, and titrate the oxalic acid with the standard potassium permanganate solution (see 3.7).

4.2.2.4 Calculation. Calculate the amount of calcium oxide (CaO) present in the sample taken for test on the basis that 1 ml of 0.1 N KMnO_4 = 0.0028 g CaO. Record this value for use in 4.3.1.

4.2.3 Free lime.^{1/} The determination is based on the solution of the uncombined lime in a hot solution of glycerol and alcohol and the subsequent titration of the dissolved lime with an alcoholic solution of ammonium acetate in accordance with the reaction.



4.2.3.1 White paint-powder. Weigh to the nearest 0.1 mg about 0.5 g of the sample and transfer it to a 200-ml Erlenmeyer flask containing 100 ml of the glycerol-alcohol solution (see 3.8) and 0.5 g of A.C.S. reagent grade barium chloride. Attach a reflux condenser and boil the mixture on a hotplate for 18 hours. Titrate with the standard alcoholic ammonium acetate solution following the procedure given in its standardization (see 3.9.1). Replace the condenser and boil again for 2 hours. If the color reappears, titrate with additional ammonium acetate solution, adding this value to that of the first titration. If there is doubt of the exact end point, add a drop of indicator to the quiescent liquid in the flask, carefully observing the point at which the indicator strikes the solution. If no color appears, the end point has been reached. Calculate the free lime content of the sample taken from the known value of the ammonium acetate used.

4.2.3.2 Tinted paint-powder. Proceed as in 4.2.3.1 until the sample solution has been boiled under reflux for 18 hours. Now filter through a coarse filter or Gooch crucible with filter paper pad into an Erlenmeyer flask. Keep the residue covered with liquid to prevent carbonation from air and ingress of

1/ This method is a modification of the "Revised Procedure for the Determination of Uncombined Lime in Portland Cement" by William Lerch and R. N. Bogue, Paper 23, Cooperative Investigations by the Portland Cement Association and the National Bureau of Standards, July 1930.

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carbon dioxide. Wash the residue with absolute ethyl alcohol or anhydrous denatured alcohol (see 3.8). Attach a reflux condenser to the flask containing the filtrate, and boil on the hotplate for 2 minutes. Then titrate the boiling filtrate with the standard alcoholic ammonium acetate solution (see 3.9). Place the residue (without removing from the filter paper) in a 200-ml Erlenmeyer flask containing 60 ml of the glycerol-alcohol solution (see 3.8), attach reflux condenser, and boil for 2 hours. If after this boiling no color appears in the solution containing the residue, the reaction may be considered complete. If the color does appear, filter, and wash as above. Boil the filtrate and titrate with additional alcoholic ammonium acetate, adding this value to that of the first titration. Calculate the free lime as in 4.2.3.1.

4.2.4 Zinc sulfide. Weigh to the nearest 0.1 mg about 1 g of the sample, transfer to a 400-ml beaker. Add 30 ml of dilute hydrochloric acid (see 3.10) and boil for 2 to 3 minutes. Add 200 ml of water and a small piece of litmus paper. Add A.C.S. reagent grade ammonium hydroxide until slightly alkaline. Render just acid with A.C.S. reagent grade hydrochloric acid and then add 3 ml of the hydrochloric acid in excess. Heat nearly to boiling, and titrate with standard potassium ferrocyanide solution as in standardizing that solution (see 3.11). When (as is generally the case) the material contains only inappreciable amounts of zinc in any form other than ZnS, the total shall be computed as ZnS (ZnO factor for ferrocyanide solution $\times 1.2 = \text{ZnS}$). When other compounds of zinc are present in appreciable amounts, take another accurately weighed portion of about 1 g, add 100 ml of 3 percent acetic acid, stir vigorously, but do not heat, cover, and let stand for 18 hours, stirring once every 5 minutes for the first half hour. Filter, wash with dilute acetic acid, and titrate the zinc in the clear filtrate. Compute as ZnS and subtract from the total zinc as ZnS determined above.

4.2.5 Titanium dioxide. When titanium dioxide is present, weigh to the nearest 0.1 mg about 1 g of the sample and transfer to a 250-ml pyrex beaker. Carefully crush any lumps with a rounded glass rod, add 25 ml of A.C.S. reagent grade sulfuric acid and 8 g of A.C.S. reagent grade ammonium sulfate. Mix well and heat on a hotplate until fumes of sulfuric acid are evolved, and then continue the heating over a strong flame until solution is complete (usually not over 5 minutes of boiling), or it is apparent that the residue is composed of silica or siliceous matter. Caution should be observed in handling this hot acid solution. Cool the solution, dilute with 100 ml of water, stir, heat carefully to boiling while stirring, let settle, filter through paper, and transfer the precipitate completely to the paper.

Wash the insoluble residue with cold sulfuric acid (see 3.13) until titanium is removed. Dilute the filtrate to 200 ml and add about 10 ml of ammonium hydroxide (see 3.16) to lower the acidity to approximately 5 percent by volume of sulfuric acid. Warm the solution to $40^{\circ}\text{C} + 5^{\circ}\text{C}$ and add the standard solution of potassium permanganate (see 3.7) to a pink coloration.

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Wash out a Jones reductor with sulfuric acid (see 3.13) and water leaving sufficient water in the reductor to fill to the upper level of the zinc. (These washings should require not more than one or two drops of the potassium permanganate solution to obtain a pink color.) Empty the receiver and put into it 25 ml (measured in a graduate) of ferric sulfate solution (see 3.20). Reduce the prepared titanium solution as follows:

1. Run 50 ml of the sulfuric acid solution (see 3.13) through the reductor at a speed of about 100 ml per minute.
2. Follow this with the titanium solution and draw through at the same speed.
3. Wash the beaker and reductor with 100 ml of sulfuric acid (see 3.13) and finally with 100 to 150 ml of distilled water.
4. Close the stopcock on the reductor.
5. Care should be taken that the reductor is always filled with solution or water to the upper level of the zinc. Gradually release the suction, raise the reductor above the receiver, and rinse the exit tube of the reductor internally (by opening the stopcock on the reductor and allowing 15 to 20 ml of water to pass through) and externally with water.

Titrate the solution with the potassium permanganate solution (see 3.7) until within 3 to 5 ml of the endpoint (approximately 38 to 40 ml), add 10 ml of phosphoric acid (see 3.21), and continue the titration to a permanent pink coloration.

Compute the percentage of titanium dioxide (see 5.2) in the pigment using the following equation:

$$\text{Percent TiO}_2 = \frac{(A - B) \times N \times 0.0799}{S} \times 100$$

where:

- A = Milliliters of standard potassium permanganate solution used to titrate sample.
 B = Milliliters of standard potassium permanganate solution used to titrate blank.
 N = Normality of potassium permanganate solution.
 S = Grams of sample used.

4.2.6 Total magnesium oxide. Acidify the filtrate from the calcium determination with A.C.S. reagent grade hydrochloric acid. Concentrate on the steam bath to about 150 ml and make slightly alkaline with A.C.S. reagent grade ammonium hydroxide. Boil and filter (to remove a little iron and aluminum, and perhaps calcium). When cool, acidify the solution with the hydrochloric acid and add 10 ml of saturated solution of dibasic ammonium phosphate (see 3.15). Add A.C.S. reagent grade ammonium hydroxide drop by drop, with constant stirring. When the crystalline ammonium-magnesium orthophosphate has formed, add 10 ml of the ammonium hydroxide in excess. Set the solution aside overnight in a cool

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place, filter, and wash with water containing 2.5 percent NH_3 . Dissolve the precipitate in a small quantity of the hot hydrochloric acid and dilute the solution to about 100 ml. Add 1 ml. of ammonium phosphate solution and the ammonium hydroxide drop by drop, with constant stirring, until the precipitate is again formed as described above and the ammonium hydroxide is in moderate excess. Allow the precipitate to stand about 2 hours, filter and wash as before. Place the paper and contents in a weighed platinum crucible, char the paper slowly, and carefully burn off the resulting carbon. Ignite the precipitate to constant weight over a Meker burner, or a blast not strong enough to soften or melt the pyrophosphate. Multiply this weight by 0.362 to find the weight of magnesium oxide.

4.2.7 Carbon dioxide. Weigh to the nearest mg about 5 g of the sample and transfer it to a small Erlenmeyer flask. Put this flask in a carbon dioxide train composed as follows: After the flask comes an upwardly inclined condenser, then U-tubes containing CaCl_2 , anhydrous CuSO_4 , CaCl_2 , and porous soda lime, and finally a U-tube containing half soda lime and half CaCl_2 , arranged so that the CaCl_2 comes last. Pour some hot water on the sample, connect the flask in the train and pass a current of carbon dioxide-free air through all of the train excepting the soda-lime tubes. Weigh and attach the soda-lime tubes. Through a separatory funnel, let some dilute hydrochloric acid (see 3.14) into the flask slowly. When any apparent action has ceased, heat the flask gradually to boiling, and continue boiling until no more gas is evolved. Cool gradually. The current of carbon dioxide-free air is continued throughout the experiment. Detach the soda-lime tubes, close them, and let them stand in the balance case, weighing them at 30-minute intervals until two successive weighings agree within 0.5 mg. Record the gain in weight of the soda-lime tubes as the weight of the carbon dioxide.

4.2.8 Loss on ignition. Heat about 1 g of the sample weighed to the nearest 0.1 mg in a weighed covered platinum or porcelain crucible of 20- to 25-ml capacity, in an electric furnace at a temperature of 750° C for 30 minutes. Cool in a desiccator and weigh.

4.2.9 Free or extraneous water. Weigh to the nearest mg about 1 g of the sample into a tared platinum or porcelain crucible and dry for 30 minutes in an electric furnace, or one protected from gas fumes; maintained at a temperature of 350° C. Cool in a desiccator and weigh rapidly. The loss in weight gives the weight of free or extraneous water.

4.3 Computations. Compute the percentages of portland cement, hydrated lime, and the percent of the sum of unhydrated calcium oxide and magnesium oxide in the hydrated lime, in the sample tested (see 4.2), as follows.

4.3.1 Portland cement. Compute the amount of calcium oxide combined with carbon dioxide (see 4.2.7) and add the amount of free lime (see 4.2.3). Subtract this sum from the total calcium oxide (see 4.2.2.4) and multiply the

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difference by 1.6. (This factor is based on the assumption that the portland cement contains 62.5 percent calcium oxide.) Report this value as the percentage of portland cement.

4.3.2 Total hydrated lime and the percent of unhydrated calcium oxide and magnesium oxide in hydrated lime.

4.3.2.1 From total magnesium oxide value (see 4.2.6) subtract 2.5 percent of the computed portland cement (see 4.3.1) to determine the magnesium oxide in the hydrated lime. (This is to make allowance for magnesium oxide which may be present in the portland cement.)

4.3.2.2 From the loss on ignition (see 4.2.8) subtract the sum of free water (see 4.2.9) and carbon dioxide (see 4.2.7) to determine the percentage of combined water.

4.3.2.3 Multiply the free lime (see 4.2.3) by 0.321 to determine the percentage of water combined with calcium oxide.

4.3.2.4 If the result in 4.3.2.3 is less than the combined water (see 4.3.2.2), proceed as in 4.3.2.5 to 4.3.2.10 inclusive.

4.3.2.4.1 If the result in 4.3.2.3 is greater than the combined water (see 4.3.2.2), proceed as in 4.3.2.11 to 4.3.2.14 inclusive.

4.3.2.4.2 If the result in 4.3.2.3 is equal to the combined water (see 4.3.2.2), compute total lime as in 4.3.2.9 and unhydrated lime as in 4.3.2.15.

4.3.2.5 Subtract the value obtained in 4.3.2.3 from the combined water (see 4.3.2.2). This gives the percentage of water combined with magnesium oxide.

4.3.2.6 Multiply the value obtained in 4.3.2.5 by 2.238 to determine the percentage of hydrated magnesium oxide.

4.3.2.7 Subtract percentage hydrated magnesium oxide (see 4.3.2.6) from magnesium oxide in the hydrated lime (see 4.3.2.1) to obtain percentage unhydrated magnesium oxide.

4.3.2.8 Compute carbon dioxide to calcium carbonate by multiplying by 2.27.

4.3.2.9 The sum of free lime (see 4.2.3), calcium carbonate (see 4.3.2.8), magnesium oxide in hydrated lime (see 4.3.2.1), and combined water (see 4.3.2.2), gives the total hydrated lime.

4.3.2.10 The percentage unhydrated magnesium oxide (see 4.3.2.7) divided by total hydrated lime (see 4.3.2.9), multiplied by 100, gives the percentage of unhydrated magnesium oxide and calcium oxide in the mixture.

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4.3.2.11 To determine the percentage of hydrated calcium oxide, multiply the percentage of combined water (see 4.3.2.2) by 3.11.

4.3.2.12 To determine the percentage of unhydrated calcium oxide and magnesium oxide, subtract the percentage hydrated calcium oxide (see 4.3.2.11) from the sum of free lime (see 4.2.3) and magnesium in hydrated lime (see 4.3.2.1).

4.3.2.13 Same as computation in 4.3.2.9.

4.3.2.14 Divide percentage of unhydrated calcium oxide and magnesium oxide (see 4.3.2.12) by the total hydrated lime (see 4.3.2.9) to obtain the percentage of unhydrated calcium oxide and magnesium oxide in mixture.

4.3.2.15 Divide magnesium oxide in hydrated lime (see 4.3.2.1) by the total hydrated lime (see 4.3.2.9) to obtain percentage of unhydrated calcium oxide and magnesium oxide in mixture.

5. NOTES

5.1 If the pink color should persist because the permanganate is in excess, discard and begin again, adding a few milliliters less of the permanganate solution.

5.2 Iron, chromium, arsenic, and any other substances reducible by zinc and acid will be included in this value. However, appreciable quantities of interfering materials are not likely to be encountered in normal, white, titanium dioxide pigments.

6. PRECISION

6.1 No data.

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ANALYSIS OF PIGMENT EXTRACTED FROM WHITE-BASE,
 COMPOSITE-PIGMENT EXTERIOR PAINTS

1. SCOPE

1.1 This method describes the analysis of composite white or white-base pigments extracted from exterior paints.

2. APPARATUS

2.1 Standard laboratory apparatus and equipment are used in this method.

3. REAGENTS

3.1 Acid ammonium acetate solution. The solution shall be composed of the following:

Glacial acetic acid - - - - -	120 ml
Ammonium hydroxide - - - - -	95 ml
Distilled water - - - - -	130 ml

It shall be prepared by adding approximately one-half of the water to the glacial acetic acid and the remainder to the ammonium hydroxide and then slowly combining the two diluted solutions while the mixture is cooled by running water.

3.2 Potassium dichromate solution, 10 percent. Dissolve 100 g of potassium dichromate in 900 ml of water.

3.3 Ethyl alcohol, 95 percent.

3.4 Nitric acid, 5 percent. Mix 1 volume of nitric acid and 19 volumes of water.

3.5 Barium chloride, 10 percent. Dissolve 117 g of barium chloride in water and dilute to 1 liter.

3.6 Bromine water. Prepare by saturating 100 ml of water with bromine.

3.7 Hydrochloric acid, concentrated.

3.8 Hydrochloric acid, 50 percent. Mix equal parts by volume of hydrochloric acid and water.

3.9 Ammonium chloride.

3.10 Sodium carbonate.

3.11 Hydrochloric acid, 33 percent. Mix 1 part by volume of hydrochloric acid with 2 parts by volume of water.

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3.12 Ammonium hydroxide, concentrated.

3.13 Standard potassium ferrocyanide solution. Dissolve 22 g of potassium ferrocyanide in water and dilute to 1 liter. (Sodium sulfite [7 g per liter] may be added to stabilize the solution.) To standardize, transfer about 0.2 g, weighed to the nearest 0.1 mg, of metallic zinc to a 400-ml beaker. Dissolve in 30 ml of 33 percent hydrochloric acid. Drop in a small piece of litmus paper, and add ammonium hydroxide until slightly alkaline. Add the hydrochloric acid until just acid, and then add 3 ml in excess. Dilute to about 250 ml with hot water and heat nearly to boiling. Add the ferrocyanide solution slowly from a burette with constant stirring until a drop tested on a white porcelain plate with a drop of the uranyl indicator shows a brown tinge after standing about 30 seconds. Run a blank with the same amounts of reagents and water. Calculate and record for use in 4.2.3.1 the weight of zinc oxide equivalent to 1 ml of the solution as follows:

$$F = \frac{S \times 1.2447}{A - B}$$

where:

F = Zinc oxide equivalent of the standard potassium ferrocyanide solution in grams per ml.

S = Grams of metallic zinc.

A = Milliliters of potassium ferrocyanide solution required for titration of the zinc.

B = Milliliters of potassium ferrocyanide solution required for titration of the blank.

3.14 Uranyl indicator, 5 percent. Dissolve 5 g of uranyl nitrate in 95 ml of water.

3.15 Standard potassium permanganate, 0.1 N.

3.15.1 Dissolve 3.2 g of potassium permanganate in a liter of water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: Transfer 0.3 g, weighed to the nearest 0.1 mg, of sodium oxalate, National Bureau of Standards standard reference material 40 (dried for 1 hour at 105° C) to a 600-ml beaker. Add 250 ml of 5 percent sulfuric acid (previously boiled for 10 to 15 minutes and then cooled to 27° C). Stir until the oxalate is dissolved. Add 30 to 40 ml of the potassium permanganate (0.3 g of the sodium oxalate required 44.75 ml of 0.1 N potassium permanganate solution) at a rate of 25 to 35 ml per minute, while stirring slowly. Let stand until the pink color disappears (about 45 seconds) (see 5.1). Heat to 55° C to 60° C and complete the titration by adding the permanganate solution until a faint pink color persists for 30 seconds. Add the last 0.5 to 1 ml dropwise, with particular care to allow each drop to become decolorized before the next is introduced.

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3.15.2 Determine the excess of permanganate required to impart a pink color to the solution, by adding the permanganate solution to an equal volume of sulfuric acid at 55° C to 60° C until a color match is obtained. (This correction varies from 0.03 to 0.05 ml.)

3.15.3 Calculate the normality of the permanganate solution using the following equation:

$$N = \frac{S}{0.06701 (A - B)}$$

where:

N = Normality of potassium permanganate solution.

S = Grams of sodium oxalate used.

A = Milliliters of potassium permanganate solution required for the sodium oxalate solution.

B = Milliliters of potassium permanganate solution required for the sulfuric acid blank.

3.15.4 The standard potassium permanganate solution shall be protected from light.

3.16 Ferric sulfate, 2 percent. A solution containing 2 percent of iron as ferric sulfate is required and may be prepared as follows: Dissolve 20 g of pure iron or plain carbon steel in a slight excess of 50 percent hydrochloric acid, oxidize with nitric acid, add approximately 80 ml of concentrated sulfuric acid, and heat until fumes of sulfur trioxide are evolved. Cool, dilute with water to 1 liter, digest on a steam bath until the sulfates are dissolved, and filter if necessary. Add 0.1 N potassium permanganate solution until a faint pink color is obtained (to oxidize any ferrous iron that may be present).

3.17 Potassium dichromate solution, 10 percent. Dissolve 100 g of potassium dichromate in 900 ml of freshly distilled water.

3.18 Ammonium sulfate.

3.19 Sulfuric acid, concentrated.

3.20 Sulfuric acid, 5 percent. Mix 5 parts by volume of sulfuric acid with 95 parts by volume of water.

3.21 Phosphoric acid, 85 percent.

4. PROCEDURE

Examine the pigment extracted from the coating material by method 4021 as follows.

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4.1 Qualitative analysis. Test the pigment by regular qualitative procedures to determine the presence of materials not permitted by the product specification.

4.2 Quantitative analysis.

4.2.1 Total "white lead".

4.2.1.1 Total lead (as lead oxide). Weigh to the nearest 0.1 mg about 1 g of pigment, transfer to 250 ml beaker, moisten with a few drops of alcohol, then add 50 ml of the acid ammonium acetate solution and digest at a slow boil for 4 minutes. Decant through a Gooch crucible prepared in the usual manner, leaving any undecomposed matter in the beaker. Wash the crucible with about 20 ml of hot water. To the residue in the beaker add 50 ml of the acid ammonium acetate solution, and boil again for 4 minutes. Filter through the same crucible, transferring the insoluble matter to the crucible, and wash thoroughly with hot water. Dilute the filtrate to about 200 ml and bring to a slow boil. Add dropwise until precipitation starts and then in a slow stream from 10 to 15 ml of the 10 percent potassium dichromate solution. Continue boiling until the yellow precipitate assumes an orange color. Let it settle and filter on a weighed Gooch crucible, washing by decantation with hot water until the washings are colorless, and finally transferring all the precipitate. Dry the crucible at 105° C, cool in a desiccator, and weigh (see 5.1). Convert the lead chromate to lead oxide using the following equation:

$$\text{Percent PbO} = \frac{\text{Weight of PbCrO}_4 \times 0.69}{\text{Weight of sample}} \times 100 \quad (1)$$

4.2.1.2 Lead sulfate. Weigh to the nearest 0.1 mg about 1 g of pigment into a 400 ml beaker. Moisten with a few drops of alcohol, add 10 ml of bromine water, 10 ml of 50 percent hydrochloric acid, and 3 grams of ammonium chloride. Cover with a watchglass and heat on a steam bath for 5 minutes. Add hot water to give a total volume of about 200 ml. Boil for 5 minutes and filter. Wash thoroughly with hot water. Neutralize the clear filtrate in a covered beaker with solid sodium carbonate. Add 1 g of solid sodium carbonate, and boil for 10 to 15 minutes. Let the precipitate settle, filter, and wash it with hot dilute aqueous sodium carbonate. Redissolve the precipitate in 50 percent hydrochloric acid and reprecipitate with sodium carbonate as above. Filter and wash thoroughly with hot water as above. Cautiously acidify the combined filtrates with concentrated hydrochloric acid, adding about 1 ml in excess. Boil to expel all bromine and carbon dioxide from the solution. To the clear boiling filtrate add slowly, while stirring, 15 ml of the barium chloride solution. Let stand on a steam bath overnight. Filter on a weighed Gooch crucible previously prepared in the usual manner, wash thoroughly with boiling water, dry, ignite, cool in a desiccator, and weigh as barium sulfate. Convert the barium sulfate to lead sulfate using the factor 1.3, and calculate the lead oxide equivalent as follows:

$$\text{Percent PbO} = \frac{\text{Weight of PbSO}_4 \times 0.736}{\text{Weight of sample}} \times 100$$

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4.2.1.3 Lead carbonate. Weigh to the nearest 0.1 mg about 2 g of pigment and transfer to a clean, dry Knorr carbon dioxide evolution flask. Connect the flask to an absorption train which has been previously flushed free of carbon dioxide. Add 100 ml of the 5 percent nitric acid through a separatory funnel to the contents of the flask. When all the acid has been added, close the stopcock of the funnel and heat the contents of the flask to a gentle boil. Boil for 5 minutes, remove the heat and aspirate carbon dioxide-free air through the system for 20 minutes. Remove the absorption tube from the system, seal, and weigh. The increase in weight is carbon dioxide. Convert carbon dioxide to lead carbonate and lead oxide using the following equations:

$$\text{Percent PbCO}_3 = \frac{\text{Weight of CO}_2 \times 6.072}{\text{Weight of sample}} \times 100 \quad (2)$$

$$\text{Percent PbO} = \frac{\text{Weight of CO}_2 \times 5.072}{\text{Weight of sample}} \times 100 \quad (3)$$

4.2.1.4 Computation. Multiply the percentage of basic lead carbonate found in 4.2.1.3 by the factor 0.863 to determine its equivalent in terms of lead oxide. Add the lead oxide equivalents of the basic lead carbonate (see 4.2.1.3) and lead sulfate (see 4.2.1.2) together and subtract this sum from the lead oxide equivalent of the total lead (see 4.2.1.1). Add this difference to the lead sulfate found in 4.2.1.2 to obtain the amount of basic lead sulfate. Finally, add this amount of basic lead sulfate to the basic lead carbonate determined in 4.2.1.3 and report as percentage of "white lead".

4.2.2 Titanium dioxide. Determine the titanium dioxide content carrying a blank through all the steps as follows.

4.2.2.1 Weigh to the nearest 0.1 mg about 0.35 g of pigment, (see 5.2) and transfer to a 250-ml beaker. Add 10 g of ammonium sulfate and 20 ml of concentrated sulfuric acid. Mix well and heat until fumes of sulfur trioxide are evolved. Boil gently until solution is complete (usually not over 5 minutes of boiling is required) or it is apparent that the residue is composed of silica or siliceous matter. Caution should be observed in visually examining this hot solution. Cool the solution, dilute with 100 ml of water, heat carefully to boiling while stirring, let settle, filter through paper and transfer the residue completely to the paper. Wash the insoluble residue with cold 5 percent sulfuric acid until titanium dioxide is removed. Dilute the filtrate to 200 ml and add 10 ml of concentrated ammonium hydroxide. Warm the solution to 40° C, and add 0.1 N potassium permanganate to a pink coloration.

4.2.2.2 Wash out a Jones reductor (see 5.3) with 5 percent sulfuric acid and water, leaving sufficient water in reductor to fill to the upper level of the zinc. (These washings should require not more than one or two drops of the potassium permanganate solution to obtain a pink color.) Empty the receiver and put into it 25 ml of ferric sulfate solution. Reduce the prepared titanium solution as follows.

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4.2.2.3 Run 50 ml of 5 percent sulfuric acid solution through the reductor at a speed of about 100 ml per minute.

4.2.2.4 Follow this with the titanium solution, and draw through at the same speed.

4.2.2.5 Wash the beaker and reductor with 100 ml of 5 percent sulfuric acid and finally with 100 to 150 ml of water.

4.2.2.6 Close the stopcock on the reductor.

4.2.2.7 Care should be taken that the reductor is always filled with solution or water to the upper level of the zinc. Gradually release the suction, raise the reductor above the receiver, and rinse the exit tube of the reductor internally (by opening the stopcock on the reductor and allowing 15 to 20 ml of water to pass through) and externally with water.

4.2.2.8 Titrate the solution with the 0.1 N potassium permanganate solution until within 3 to 5 ml of the endpoint (approximately 38 to 40 ml), add 10 ml of phosphoric acid, and continue the titration to a permanent pink coloration.

4.2.2.9 Compute the percentage of titanium dioxide in the pigment using the following equation:

$$\text{Percent TiO}_2 = \frac{(A - B) \times N \times 0.0799}{S} \times 100$$

where:

A = Milliliters of standard potassium permanganate solution used to titrate the sample.

B = Milliliters of standard potassium permanganate solution used to titrate the blank.

N = Normality of potassium permanganate solution.

S = Grams of sample used.

4.2.3 Zinc oxide.

4.2.3.1 Weigh to the nearest 0.1 mg about 0.25 g of pigment and transfer to a 400-ml beaker. Add 30 ml of 33 percent hydrochloric acid, boil for 2 or 3 minutes, and add 200 ml of water and 3 drops of methyl orange indicator. Add ammonium hydroxide until slightly alkaline. If iron is found to be present, it shall be removed at this point by a double precipitation with ammonium hydroxide. Neutralize the ammoniacal solution with the hydrochloric acid, and add 3 ml in excess. Heat nearly to boiling, and titrate with standard ferrocyanide as in standardizing that solution. Run a blank using the same amounts of reagents and water.

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4.2.3.2 Calculate the total zinc as percentage of zinc oxide in the pigment using the following equation:

$$\text{Percent ZnO} = \frac{(A - B) \times F}{S} \times 100$$

where:

- A = Milliliters of standard potassium ferrocyanide solution used for titration of the sample.
- B = Milliliters of standard potassium ferrocyanide solution used for titration of the blank.
- F = Zinc oxide equivalent of the standard potassium ferrocyanide solution in g per ml (see 3.13).
- S = Grams of sample used.

4.2.4 Tinting and extender pigments. Obtain the percentage of tinting and extender pigments by subtracting the sum of the percentage of "white lead" (see 4.2.1.4), titanium dioxide (see 4.2.2), and zinc oxide (see 4.2.3) from 100.

5. NOTES

5.1 If the pink color should persist because the permanganate is in excess, discard and begin again, adding a few ml less of the permanganate solution.

5.2 In the case of pigment extracted from tinted paint, first transfer to a 250-ml beaker, add 30 ml of 50 percent hydrochloric acid, boil, dilute with hot water, and let the precipitate settle. Filter the precipitate, wash it thoroughly with hot water, burn off the filter paper in a crucible over a low flame, and perform the analysis on the residue.

5.3 Directions for preparing a Jones reductor may be found in Hillebrand, Lundell, Bright, and Hoffman. "Applied Inorganic Analysis", Second Edition (1953), p. 108. It is recommended that the reductor have a bore of 19 mm and a zinc column of 45 cm in length.

6. PRECISION

6.1 No data.

METHOD 7271
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ANALYSIS OF PIGMENT EXTRACTED FROM CHROME YELLOW AND
CHROME ORANGE PAINTS

1. SCOPE

1.1 This method covers procedures for the analysis of chrome orange and chrome yellow pigments extracted from paints.

2. APPARATUS

2.1 Beaker. A 400-ml beaker.

2.2 Steam bath.

2.3 Porcelain crucible.

2.4 Desiccator.

2.5 Gooch crucible.

2.6 Electric hotplate.

3. REAGENTS

3.1 Nitric acid (1 to 3). Mix 1 part by volume of A.C.S. reagent grade nitric acid with 3 parts by volume of freshly distilled water.

3.2 Ethyl alcohol (95 percent) or specially denatured alcohol formula 30 or 3A of the U.S. Bureau of Internal Revenue.

3.3 Hydrochloric acid. A.C.S. reagent grade concentrated hydrochloric acid.

3.4 Ammonium hydroxide. A.C.S. reagent grade ammonium hydroxide.

3.5 Sulfuric acid. A.C.S. reagent grade sulfuric acid.

4. PROCEDURE

Examine the pigment extracted from the coating material by method 4021 as follows.

4.1 Qualitative analysis. Test the pigment by regular qualitative procedures to determine the presence of materials not permitted in the individual product specification.

4.2 Quantitative analysis.

4.2.1 Insoluble siliceous material. Transfer about 1 g of the pigment, weighed to the nearest 0.1 mg, to a 400-ml beaker. Add 20 ml of A.C.S. reagent

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grade hydrochloric acid, heat on steam bath for 30 minutes. Boil for 5 minutes while adding a drop or two of alcohol (see 3.2). Remove cover and evaporate to dryness on the steam bath. Moisten with the hydrochloric acid and dilute with hot water to about 250 ml. Filter on paper while hot, wash thoroughly with hot water until the washings are free from lead and chlorine. Retain the filtrate and washings for use in 4.2.2. Transfer the filter paper and contents to a weighed porcelain crucible and ignite. Cool in a desiccator and weigh. The residue will consist of siliceous material, or a mixture of siliceous matter and barium sulfate when the latter is present (see 4.1).

4.2.1.1 When barium sulfate is present and it is desired to determine its percentage, treat the residue in accordance with the procedure given in method 7281.

4.2.2 Total lead as PbSO₄. To the combined filtrate and washings from 4.2.1 add A.C.S. reagent grade ammonium hydroxide until a faint precipitate begins to form. Then add 5 ml of A.C.S. reagent grade hydrochloric acid and dilute to 500 ml. Pass in a rapid stream of hydrogen sulfide until all the lead is precipitated as PbS. Allow the precipitate to settle, filter, wash with water containing a little hydrogen sulfide. Retain the filtrate and washings for use in 4.2.3. Dissolve the precipitate from the filter with hot dilute nitric acid (see 3.1). Add 10 ml of A.C.S. reagent grade sulfuric acid and evaporate until copious fumes of sulfur trioxide are evolved. Cool, add 75 milliliters of distilled water and 75 ml of the alcohol (see 3.2). Stir, let settle, and filter through a weighed Gooch crucible. Wash with alcohol diluted with an equal volume of distilled water. Retain the filtrate and washings for use in 4.2.3. Dry, ignite gently, and weigh the precipitate as PbSO₄.

4.2.3 Zinc oxide. Combine the filtrates from the lead sulfide and lead sulfate precipitations (see 4.2.2) and boil on an electric hotplate to remove hydrogen sulfide and alcohol. Adjust volume to 150 ml with distilled water and continue as prescribed in method 7331 beginning at "add 25 ml of the solution of tartaric acid".

4.2.4 Lead chromate. Use the procedure and calculation prescribed in method 7131.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 7281
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ANALYSIS OF PIGMENT EXTRACTED FROM CHROME GREEN PAINTS

1. SCOPE

1.1 This method covers procedures for the analysis of chrome green pigment extracted from paints.

2. APPARATUS

- 2.1 Porcelain dish (small).
- 2.2 Beaker. A 250- and a 400-ml beaker.
- 2.3 Steam bath.
- 2.4 Porcelain crucible.
- 2.5 Desiccator.
- 2.6 Agate mortar.
- 2.7 Covered platinum crucible.
- 2.8 Gooch crucible.
- 2.9 Volumetric flask. A 500-ml volumetric flask.

3. REAGENTS

3.1 Hydrochloric acid (1:1). Mix equal parts by volume of A.C.S. reagent grade hydrochloric acid and freshly distilled water.

3.2 Sulfuric acid (1:4). Mix 1 part by volume of A.C.S. reagent grade sulfuric acid with 4 parts by volume of distilled water.

3.3 Sodium hydroxide solution (30 percent). Dissolve 30 g of A.C.S. reagent grade sodium hydroxide in 70 ml of freshly distilled water.

3.4 Ethyl alcohol (95 percent). Or specially denatured alcohol formula 30 or 3A of the United States Bureau of Internal Revenue.

3.5 Chloroform.

3.6 Hydrochloric acid. A.C.S. reagent grade hydrochloric acid.

3.7 Sodium carbonate. A.C.S. reagent grade anhydrous sodium carbonate.

3.8 Nitric acid. A.C.S. reagent grade nitric acid.

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4. PROCEDURE

Examine the pigment extracted from the paint by method 4021 as follows.

4.1 Qualitative analysis. Test for iron ferro-ferric cyanide (Prussian) blue by boiling a portion of the pigment with sodium hydroxide solution (see 3.3). A yellow or yellow-brown precipitate with a yellow liquid above it should result. Filter, add a mixture of ferric and ferrous salts to the filtrate, and render acid with dilute hydrochloric acid (see 3.1). CAUTION. HYDROGEN CYANIDE MAY BE EVOLVED BY THIS PROCEDURE! A blue color indicates Prussian blue in the sample. Ignite another portion very gently to decompose the Prussian blue in the sample, and test the residue for barium and calcium, using appropriate qualitative procedures. Boil a portion of the pigment with water, alcohol, and chloroform. No blue should go into the organic solution.

4.2 Quantitative analysis.

4.2.1 Insoluble siliceous material. Heat about 1 g weighed to the nearest 0.1 mg of the pigment very gently in a small porcelain dish. The heat must be so regulated, by moving the burner, that the Prussian blue is thoroughly decomposed without rendering the iron difficultly soluble. Allow to cool, transfer to a 400-ml beaker, add 20 ml of A.C.S. reagent grade hydrochloric acid, heat on steam bath for 30 minutes, boil for 5 minutes, remove cover and evaporate to dryness on the steam bath. Moisten with A.C.S. reagent grade hydrochloric acid, dilute with hot water to about 250 ml, filter on paper while hot and wash thoroughly with hot water until the washings are free from lead and chlorine. Retain the filtrate and washings for use in 4.2.2. Transfer the filter paper and contents to a weighed porcelain crucible and ignite. Cool in a desiccator, and weigh. The residue shall be considered entirely insoluble siliceous material unless barium sulfate has been found to be present by qualitative analysis (see 4.1). When barium sulfate is present and it is desired to determine its percentage proceed as follows.

4.2.1.1 Mix the ignited residue with about 10 times its weight of A.C.S. reagent grade anhydrous sodium carbonate (grinding the mixture in an agate mortar if necessary), and fuse the mixture in a covered platinum crucible, heating about 1 hour. Let cool, place crucible and cover in a 250-ml beaker, add about 100 ml of water, and heat until the melt is disintegrated. Filter on paper (leaving crucible and cover in the beaker) and wash the beaker and filter thoroughly with hot water to remove soluble sulfates. Place the beaker containing the crucible and cover under the funnel, pierce the filter with a glass rod, and wash the carbonate residue into the beaker by means of a jet of hot water. Wash the paper with hot, dilute hydrochloric acid (see 3.1) and then with hot water. If the carbonate residue is not completely dissolved, add sufficient dilute hydrochloric acid to effect solution. Remove crucible and cover, washing them with a jet of water. Heat the solution to boiling and add 10 to 15 ml of dilute sulfuric acid (see 3.2) and continue the boiling for 10 to

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15 minutes longer. Let the precipitate settle, filter on a weighed Gooch crucible, wash with hot water, ignite, cool, and weigh as BaSO_4 . Subtract from the result of the previous determination to obtain the siliceous material.

4.2.2 Total lead as PbSO_4 . Use the combined filtrate and washings from 4.2.1 and follow the procedure given in method 7271.

4.2.3 Total chromium as PbCrO_4 . Use the procedure and calculations prescribed in method 7111.

4.2.4 Calcium. Ignite 2 g weighed to the nearest 0.1 mg of the pigment and dissolve the residue in hydrochloric acid as in 4.2.1. Then, without filtering the insoluble matter, transfer to a 500-ml volumetric flask, saturate with hydrogen sulfide, make alkaline with ammonia, fill to the mark, mix, and filter through a dry paper, discarding the first 20 ml. Transfer a 250-ml aliquot of the filtrate (corresponding to 1/2 of the weight of sample used) to a 400-ml beaker. Make slightly acid to litmus with dilute hydrochloric acid (see 3.1). Boil to expel the hydrogen sulfide. Add a few drops of A.C.S. reagent grade nitric acid. Make just alkaline with ammonium hydroxide and add 5 ml in excess. Heat to boiling and slowly add, while stirring, 10 to 15 ml of hot saturated ammonium oxalate solution. Continue boiling until the precipitate becomes granular; let stand about 30 minutes, filter, and wash with hot water until free from ammonium oxalate. Pierce the point of the filter paper with hot water. Pour warm 20 percent sulfuric acid through the paper, and wash a few times. Add about 30 ml of the sulfuric acid, dilute to about 250 ml with hot water, and titrate at once with the 0.1N potassium permanganate solution. The solution should not be below 60° C when the end point is reached.

$$\text{Percent CaO} = \frac{A \times N \times 0.02804}{S} \times 100$$

Where: A = ml of standard potassium permanganate solution used for titration.
 N = Normality of the potassium permanganate solution.
 S = g of sample used.

4.3 Computations.

4.3.1 Percentage of total lead (Pb), present in the form of PbCrO_4 .

4.3.1.1 Compute the percentage of lead (Pb) present in the form of lead chromate of the total lead (Pb) in the pigment as follows.

4.3.1.2 Compute the percentage of lead sulfate as determined in 4.2.2 by the factor 0.683 to obtain the percentage of total lead (Pb) in the pigment. Call this value "B".

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4.3.1.3 Multiply the percentage of lead chromate as determined in 4.2.3 by the factor 0.641 to obtain the percentage of lead (Pb) present as lead chromate in the pigment. Call this value "A".

4.3.1.4 Then "C", the percentage of total lead (Pb) present in the pigment in the form of PbCrO_4 , is obtained by the following equation:

$$C = \frac{A}{B} \times 100$$

4.3.2 Pure chrome green. Add the percentages of barium sulfate, when present, calcium oxide, and insoluble siliceous material and subtract the sum from 100 to determine the percentage of pure chrome green in the pigment.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 7291.1
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ANALYSIS OF PIGMENT EXTRACTED FROM IRON OXIDE PAINTS

1. SCOPE

1.1 This method covers procedures for the analysis of pigment extracted from iron oxide paints.

2. APPARATUS

- 2.1 Beaker. A 250-ml and a 600-ml beaker.
- 2.2 Steam bath.
- 2.3 Filter paper. Filter paper, slow speed.
- 2.4 Porcelain dish.
- 2.5 Porcelain crucible.
- 2.6 Desiccator.

3. REAGENTS

- 3.1 Acetic acid solution (4 percent). Mix 40 ml of A.C.S. reagent grade glacial acetic acid with freshly distilled water and dilute to 1 liter.
- 3.2 Hydrochloric acid (1 to 1). Mix equal parts by volume of A.C.S. reagent grade hydrochloric acid and freshly distilled water.
- 3.3 Methyl orange indicator.
- 3.4 Ammonium hydroxide. A.C.S. reagent grade ammonium hydroxide.
- 3.5 Hydrochloric acid. A.C.S. reagent grade hydrochloric acid.
- 3.6 Standard potassium ferrocyanide solution. Dissolve 22 g of potassium ferrocyanide in water and dilute to 1 liter. (Sodium sulfite may be added to stabilize the solution in the proportion of 7 g per liter of solution). To standardize, transfer about 0.2 g, weighed to the nearest 0.1 mg, of metallic zinc to a 400-ml beaker. Dissolve in 30 ml of 33 percent hydrochloric acid. Drop in a small piece of litmus paper and add ammonium hydroxide until slightly alkaline. Add hydrochloric acid until just acid, and then add 3 ml in excess. Dilute to about 250 ml with hot water and heat nearly to boiling. Add the ferrocyanide solution slowly from a burette with constant stirring until a drop rested on a white porcelain plate with a drop of the uranyl indicator shows a brown tinge after standing about 30 seconds. Run a blank with the same amounts of reagents and water. Calculate the weight of zinc oxide in g equivalent to 1 ml of the solution as follows:

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$$F = \frac{S \times 1.2447}{A - B}$$

Where: F = zinc oxide equivalent of the standard potassium ferrocyanide solution in g per ml.
 S = g of metallic zinc.
 A = ml of potassium ferrocyanide solution required for titration of the zinc.
 B = ml of potassium ferrocyanide solution required for titration of the blank.

3.7 Distilled water.

4. PROCEDURE

Examine the pigment extracted from the paint by method 4021 as follows.

4.1 Organic coloring matter. Use the applicable qualitative procedures given in method 5021.

4.2 Total iron oxide. Use the procedure and calculation given in ASTM D 50.

4.3 Total zinc oxide. Weigh to the nearest 0.1 mg about 1 g of the pigment and transfer to a 250-ml beaker. Wet with few drops of acetone and add 50 ml of acetic acid solution (see 3.1). Warm on a steam bath for 10 minutes with frequent stirring. Remove, allow to settle, and filter through a high retention filter paper. Catch filtrate in a 600-ml beaker. Wash residue 3 times with the acetic acid solution and twice with distilled water. Add 3 drops of methyl orange indicator to the filtrate and make slightly alkaline with A.C.S. reagent grade ammonium hydroxide. Neutralize with A.C.S. reagent grade hydrochloric acid and add 3 ml of the acid in excess. Adjust with hot water to a volume of 250 ml and heat nearly to boiling. Titrate the not solution with standard potassium ferrocyanide solution. Run a blank using the same amounts of reagents and water.

4.3.1 Calculate the total zinc and percentage of zinc oxide in the pigment using the following equation;

$$\text{Percent ZnO} = \frac{(A - B) \times F \times 100}{S}$$

Where: A = ml of standard potassium ferrocyanide solution used for titration of the sample.
 B = ml of standard potassium ferrocyanide solution used for titration of the blank.
 F = zinc oxide equivalent of the standard potassium ferrocyanide solution in g per ml (see 3.6)
 S = c of sample used.

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4.4 Total calcium. Use the procedure and calculation given in ASTM D 50.

4.5 Insoluble siliceous matter. Transfer about 1 g weighed to the nearest 0.1 mg of the pigment to a porcelain dish and ignite at a dull red heat to destroy organic matter. Cool, add 20 ml of hydrochloric acid (see 3.2); cover and heat on a steam bath until no dark specks can be seen in the insoluble residue. Remove cover, add 10 ml of A.C.S. reagent grade hydrochloric acid, and evaporate to dryness on a steam bath. Moisten the residue with the hydrochloric acid and dilute with about 100 ml of distilled water. Bring the solution to boiling and allow the insoluble matter to settle out on a steam bath. Transfer the insoluble matter to a filter paper and wash well with hot water. Place the filter paper and its contents in a weighed porcelain crucible and ignite. Cool in a desiccator, weigh and calculate the percentage of insoluble siliceous matter.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 7301
January 24, 1986

ANALYSIS OF PIGMENT EXTRACTED FROM WOOD-SASH PUTTY

1. SCOPE

1.1 This method covers procedures for the analysis of pigment extracted from wood-sash putty.

2. APPARATUS

2.1 Beakers, 200- and 250-ml beakers.

2.2 Steam bath.

2.3 Gooch crucible.

3. REAGENTS

3.1 Hydrochloric acid (0.1 N). Add about 9 ml of A.C.S. reagent grade hydrochloric acid to about 900 ml of distilled water, cool and dilute to 1 liter. Standardize against freshly standardized sodium hydroxide solution or by any other accurate method, using phenolphthalein indicator solution.

3.2 Alcohol. Ethyl alcohol (95 percent) or specially denatured alcohol formula 30 or 3A of the United States Bureau of Internal Revenue.

3.3 Hydrochloric acid (1:1). Mix equal parts by volume of A.C.S. reagent grade hydrochloric acid and freshly distilled water.

3.4 Bromine water. Prepared by saturating freshly distilled water with A.C.S. reagent grade bromine.

3.5 Ammonium oxalate solution. A saturated solution of A.C.S. reagent grade ammonium oxalate in freshly distilled water.

3.6 Standard potassium permanganate solution (0.1 N). Dissolve 3.161 g of A.C.S. reagent grade potassium permanganate in a liter of freshly distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: Transfer 0.3 g, weighed to the nearest 0.1 mg of sodium oxalate, National Bureau of Standards standard sample 40F (dried for 1 hour at 105° C), to a 600-ml beaker. Add 250 ml of dilute sulfuric acid (see 3.10), previously boiled for 10 to 15 minutes and then cooled to 27° C +3° C. Stir until the oxalate has dissolved. Add 39 to 40 ml of the potassium permanganate (0.3 g of the sodium oxalate required 44.75 ml of 0.1 N potassium permanganate solution) at a rate of 25 to 35 ml per minute, while stirring slowly. Let stand until the pink color disappears (about 45 seconds) (see 5.1). Heat to 55°-60° C and complete the titration by adding the permanganate solution until a faint pink color persists for 30 seconds. Add the last 0.5 to 1 ml dropwise, with particular care to allow each drop to become decolorized before the next is introduced.

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3.7 Hydrochloric acid (1 to 10). Mix 1 part by volume of A.C.S. reagent grade hydrochloric acid with 10 parts by volume of freshly distilled water.

3.8 Nitric acid (1 to 3). Mix 1 part by volume of A.C.S. reagent grade nitric acid with 3 parts by volume of freshly distilled water.

3.9 Sulfuric acid (1 to 1). Mix equal volumes of A.C.S. reagent grade sulfuric acid and freshly distilled water.

3.10 Sulfuric acid (1 to 19). Mix 5 parts by volume of A.C.S. reagent grade sulfuric acid with 95 parts by volume of freshly distilled water.

4. PROCEDURE

Examine the pigment extracted from the putty by method 4021 as follows.

4.1 Qualitative analysis. Test the pigment by regular qualitative procedures to determine the presence of materials not permitted in the product specification.

4.2 Quantitative analysis.

4.2.1 Reaction and free alkali. Weigh 2 ± 0.01 g of the pigment into a 200-ml beaker, and add 100 ml of distilled water. Boil for 5 minutes and filter through paper. Add a drop of methyl orange indicator to a 5 ml aliquot of the filtrate to determine its neutrality. To the remainder of the filtrate add two drops of phenolphthalein indicator and titrate with the standard hydrochloric acid solution (see 3.1).

4.2.2 Calcium carbonate (in the absence of white lead). Determine the calcium carbonate content of type I putty as follows.

4.2.2.1 Weigh to the nearest 0.1 mg 0.25 g of the dry pigment and transfer to 250-ml beaker. Moisten with alcohol (see 3.2) and dissolve in about 20 ml of hydrochloric acid (see 3.3), keeping the beaker covered. Digest for 10 minutes on the steam bath and dilute to about 150 ml. Filter the solution and wash the insoluble residue with hot water. Add a few ml of the bromine water (see 3.4) to the combined filtrate and washings and heat to boiling to expel the excess bromine. Allow to cool somewhat and make alkaline with A.C.S. reagent grade ammonium hydroxide. Heat for a short time with stirring to coagulate any precipitate, of iron, aluminum, etc., that may have formed. Allow the precipitate to settle, filter through paper and wash thoroughly with hot water. Reduce the volume of the filtrate and washings to about 200 ml. Add a few ml of the ammonium hydroxide and continue as prescribed in the following.

4.2.2.1.1 Heat to boiling and slowly add, while stirring, 10 to 15 ml of hot saturated ammonium oxalate solution (see 3.5). Continue boiling until the

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precipitate becomes granular; let stand about 30 minutes, filter, wash with hot water until free from ammonium oxalate. Pierce the apex of the filter paper with a stirring rod and wash the precipitate into the beaker with hot water. Pour warm dilute sulfuric acid (see 3.10) through the paper and wash a few times. Add about 30 milliliters of the sulfuric acid, dilute to about 250 ml with hot water, and titrate at once with the standard potassium permanganate solution (see 3.6). The solution should not be below 60° C when the end point is reached.

4.2.2.2 Calculation. Calculate the percentage calcium carbonate in the pigment as follows.

$$\text{Percent CaCO}_3 = \frac{A \times N \times 0.050}{S} \times 100$$

where:

- A = Milliliters of standard potassium permanganate solution required for titration of the sample.
- N = Normality of the potassium permanganate solution.
- S = Grams of sample used.

4.2.3 White lead and calcium carbonate. Determine the white lead and calcium carbonate of type II putty as follows.

4.2.3.1 White lead. Weigh to the nearest 0.1 mg 1 g of the dry pigment into a 250-ml beaker, moisten with a few drops of the alcohol (see 3.2), add slowly 25 to 30 ml of A.C.S. reagent grade hydrochloric acid, cover, and boil for 5 to 10 minutes. Dilute to about 150 ml with hot water and heat for about 15 minutes. Let settle on the steam bath, and filter while hot, washing any insoluble residue thoroughly with hot water. (Avoid allowing the filter paper to become cold.) Make the solution just alkaline with A.C.S. reagent grade ammonium hydroxide, then just faintly acid to litmus, using dilute hydrochloric acid (see 3.7). The final volume should contain about 1 percent by volume hydrochloric acid. Dilute to about 300 ml and pass hydrogen sulfide gas into the clear solution to complete precipitation. (Sometimes it is necessary to start the precipitation by adding a drop or two of ammonium hydroxide.) Let settle, filter on paper, and wash with water containing some hydrogen sulfide. Retain the filtrate and washings for use in 4.2.3.2. Dissolve the sulfide from the filter paper with hot dilute nitric acid (see 3.8). Add 5 to 10 ml of dilute sulfuric acid (see 3.9), and evaporate until copious fumes of sulfur trioxide are evolved. Cool, add about 75 ml of alcohol (see 3.2). Stir, let settle, filter on a weighed Gooch crucible. Wash with the alcohol diluted with an equal volume of distilled water. Dry, gently ignite, and weigh as lead sulfate. Multiply this weight by the factor 0.854 and report the result as basic carbonate white lead.

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4.2.3.2 Calcium carbonate. Boil the combined filtrate and washings from the sulfide separation (see 4.2.3.1) to expel the hydrogen sulfide. Add a few drops of A.C.S. reagent grade nitric acid and heat for a few minutes longer. Allow to cool somewhat and make alkaline with A.C.S. reagent grade ammonium hydroxide. Heat for a short time with stirring to coagulate any precipitate, of iron, aluminum, etc., that may have formed. Allow the precipitate to settle, filter through paper and wash thoroughly with hot water. Reduce the volume of the filtrate and washings to about 200 ml. Add a few ml of the ammonium hydroxide and continue as prescribed in the following.

4.2.3.2.1 Heat to boiling and slowly add, while stirring, 10 to 15 ml of hot saturated ammonium oxalate solution (see 3.5). Continue boiling until the precipitate becomes granular; let stand about 30 minutes, filter, wash with hot water until free from ammonium oxalate. Pierce the apex of the filter paper with a stirring rod and wash the precipitate into the beaker with hot water. Pour warm dilute sulfuric acid (see 3.10) through the paper and wash a few times. Add about 30 milliliters of the sulfuric acid, dilute to about 250 ml with hot water, and titrate at once with the standard potassium permanganate solution (see 3.6). The solution should not be below 60° C when the end point is reached. Calculate the percent calcium carbonate as prescribed in 4.2.2.2.

5. NOTES

5.1 If the pink color should persist because the permanganate is in excess, discard and begin again, adding a few ml less of the permanganate solution.

6. PRECISION

6.1 No data.

METHOD 7311
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ANALYSIS OF PIGMENT EXTRACTED FROM BLACK PAINTS

1. SCOPE

1.1 This method covers procedures for the analysis of pigment extracted from black paints.

2. APPARATUS

2.1 Beaker, 400-ml beaker.

2.2 Porcelain dish.

2.3 Steam bath.

2.4 Gooch crucible.

2.5 Muffle furnace.

3. REAGENTS

3.1 Alcohol. Ethyl alcohol (95 percent) or specially denatured alcohol formula 30 or 3A of the United States Bureau of Internal Revenue.

3.2 Nitric acid (1 to 3). Mix 1 part by volume of A.C.S. reagent grade nitric acid with 3 parts by volume of freshly distilled water.

3.3 Bromine water. Prepared by saturating freshly distilled water with A.C.S. reagent grade bromine.

3.4 Sulfuric acid. A.C.S. reagent grade.

3.5 Ammonium hydroxide. A.C.S. reagent grade.

3.6 Hydrochloric acid. A.C.S. reagent grade.

3.7 Hydrogen peroxide. A.C.S. reagent grade.

3.8 Stannous chloride solution. Dissolve 50 g of A.C.S. reagent grade stannous chloride in 300 ml of A.C.S. reagent grade hydrochloric acid and dilute with freshly distilled water to 500 ml. Keep the clear solution in a tightly stoppered bottle containing some metallic tin.

3.9 Potassium permanganate solution (0.1 N). Dissolve 3.161 g of A.C.S. reagent grade potassium permanganate in a liter of freshly distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: Transfer 0.3 gram weighed to the nearest 0.1 mg of sodium oxalate, National Bureau of Standards standard sample 40 F,

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(dried for 1 hour at 105° C) to 600-ml beaker. Add 250 ml of dilute sulfuric acid (see 3.10), previously boiled for 10 to 15 minutes and then cooled to 27° C \pm 3° C. Stir until the oxalate has dissolved. Add 39 to 40 ml of the potassium permanganate (0.3 g of the sodium oxalate requires 44.75 ml of the potassium permanganate solution) at a rate of 25 to 35 ml per minute, while stirring slowly. Let stand until the pink color disappears (about 45 seconds) (see 5.1). Heat to 55° C to 60° C and complete the titration by adding the permanganate solution until a faint pink color persists for 30 seconds. Add the last 0.5 to 1 ml dropwise, with particular care to allow each drop to become decolorized before the next is introduced.

3.10 Sulfuric acid (1 to 19). Mix 5 parts by volume of A.C.S. reagent grade sulfuric acid with 95 parts by volume of freshly distilled water.

3.11 Mercuric chloride solution. A saturated solution of A.C.S. reagent grade mercuric chloride (60 to 100 g per liter).

3.12 Sulfuric-phosphoric acid mixture. Mix 150 ml of A.C.S. reagent grade sulfuric acid with 150 ml of A.C.S. reagent grade phosphoric acid and dilute the mixture with freshly distilled water to 1 liter.

3.13 Sodium diphenylamine sulfonate indicator solution. Dissolve 0.2 g of p-diphenylamine sulfonic acid sodium salt, (C₂H₅NHC₆-H₄-SO₃Na) in 100 ml of freshly distilled water.

3.14 Standard potassium dichromate solution (0.1 N). Dissolve 4.904 g of A.C.S. reagent grade potassium dichromate in freshly distilled water and dilute to 1 liter. Standardize against NBS standard sample No. 27.

3.15 Other reagents. As prescribed in the individual methods referenced herein.

4. PROCEDURE

Separate the pigment from the vehicle of a weighed amount of the paint by method 4021 and examine the pigment and vehicle individually as follows.

4.1 Carbon and insoluble mineral material. Weigh to the nearest 0.1 mg about 1 g of the pigment and transfer to a porcelain dish. Moisten with a few drops of the alcohol (see 3.1), and add 20 ml of A.C.S. reagent grade hydrochloric acid. Cover, and heat on steam bath for 15 minutes. Remove cover and evaporate to dryness. Moisten the residue with a few drops of the hydrochloric acid, and add 25 ml of water. Bring this solution to a boil and let the insoluble matter settle out on a steam bath. Filter on a weighed Gooch crucible, and wash with hot water until the washings are free from lead and iron. Retain the filtrate and washings for use in 4.2.1. Dry the crucible and contents at 105° C \pm 2° C (221° F \pm 3.6° F) for 2 hours. Ignite for 7 minutes in a current of dry carbon dioxide (using a Rose crucible cover) with a flame about 20 cm high. Cool in a

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current of dry carbon dioxide and weigh. The increase of weight represents the weight of combined carbon and insoluble mineral matter in the sample. Then ignite with free access of air or in a current of oxygen until all carbon is consumed. Cool the crucible in a desiccator and weigh. Calculate and report the loss in weight as percentage of carbon and the weight of the residue in the crucible as percentage of insoluble mineral matter in the pigment.

4.2 Lead oxide as Pb_3O_4 .

4.2.1 Lead oxide in pigment. Dilute the combined filtrate and washings from 4.1 to about 250 ml with distilled water. Neutralize with A.C.S. reagent grade ammonium hydroxide and then add between 2 and 3 ml of A.C.S. reagent grade hydrochloric acid in excess. Pass in a rapid stream of hydrogen sulfide until all the lead is precipitated. Allow the precipitate to settle, filter through a paper and wash the precipitate with water containing a little hydrogen sulfide. Retain the filtrate and washings for use in 4.3. Dissolve the precipitate from the filter with hot dilute nitric acid (see 3.2). Add 10 ml of A.C.S. reagent grade sulfuric acid and evaporate until copious fumes of sulfur trioxide are evolved. Cool, add 75 milliliters of distilled water and 75 ml of the alcohol (see 3.1). Stir, let settle and filter through a weighed Gooch crucible. Wash with alcohol diluted with an equal volume of distilled water. Dry, ignite gently, and weigh the precipitate as $PbSO_4$.

4.2.2 Lead oxide in vehicle. Evaporate the organic solvents from the vehicle obtained in 4 in a porcelain crucible on a steam bath. Heat the crucible and its contents in an oven at $105^\circ \pm 2^\circ \text{C}$ ($221^\circ \text{F} \pm 3.6^\circ \text{F}$) for several hours to remove all traces of water. Place the crucible on the floor of a hood and burn off most of the organic matter using a low flame. When the contents of the crucible have reached a charred state place the crucible in a muffle furnace and ignite at a dull red heat, not over 600°C . Dissolve the ash in dilute nitric acid (see 3.2) using a few drops of A.C.S. reagent grade hydrogen peroxide and warming on a steam bath if necessary. Carefully transfer the acid solution to a 400-ml beaker filtering through paper, if necessary to remove any carbonaceous matter. Continue by adding 10 ml of A.C.S. reagent grade sulfuric acid and evaporate until copious fumes of sulfur trioxide are evolved. Cool, add 75 ml of distilled water and 75 ml of the alcohol (see 3.1). Stir, let settle and filter through a weighed Gooch crucible. Wash with alcohol diluted with an equal volume of distilled water. Dry, ignite gently, and weigh the precipitate as $PbSO_4$.

4.2.3 Calculation. Calculate the percentage of lead oxide expressed as Pb_3O_4 on the basis of the pigment content of the paint as follows.

4.2.3.1 Convert the weights of the $PbSO_4$ obtained in 4.2.1 and 4.2.2 respectively, to grams of Pb_3O_4 by multiplying by the factor 0.7536.

4.2.3.2 From the weight of Pb_3O_4 obtained in pigment (see 4.2.3.1) and the weight of the pigment used, determine the percentage of Pb_3O_4 in the extracted pigment.

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4.2.3.3 Calculate the amount of Pb_3O_4 found in the vehicle (see 4.2.3.1) to a percentage of pigment basis using the following equation:

$$\text{Percent } Pb_3O_4 = \frac{A}{B \times D} \times 100$$

where:

- A = Grams of Pb_3O_4 obtained in vehicle (see 4.2.3.1).
 B = Grams of paint used in extraction of pigment (see 4).
 D = Percentage of pigment in paint expressed as a decimal fraction.

4.2.3.4 Report the sum of the percentage of Pb_3O_4 found in 4.2.3.2 and 4.2.3.3 as the percentage of lead oxide in the pigment.

4.3 Iron oxide as Fe_2O_3 .

4.3.1 Iron oxide in pigment. Heat the combined filtrate and washings from 4.2.1 to expel the hydrogen sulfide. Add a few milliliters of bromine water (see 3.3) and continue heating to expel the excess bromine. Drop in the stannous chloride solution slowly until the last drop makes the solution colorless or free from any tinge of yellow. Add 1 or 2 drops in excess of stannous chloride solution. Keep the watchglass on the beaker while adding the stannous chloride solution, and agitate the hot iron solution after each addition. If too much stannous chloride is added by mistake, add the potassium permanganate solution (see 3.9) to the solution until a yellow color appears, then again add the stannous chloride solution drop by drop until the yellow color just disappears, again adding one or two drops in excess. Dilute with 200 ml of cold water, then add all at once with vigorous stirring 15 ml of the mercuric chloride solution (see 3.11). Let stand 3 or 4 minutes. A slight white precipitate should form. If none, or a heavy grayish precipitate forms, the determination should be discarded and repeated. Add 15 ml of the sulfuric-phosphoric acid mixture (see 3.12) and 6 to 8 drops of the sodium diphenylamine indicator solution (see 3.13). Titrate with the standard potassium dichromate solution (see 3.14), taking the sudden change of the green color to a purple color as the end point.

4.3.2 Calculate the total iron as percentage of iron oxide in the pigment using the following equation:

$$\text{Percent } Fe_2O_3 = \frac{A \times N \times 0.07985}{S} \times 100$$

where:

- A = Milliliters of standard potassium dichromate solution required for the titration.
 N = Normality of potassium dichromate solution.
 S = Grams of sample used.

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5. NOTES

5.1 If the pink color should persist because the permanganate is in excess, discard and begin again, adding a few milliliters less of the permanganate solution.

6. PRECISION

6.1 No. data.

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METHOD 7331
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ANALYSIS OF PIGMENT EXTRACTED FROM IRON OXIDE-ZINC CHROMATE

1. SCOPE

1.1 This method covers procedures for the analysis of pigment extracted from iron oxide-zinc chromate paints and primers.

2. APPARATUS

2.1 Beakers, 400- and 600-ml beakers.

2.2 Porcelain crucible.

3. REAGENTS

3.1 Stannous chloride solution (10 percent). Dissolve 50 g of A.C.S. reagent grade stannous chloride in 300 ml of A.C.S. reagent grade hydrochloric acid and dilute with freshly distilled water to 500 ml. Keep the clear solution in a tightly stoppered bottle containing some metallic tin.

3.2 Sodium hydroxide solution (30 percent). Dissolve 30 g of A.C.S. reagent grade sodium hydroxide in 70 ml of freshly distilled water.

3.3 Hydrochloric acid (1 to 3). Mix 1 part by volume of A.C.S. reagent grade hydrochloric acid with 3 parts by volume of freshly distilled water.

3.4 Sulfuric acid (1 to 4). Mix 1 part by volume of A.C.S. reagent grade sulfuric acid with 4 parts by volume of distilled water.

3.5 Formic acid mixture. Add 60 ml of A.C.S. reagent grade ammonium hydroxide to 400 ml of A.C.S. reagent grade formic acid. In another container, dissolve 500 g of A.C.S. reagent grade ammonium sulfate in approximately 1 liter of freshly distilled water. Mix the two solutions and add sufficient water to make 2 liters.

3.6 Hydrochloric acid (1 to 1). Mix equal parts by volume of A.C.S. reagent grade hydrochloric acid and freshly distilled water.

3.7 Tartaric acid (25 percent). Dissolve 250 g of A.C.S. reagent grade tartaric acid in freshly distilled water and dilute to 1 liter.

3.8 Standard potassium permanganate solution (0.1 N). Dissolve 3.161 g of A.C.S. reagent grade potassium permanganate in a liter of freshly distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: Transfer 0.3 g weighed to the nearest 0.1 mg of sodium oxalate, National Bureau of Standards standard sample 40F (dried for 1 hour at 105° C) to a 600-ml beaker. Add 250 ml of dilute

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sulfuric acid (see 3.11) previously boiled for 10 to 15 minutes and then cooled to 27° +3° C. Stir until the oxalate has dissolved. Add 39 to 40 ml of the potassium permanganate (0.3 g of the sodium oxalate requires 44.75 ml of the potassium permanganate solution-0.1 N) at a rate of 25 to 35 ml per minute, while stirring slowly. Let stand until the pink color disappears (about 45 seconds) (see 5.1). Heat to 55°-60° C and complete the titration by adding the permanganate solution until a faint pink color persists for 30 seconds. Add the last 0.5 to 1 ml dropwise, with particular care to allow each drop to become decolorized before the next is introduced.

3.8.1 Determine the excess of permanganate required to impart a pink color to the solution, by matching the color by adding the permanganate solution to the same volume of the sulfuric acid (see 3.8) at 55° C to 60° C. (This correction usually amounts to from 0.03 to 0.05 ml).

3.8.2 Calculate the normality of the permanganate solution using the following equation:

$$N = \frac{S}{0.06701 (A - B)}$$

where:

- N = Normality of potassium permanganate solution.
- S = Grams of sodium oxalate used.
- A = Milliliters of potassium permanganate solution required for the sodium oxalate solution.
- B = Milliliters of potassium permanganate solution required for the sulfuric acid and blank.

3.8.3 The standard potassium permanganate solution shall be stored in a glass-stoppered bottle painted black to keep out light.

3.9 Silver nitrate solution (2.5 percent). Dissolve 2.5 g of A.C.S. reagent grade silver nitrate in freshly distilled water and make up to 100 ml volume.

3.10 Ammonium persulfate solution. A freshly prepared solution of A.C.S. reagent grade ammonium persulfate in freshly distilled water in the proportion of 4 g per 20 ml of solution.

3.11 Sulfuric acid (1 to 19). Mix 5 parts by volume of A.C.S. reagent grade sulfuric acid with 95 parts by volume of freshly distilled water.

3.12 Hydrochloric acid (1 to 2). Mix 1 part by volume of A.C.S. reagent grade hydrochloric acid with 2 parts by volume of freshly distilled water.

3.13 Ammonium hydroxide. A.C.S. reagent grade.

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3.14 Standard potassium ferrocyanide solution. Dissolve 22 g of A.C.S. reagent grade potassium ferrocyanide in freshly distilled water and dilute to 1,000 ml. (A.C.S. reagent grade sodium sulfite may be added to stabilize the solution in the proportion of 7 g per liter of solution.) To standardize, transfer about 0.2 g weighed to the nearest 0.1 mg of A.C.S. reagent grade metallic zinc to a 400-ml beaker. Dissolve in 30 ml of hydrochloric acid (see 3.12). Drop in a small piece of litmus paper, add the ammonium hydroxide (see 3.13) until slightly alkaline. Add the hydrochloric acid until just acid, and then add 3 ml in excess. Dilute to about 250 ml with hot water and heat nearly to boiling. Run in the ferrocyanide solution slowly from a burette with constant stirring until a drop tested on a white porcelain plate with a drop of the uranyl indicator (see 3.15) shows a brown tinge after standing about 1/2 minute. Run a blank with the same amounts of reagents and water. Calculate and record for use in 4.3.2.1 the grams of zinc oxide equivalent to 1 ml of the solution as follows:

$$F = \frac{S \times 1.2447}{A - B}$$

where:

- F = Zinc oxide equivalent of the standard potassium ferrocyanide solution in grams per milliliter.
- S = Grams of metallic zinc taken.
- A = Milliliters of potassium ferrocyanide solution required for titration of zinc.
- B = Milliliters of potassium ferrocyanide solution required, for titration of blank.

3.15 Uranyl indicator solution. A 5-percent solution of A.C.S. reagent grade uranyl nitrate in freshly distilled water or a 5-percent solution of A.C.S. reagent grade uranyl acetate in water made slightly acid with A.C.S. reagent grade glacial acetic acid.

3.16 Mercuric chloride solution. A saturated solution of A.C.S. reagent grade mercuric chloride (60 to 100 g per liter).

3.17 Sulfuric-phosphoric acid mixture. Mix 150 ml of A.C.S. reagent grade sulfuric acid with 150 ml of A.C.S. reagent grade phosphoric acid and dilute the mixture with freshly distilled water to 1 liter.

3.18 Sodium diphenylamine sulfonate indicator solution. Dissolve 0.2 g of P-diphenylamine sulfonic acid sodium salt in 100 ml of freshly distilled water.

3.19 Standard potassium dichromate solution (0.1 N). As prescribed in ASTM D 521.

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3.20 Standard ferrous ammonium sulfate solution (0.1 N). Dissolve 42 g of A.C.S. reagent grade ferrous ammonium sulfate in a cool mixture of 50 ml of A.C.S. reagent grade sulfuric acid and 950 ml of freshly distilled water. To obtain the ratio of the ferrous ammonium sulfate solution to the potassium permanganate solution (see 3.8) take 25 ml of the former, dilute to 350 ml with cool diluted sulfuric acid (see 3.11), add 2 ml of A.C.S. reagent grade phosphoric acid, titrate with standard potassium permanganate solution to a faint permanent pink tint. Determine a blank on the same volume of water and acids, deduct and calculate the volume of potassium permanganate solution which is equivalent to 1 ml of the ferrous ammonium sulfate solution. The ratio of the ferrous ammonium sulfate solution to potassium permanganate solution must be determined each day the solution is used for titrating a test specimen, unless the ferrous ammonium sulfate solution is kept in an inert atmosphere, i.e., nitrogen or other inert gas.

3.21 Other reagents. As prescribed in the individual methods referenced herein.

4. PROCEDURE

Examine the pigment extracted from the coating material by method 4021 as follows.

4.1 Total iron oxide. Weigh to the nearest 0.1 mg about 0.5 g of the pigment into a porcelain crucible and ignite gently to destroy organic matter. Transfer to a 400-ml beaker and add 25 ml of A.C.S. reagent grade hydrochloric acid. Cover with a watchglass and digest just short of boiling (80° C to 90° C) till no dark specks can be seen in the insoluble residue. If necessary, add a few drops of the stannous chloride solution (see 3.1) to assist in dissolving the iron. When the residue is light in color, the solution of iron may be considered complete. Dilute with water to about 100 ml, neutralize with the sodium hydroxide adding an excess and boil. Decant through a filter paper and wash the precipitate and residue with water. Redissolve the precipitate with 10 ml of dilute hydrochloric acid (see 3.3). Dilute and reprecipitate the iron with the sodium hydroxide solution. Heat to boiling, then filter and wash the precipitate. Dissolve the precipitate from the filter paper into the same beaker with the dilute hydrochloric acid. Then add 25 to 50 ml of water and heat to gentle boiling (avoid vigorous prolonged boiling). Drop in the stannous chloride solution slowly until the last drop makes the solution colorless or free from any tinge of yellow. Add 1 or 2 drops in excess of stannous chloride solution. Keep the watchglass on the beaker while adding the stannous chloride solution, and agitate the hot iron solution after each addition. If too much stannous chloride is added by mistake, add the potassium permanganate solution (see 3.8) to the solution until a yellow color appears, then again add the stannous chloride solution drop by drop until the yellow color just disappears, again adding 1 or 2 drops in excess. Dilute with 200 ml of cold water, then add all at once with vigorous stirring 15 ml of the mercuric chloride solution (see 3.16). Let stand 3 or 4 minutes. A slight white precipitate should form. If

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none, or a heavy grayish precipitate forms, the determination should be discarded and repeated. Add 15 ml of the sulfuric-phosphoric acid mixture (see 3.17) and 6 to 8 drops of the sodium diphenylamine indicator solution (see 3.18). Titrate with the standard potassium dichromate solution (see 3.19), taking the sudden change of the green color to a purple color as the end point. Calculate the total iron as percentage of iron oxide in the pigment using the following equation:

$$\text{Percent Fe}_2\text{O}_3 = \frac{A \times N \times 0.07985}{S} \times 100$$

where:

A = Milliliters of standard potassium dichromate solution required for the titration.

N = Normality of potassium dichromate solution.

S = Grams of sample used.

4.2 Total chromium and zinc yellow.

4.2.1 Weigh to the nearest 0.1 mg about 1 g of the pigment and transfer it to a 600-ml beaker. Add 50 ml of dilute sulfuric acid (see 3.4) and heat until the zinc yellow is dissolved. (The iron oxide may not entirely dissolve. If it does not, filter the solution into another 600-ml beaker.) Dilute to about 300 ml with distilled water, stir and heat to boiling. To the boiling solution add a small amount of the potassium permanganate solution (see 3.8) to insure the presence of some manganese. Then add to the boiling solution 10 ml of the silver nitrate solution (see 3.9) and 20 ml (adding this slowly) of the ammonium persulfate solution (see 3.10). The hot solution should show the usual permanganate color. If this color does not develop, or if it disappears, add more persulfate. When the permanganate color is permanent, boil (best done on a hotplate) for 10 to 15 minutes to destroy excess persulfate. Then add 5 ml of dilute hydrochloric acid (see 3.3) and boil for 5 to 8 minutes to reduce oxidized manganese. The color now should be the usual chromate yellow (see 5.2). Cool to about 20° C, dilute to 400 ml, and add exactly 50 ml of the standard ferrous ammonium sulfate solution (see 3.20) from a burette. Titrate the excess of ferrous salt with the standard potassium permanganate solution (see 3.8). The first faint permanent (1 minute of stirring) darkening of the green color is taken as the end point. (The end point is quite sharp and well defined, but requires some practice. Addition of excess permanganate increases the depth of the color which finally becomes purple).

Determine the volume of the potassium permanganate solution that is equivalent to 50 ml of the ferrous ammonium sulfate solution according to the procedure for standardization given in 3.20.

4.2.2 Calculation. Calculate the percentage of total chromium as CrO₃ and the percentage of zinc yellow as follows:

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$$\text{Percent CrO}_3 = \frac{(A - B) \times N \times 0.03334}{S} \times 100$$

where:

- A = Milliliters of standard potassium permanganate solution equivalent to the 50 milliliters of the ferrous ammonium sulfate solution added.
- B = Milliliters of standard potassium permanganate solution required to titrate the excess of ferrous salt in sample.
- N = Normality of the potassium permanganate solution.
- S = Grams of sample used.

4.2.2.1 Multiply the percentage of CrO₃ by the factor 2.4 to determine the percentage of zinc yellow in the pigment.

4.3 Zinc oxide.

4.3.1 Weigh to the nearest 0.1 mg about 1 g of the pigment and transfer to a 250-ml beaker. Add 25 ml of the dilute sulfuric acid (see 3.4) and let stand at room temperature with occasional stirring for several hours. Filter into a 400-ml beaker, wash paper and insoluble material with the dilute sulfuric acid until free of chromium. Dilute to 150 ml with distilled water and add 25 ml of the solution of tartaric acid (see 3.7). Make just neutral to litmus paper with A.C.S. reagent grade ammonium hydroxide and add 25 ml of the formic acid mixture (see 3.5). Care must be observed to adjust the acidity to the proper pH (between 2 and 3) for quantitative precipitation of zinc as sulfide. Pass a rapid stream of hydrogen sulfide through the solution for 30 minutes. Allow the precipitate to settle for 30 minutes, filter, and wash with distilled water saturated with hydrogen sulfide. Place the paper and precipitate in a beaker and dissolve the precipitate with hot dilute hydrochloric acid (see 3.6). Boil to remove hydrogen sulfide, filter, wash, and cool. Neutralize with ammonium hydroxide, then add 3 ml of A.C.S. reagent grade hydrochloric acid in excess. Dilute to approximately 250 milliliters with hot water, heat nearly to boiling. Titrate the solution while hot with standard potassium ferrocyanide solution in 3.14.

4.3.2 Calculations.

4.3.2.1 Calculate the percentage of total zinc expressed as ZnO as follows:

$$\text{Percent ZnO} = \frac{(A - B) \times F}{S} \times 100$$

where:

- A = Milliliters of standard potassium ferrocyanide solution used for titration of sample.
- B = Milliliters of standard potassium ferrocyanide solution used for titration of blank.

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F = Zinc oxide equivalent of the standard potassium ferrocyanide solution in grams per milliliter (see 3.14).
S = Grams of sample used.

4.3.2.2 Convert the percentage of zinc yellow obtained in 4.2.2.1 to percentage of ZnO by multiplying by the factor 0.37.

4.3.2.3 Subtract the percentage of ZnO obtained in 4.3.2.2 from that obtained in 4.3.2.1, and report the remainder as percentage of zinc oxide in the pigment.

4.4 Insoluble siliceous material. Weigh to the nearest 0.1 mg about 0.5 g of the pigment and transfer to a 400-ml beaker. Add 15 ml of A.C.S. reagent grade hydrochloric acid and heat gently until the iron oxide and zinc yellow are completely dissolved. Evaporate to dryness and heat at $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ($221^{\circ}\text{F} \pm 3.6^{\circ}\text{F}$) for 1 hour. Moisten the residue with a few drops of the hydrochloric acid, dilute to 100 ml with hot water, boil, filter, and wash with hot water. Transfer the filter paper and contents to a weighed porcelain crucible. Ignite, cool in a desiccator and weigh. From the weights of the ash and sample used, calculate the percentage of insoluble siliceous material in the pigment.

5. NOTES

5.1 If the pink color should persist because the permanganate is in excess, discard and begin again, adding a few ml less of the permanganate solution.

5.2 After the addition of the ammonium persulfate the elapsed boiling time should be not less than 15 nor more than 30 minutes.

6. PRECISION

6.1 No data.

METHOD 7340.1
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ZINC OXIDE AND ZINC CHROMATE IN PAINT PIGMENTS
(COMPLEXOMETRIC METHOD)

1. SCOPE

1.1 This method describes a rapid, direct, volumetric method for the analysis of mixed pigments containing zinc oxide or zinc chromate but is not applicable to the determination of zinc in zinc sulfide pigments. The only interference may come from the presence of alkaline earths, oxides or hydroxides of calcium or magnesium which are slightly soluble in the buffered mixture used to effect solution, but interference can be eliminated by the use of the alternate procedure described in 4.2.

2. APPARATUS

2.1 Flask. A 250 ml glass-stoppered Erlenmeyer flask.

3. REAGENTS

3.1 Buffer solution (pH 10). Dissolve 54 g of ammonium chloride in some water, add 350 ml of concentrated ammonium hydroxide and dilute to one liter with water.

3.2 Disodium ethylenediaminetetraacetate, 0.2 N. Dissolve 37.2 g of disodium ethylenediaminetetraacetate dihydrate in water and dilute to 1 liter.

3.3 Eriochrome Black T (0.5 percent). Dissolve 0.25 g of Eriochrome Black T indicator and 2.2 grams hydroxylamine hydrochloric (as stabilizer) in 50 ml methanol.

3.4 Primary standard. Weigh accurately, about 3 g of oven-dried, high purity zinc oxide. Dissolve in the buffer solution and dilute to 500 ml with the buffer solution. Use 10 to 25 ml pipetted aliquots as blanks for standardization with each day's analyses. Calculate the normality of the standard solution (Normality = weight of zinc oxide \times 0.04915).

4. PROCEDURE

4.1 Procedure when alkaline earth pigments are absent.

4.1.1 Weigh a sample of pigment that will contain the equivalent of 0.1 g of zinc oxide into a 250-ml glass-stoppered Erlenmeyer flask.

4.1.2 Pipette an accurate aliquot of 10 to 25 ml of the primary standard zinc oxide into a separate 250-ml flask.

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4.1.3 Add 25 ml of the ammonia-ammonium chloride buffer mixture to the sample, stopper and shake vigorously every few minutes over a period of 15 minutes. Filter through paper of fine porosity into a 400-ml beaker and dilute to 300-ml with water.

4.1.4 Titrate both sample and standard with disodium ethylenediaminetetraacetate using 10 drops of the Eriochrome Black T as indicator. Just before the end point is reached, add another five drops of the indicator. The color change will be from wine red to blue or from orange-red to green, depending on whether or not chromium is present.

4.2 Procedure when alkaline earth pigments are present.

4.2.1 Treat as in 4.1.1 and 4.1.3 except add 2 grams of ammonium diphosphate to the pigment sample before the buffer mixture is added. After 15 minutes of agitation add a Whatman filtration accelerator pad and agitate the sample again. Filter through paper of medium retention into a 400-ml beaker and wash the residue with at least 200 ml of a wash solution that contains 250 ml of the buffer mixture and 10 grams of the monobasic ammonium phosphate per liter of solution. Do not use water for washing. Proceed as in 4.1.4.

4.3 Calculation.

4.3.1 Calculate the normality of the reagent from the normality of the standard (see 3.4).

4.3.2 Calculate the percent zinc oxide:

$$\text{Percent ZnO} = \frac{\text{ml of EDTA} \times \text{normality of EDTA} \times 4.069}{\text{Weight of pigment sample}}$$

4.3.3 If preferred, percent zinc chromate can be calculated from the percent zinc oxide by the factor 2.62.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 7345
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KETONE CONTENT OF PAINT SOLVENTS

1. SCOPE

1.1 This method determines four of the ketones frequently used in paint thinners. It provides for both qualitative and quantitative analysis.

2. APPARATUS

2.1 Gas chromatograph equipped with a flame ionization detector, recorder, and capability for programmed column temperature.

2.2 Column. A 0.92 meter by 3.2 millimeters diameter Teflon-coated, stainless steel tube packed with 50-80 mesh porous polymer material.^{1/}

3. REAGENTS

3.1 Glacial acetic acid.

3.2 Sodium nitroprusside.

3.3 Isopropyl alcohol.

3.4 Ethyl alcohol.

3.5 Ammonium hydroxide.

3.6 p-nitrophenylhydrazine.

3.7 Hydrochloric acid.

3.8 Potassium hydroxide.

3.9 Levulinic acid.

3.10 2, 4-dinitrophenylhydrazine. Place 6.0-6.1 g of 2,4-dinitrophenylhydrazine (DNPH) into a 125 ml Erlenmeyer flask. Slowly and carefully add 30 ml of concentrated sulfuric acid while stirring and cooling in an ice bath. Stir about 1 hour to dissolve the DNPH. Let cool to room temperature, then carefully add 90 ml of a 7:2 mixture of 95 percent ethanol:water in 1- to 2-ml increments. Cool in an ice bath for about 10 minutes. Crystals may form. Transfer the reagent to a 200-ml Erlenmeyer flask with the aid of another 90 ml of the 7:2 95 percent ethyl alcohol:water mixture, adding it rapidly and with agitation. Cover the flask with an acid-resistant closure and allow to stand overnight. Transfer the reagent to a glass-stoppered bottle, filtering through glass wool if a residue is present.

^{1/} Porapak Q has been found suitable for this purpose.

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4. PROCEDURE

4.1 Qualitative tests for ketones. The solvent obtained from distillation method ASTM D 3272 is subjected to the following tests.

4.1.1 Qualitative test no. 1. Pipet 0.3 ml of the solvent into a micro test tube. Add 2 drops of glacial acetic acid followed by 2 drops of saturated sodium nitroprusside in 50:30:20 - water:isopropyl alcohol:ethyl alcohol. After thorough mixing, tilt the test tube to an angle of 30 degrees and add 5 drops of 28-percent ammonium hydroxide. Mix the solution and allow the color to develop for one minute. An orange-to-purple color is positive for methyl isobutyl ketone, methyl isoamyl ketone, or mesityl oxide. (Mineral spirits alone yields an off-yellow color).

4.1.2 Qualitative test no. 2. Place 1 drop of the solvent into a micro test tube to which has been added 2 mg of p-nitrophenylhydrazine dissolved in 10 ml of isopropyl alcohol and 5 drops of concentrated hydrochloric acid. Boil the mixture for 30 seconds and add 3 drops of a saturated solution of potassium hydroxide in 95:1 - isopropyl alcohol: water. An orange-to-red-violet color is positive for isophorone. The color changes from orange to purple as the isophorone concentration is increased from 2 to 5 percent. (Mineral spirits containing no ketones yield a bright yellow color when subjected to this test.) When either of the above qualitative tests is positive, the sample is analyzed further as follows.

4.2 Quantitative test.

4.2.1 Ketone extraction. Pipet 1.0 ml of the solvent into a 50 ml glass-stoppered Erlenmeyer flask containing a Teflon-coated magnetic stirring bar. Add 25 ml of the DNPE reagent and stopper the flask. (This procedure is designed for solvent mixtures containing 5 percent or less ketones. If more are found to be present, the original solvent blend should be quantitatively thinned with mineral spirits and an aliquot of this dilution carried through the procedure.) Stir for 1 hour after the initial appearance of a precipitate. Place the flask in a refrigerator and swirl periodically over a period of 100 minutes. Vacuum filter by transferring the precipitate and stirring bar to a 15-ml medium frit glass crucible using 25 ml of 1 N hydrochloric acid followed by 25 ml of water for the transfer and washing. Maintain the vacuum overnight. More crystals will form in the filtrate. Refilter through the same crucible using a minimum amount of water for transferring and washing. Maintain the vacuum for about 1 hour. Transfer the crucible containing the precipitate and stirring bar to a one-necked, 50-ml, resin-reaction flask. Add 40 ml of levulinic acid which contains 10 percent of 1 N hydrochloric acid by volume. Clamp the top, which is fitted with a Graham reflux condenser, in place and loosely stopper the top of the condenser. Warm on a steam bath for 80 minutes with periodic agitation. Cool the mixture to room temperature and quantitatively transfer it to a glass-stoppered container using ethyl ether. The ether solution of the extracted ketones is ready for the following treatment and chromatographic analysis.

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4.2.2 Sample for gas chromatographic analysis. Add 0.1 g of nonane (internal standard) weighted to the nearest milligram to the ether solution of the extracted ketones. Mix thoroughly. Transfer 2 ml of the mixture to a 5-inch by 1/2-inch test tube and add 1 ml of distilled water. Stopper the test tube, agitate gently, and cool to equilibrium in an ice-water bath. Add, dropwise, 1 ml of 40-percent aqueous sodium hydroxide. Maintain the temperature below 20° C and agitate intermittently for 5 minutes. Centrifuge the mixture until the two layers separate completely. Transfer most of the upper layer to a 2-ml, septum-fitted vial and reserve for chromatographic analysis.

4.2.3 Gas chromatographic analysis. With a 10 ml syringe inject 2 microliters of the solution prepared as above into the gas chromatograph which is operated under the following conditions:

Carrier (helium) flow-rate	25 ml/min
Injection port temperature	250° C
Flame ionization detector temperature	300° C
Initial column temperature	180° C
Rate of column temperature increase beginning at 10 min	3° C/min
Final column temperature	225° C
Total length of run	40 min

Identify the ketones from previously determined retention times.

Calculate the concentration of each ketone from the relationship:

$$C_x = C_{IS} \times \frac{A_x}{A_{IS}} \times F$$

where:

- C_x is the ketone concentration.
- C_{IS} is the internal standard concentration.
- A_x is the area under the ketone peak.
- A_{IS} is the area under the internal standard peak.
- F is a correction factor determined with known concentrations.

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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1. SCOPE

1.1 This method includes two procedures for the determination of the vinyl chloride-acetate copolymer in modified alkyds and for the phthalic anhydride content.

2. APPARATUS

2.1 Filter crucible. Prepare in advance a special filter crucible for the vinyl resin by adding a mat of asbestos fiber to a fritted glass crucible of coarse porosity, dry in an oven, cool in a desiccator, and weigh.

3. REAGENTS

3.1 Alcoholic potassium hydroxide, 0.125N. Prepare by dissolving 8.3 g of potassium hydroxide pellets in one liter of absolute ethyl alcohol by refluxing, then filter.

3.2 Dioxane

4. PROCEDURE

4.1 Vinyl resin.

4.1.1 Weigh accurately a sample of the vehicle isolated by method 4032, representing 0.1 to 0.2 g of vinyl resin (about 1 to 2 g of vehicle) into a 50-ml stoppered flask. Add 5 ml of 1,4-dioxane, mix and reweigh.

4.1.2 Pour 200 ml of the filtered 0.125 N alcoholic potassium hydroxide into a wide-mouth 500-ml glass-stoppered flask. While swirling the alkali solution, add the contents of the sample flask (see 4.1.1) very slowly, then stopper and reweigh the 50-ml flask. Without delay, shake the stoppered 500-ml flask constantly for 5 minutes, starting with the pouring of the sample (see 5.1).

4.1.3 Filter the sample promptly at the end of the agitation through the specially prepared filter crucible, collecting the filtrate in a clean, dry suction flask. When all the filtrate has drained through, set aside the filtrate for phthalic anhydride analysis (see 4.2). Continue the transfer and washing of the precipitate in the crucible with 95 percent ethyl alcohol but do not add the washing to the separated filtrate. Wash the crucible twice with water and finally with 95 percent ethyl alcohol. Dry at 60° C in an oven to constant weight (about 2 hours), cool and weigh.

4.2 Phthalic anhydride.

4.2.1 Transfer the filtrate from 4.1.3 to a 500-ml Erlenmeyer flask using absolute ethanol for a quantitative transfer. Add an additional 50 ml of filtered 1.0 N potassium hydroxide in absolute ethanol. Attach a condenser and

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reflux for two hours in a water bath. Cool and filter through a weighed Gooch or a medium porosity fritted glass crucible, using absolute ethanol as a wash medium. Dry the crucible at 105° C, cool in a desiccator, and weigh.

4.3 Calculations.

4.3.1 Determine the size of the resin sample from the three weighings made in 4.1.1 and 4.1.2 in the following manner:

$$\text{Actual sample weight} = \frac{B-C}{B-A} \times \text{original sample weight}$$

Where:

A = Weight of the stoppered empty flask

B = Weight of flask plus sample, plus dioxane (before pouring)

C = Weight of flask plus residual sample (after pouring)

4.3.2 Calculate the percent of vinyl chlorideacetate resin as follows:

$$\text{Percent resin} = \frac{\text{Weight of precipitate} \times 100}{\text{Weight of sample} \times \text{nonvolatile fraction}}$$

4.3.3 Calculate the percent phthalic anhydride as follows:

$$\text{Percent phthalic anhydride} = \frac{\text{Weight of dipotassium phthalate} \times 61.1}{\text{Weight of sample} \times \text{nonvolatile fraction}}$$

5. NOTES

5.1 There must be no delay in beginning the agitation of the sample and it must be filtered promptly at the end of the 5-minute period. The weighing of the sample flask may have to be delayed or accomplished by an assistant.

6. PRECISION

6.1 No data.

METHOD 7356.1
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SOLVENT CONTENT OF ENAMELS AND ENAMEL THINNERS
 (GAS LIQUID CHROMATOGRAPHY)

1. SCOPE

1.1 This method provides for the qualitative and quantitative analysis of solvents for enamels by application of gas liquid chromatography. Two separate procedures are included in this method to include analysis for total aromatics specifically, and a separate procedure for toluene and ethylbenzene specifically. (In this form some of the tests may be omitted for some samples, depending on specification requirements.) A test for olefinic compounds is included.

2. MATERIALS

2.1 Gas chromatograph equipped with a suitable recorder and thermal conductivity detector. The operating conditions for the two procedures are as follows:

	<u>6-ft. column</u>	<u>18-ft. column</u>
Detector cell temperature, °C	300	300
Detector cell current, ma	150	150
Injection port temperature, °C	300	300
Helium flow at exit, cc/minute	175	110
Column temperature, °C	125	100

2.2 Columns. Two lengths of 1/4-inch copper tubing, 6-feet and 18-feet long, both packed with 35 percent N, N-Bis (2-cyanoethyl) formamide on 60- to 80-mesh Chromosorb P.

2.3 Syringe, fixed needle, 10 microliter capacity.

3. REAGENTS

3.1 Benzene, A.C.S. reagent grade.

3.2 Acetone, A.C.S. reagent grade.

3.3 Phenylcyclohexane, high purity.

3.4 Potassium permanganate, 1 percent by solution in 95 mL of acetone and 5 mL of water.

3.5 Sulfuric acid. Mix concentrated sulfuric acid with 15 mL of water, cool, and dilute to 100 mL volume with concentrated acid.

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4. PROCEDURE

4.1 Total aromatic content - Procedure A. Transfer precisely 3 mL of distillate or thinner to a 25-mL glass-stoppered volumetric flask and add exactly 0.3 mL of high purity phenylcyclohexane. While cooling the graduate under tap water, add 15 mL of 85 percent sulfuric acid slowly. After all the acid has been added, shake vigorously for 2 minutes and allow the layers to separate. Add sufficient 85 percent acid to force the top layer into the neck of the flask and then transfer most of the top layer to a micro-separatory funnel. Wash the distillate with 5 mL portions of distilled water until all acid has been removed and reserve the washed solvent for chromatographic analysis. Install the 6-foot column and follow the operating conditions described above. Inject about 5 microliters of the acid-treated sample and allow the chromatogram to develop until the internal standard, phenylcyclo-hexane, emerges.

4.2 Calculate the percent of total aromatic solvents as follows:

$$\% \text{ total aromatic solvents, v/v} = \frac{A \times 1.07^* \times 10^{**}}{B}$$

where, A = area of aromatic solvent peaks.

B = area of internal standard peak.

* = correction factor for the detector response.

** = percentage of internal standard added.

4.3 Toluene and ethylbenzene - Procedure B (NOTE 1). Treat 3 mL of solvent in the same manner as described in procedure A except substitute benzene for phenylcyclohexane. Install the 18-foot column and follow the operating conditions described for that column. Inject about 3 microliters of sample and allow the chromatograph to develop until all of the xylene isomers appear. Purge the column by raising the column temperature to 120 °C. After the high boiling materials emerge, reset the column temperature to 100 °C. Calculate the percent of toluene and ethylbenzene as follows:

$$\% \text{ toluene, v/v} = \frac{(\text{area of toluene peak}) (1.017)^* (10)^{**}}{(\text{area of benzene peak})} \quad (\text{NOTE 2})$$

$$\% \text{ ethylbenzene, v/v} = \frac{(\text{area of ethylbenzene peak}) (1.054)^* (10)^{**}}{(\text{area of benzene peak})}$$

where, * = correction factor for the detector response.

** = percentage of internal standard added.

4.4 Test for olefinic or cyclo-olefinic compounds. Place 2-3 drops of the solvent or distillate into a small, clean test tube. Dissolve the sample in 1 mL of acetone and add 1 drop of 1 percent permanganate solution (see 3.4). Shake and allow to stand for 2 minutes. A positive test is indicated by the decolorization

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of the purple solution and the solvent is considered to contain olefinic or cyclo-olefinic compounds (NOTE 3).

5. NOTES

5.1 Note 1. If the total aromatic solvent content does not exceed the limitations of the specification for aromatics, procedure B may be omitted.

5.2 Note 2. Sensitivity of the instrument should be adjusted to keep peaks from running off the scale. Appropriate corrections must be made for changes in sensitivity when computing the peak areas.

5.3 Note 3. This test will not be positive for solvents containing less than 1 percent of these compounds.

6. PRECISION

6.1 No data.

METHOD 7360
January 24, 1986

SOLVENT CONTENT OF LACQUERS AND LACQUER THINNERS
(GAS CHROMATOGRAPHY)

1. SCOPE

1.1 This method describes the qualitative and quantitative analysis of solvents in lacquers and in lacquer thinners by programmed-temperature gas chromatography. Distillation of solvents from lacquers is avoided by precipitation of the resin.

2. APPARATUS

2.1 A gas chromatograph equipped with a suitable recorder, a flow control assembly to provide constant flow rate of the carrier gas through the detector during programmed temperature operation, and separate temperature controls for the injection port, detector, and column. The detector temperature should be maintained at 200° C, the injection port temperature at 175° C, and the detector cell current at 150 mA (for thermal conductivity only), and the gas flow should be adjusted to 60 ml per minute at the exit.

2.2 Column. 2.5 meters of 6-mm diameter stainless steel tubing packed with 20 percent by weight of didcyl phthalate on 60- to 80-mesh Chromosorb P.

2.3 Syringe. Fixed needle, 10 microliter capacity.

3. REAGENTS

3.1 Pentane.

4. PROCEDURE

4.1 Pour approximately 2 or 3 g of lacquer into a small testtube (13 X 100 mm) and insert a small stirring rod. Precipitate the resins by the slow addition of 3 ml of pentane with constant stirring of the sample.

4.2 Heat the chromatographic column to 50° C. Withdraw a 5 microliter sample of the supernatant liquid prepared in 4.1 and introduce on the column. Immediately engage the temperature programming mechanism for a column-heating rate of 3° C per minute. Set the attenuation control at a point of low sensitivity for the first five minutes and change to more sensitive positions as the chromatogram develops, recording the settings. Discontinue the temperature increase at 140° C, but allow the chromatogram to develop for a total of 40 minutes.

4.3 Identification of the major constituents.

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4.3.1 The retention time of each constituent may be measured relative to benzene. For this purpose, table I is provided as a guide, but may not be completely accurate for all chromatographic columns.

TABLE I. Relative retention data.

Solvent	Retention time relative to benzene
Ethanol	0.42
Isopropanol	.47
Ethyl acetate	.65
Methyl ethyl ketone	.75
Isopropyl acetate	.85
Benzene	1.00
n-Butanol	1.41
Methyl isobutyl ketone	1.51
Toluene	1.64
n-Butyl acetate	1.74
Methyl isobutyl carbinol	1.82
n-Amyl acetate	2.04
Methyl amyl acetate and ethyl benzene	2.14
m-Xylene and p-Xylene	2.19
o-Xylene	2.30
Butyl cellosolve	2.55
Cellosolve acetate	2.30

4.4 Calculation.

4.4.1 Determine the detector response correction factor (see 5.1) for each solvent likely to be encountered by chromatographing known mixtures of each with the internal standard. Retain the data for future reference.

4.4.2 Measure the area of each peak obtained from chromatographing the unknown samples, and correct for detector response.

4.4.3 Calculate the percent of each solvent as follows:

$$\text{Percent} = \frac{\text{Corrected area of solvent peak} \times 100}{\text{Total corrected peak areas}}$$

5. NOTES

5.1 The correction factors for the detector responses are dependent on the internal standard used and on the performance of the chromatograph, and should be determined daily.

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6. PRECISION

6.1 No data.

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METHOD 7361.1
 January 24, 1986

SOLVENT CONTENT OF LACQUERS CONTAINING NAPHTHA DILUENT
 (GAS LIQUID CHROMATOGRAPHY)

1. SCOPE

1.1 This method provides for the qualitative and quantitative analysis of solvents for lacquers containing aliphatic hydrocarbons, by application of gas liquid chromatography. Lacquers that do not contain naphtha type diluents may be analyzed by method 7360. The solvents in lacquers must first be removed by ASTM D 3272, "Distillation of Solvents from Enamels and Lacquers". Determinations may be made on a weight or volume basis.

2. MATERIALS

2.1 Gas chromatograph equipped with a suitable recorder and thermal conductivity detector, equipped for programmed temperature operation. The operating conditions are as follows:

Detector cell temperature, ° C	300
Detector cell current, ma	160
Injection port temperature, ° C	250
Helium flow at exit, cc/minute	100
Column heating rate, ° C/minute	2
Initial column temperature, ° C	60
Terminal column temperature	140

2.2 Column. Two lengths of 1/4-inch copper tubing: an 8-foot section packed with 20 percent by weight of N, N-Bis (2-cyanoethyl) formamide on 60- to 80-mesh Chromosorb P and a 12-foot length packed with 30 percent by weight of diethylene glycol succinate on 60- to 80-mesh Chromosorb P. The two sections are then coupled together and installed so that the sample passes first through the 8-foot section.

2.3 Syringe, fixed needle, 10 microliter capacity.

3. PROCEDURE

3.1 Weigh 5 ml of the isolated solvent into a small stoppered flask. Rapidly weigh 1 ml of internal standard, ethyl acetate, into the same flask. Mix thoroughly and inject about 3 microliters of sample onto the column and chromatograph according to the directions described above. If the lacquer contains ethyl acetate, alternate internal standards such as methyl isobutyl ketone, butyl acetate, etc., may be used.

4. CALCULATION

4.1 Measure the area of each solvent peak and relate to the area of the known amount of internal standard. Compute percentages on either a weight or volume basis as required, as follows:

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$$\text{Percent solvent, weight basis} = \frac{A \times F \times P}{I \times S}$$

where, A = area of solvent peak.

F = detector response correction factor for weight (see method 7360).

P = weight of internal standard x 100.

I = area of internal standard peak.

S = weight of sample.

$$\text{Percent solvent, volume} = \frac{A \times F \times P}{I \times S}$$

where, A = Area of solvent peak.

F = detector response correction factor for volume (NOTE 1).

P = volume of internal standard x 100.

I = area of internal standard peak.

S = volume of sample.

5. NOTES

5.1 Note 1. Detector response correction factors for volume can be calculated from the specific gravity of each solvent and the weight basis correction factors.

6. PRECISION

6.1 No data.

METHOD 7371.1
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DETERMINATION OF PLASTICIZERS IN LACQUERS
(GAS-LIQUID CHROMATOGRAPHY)

1. SCOPE

1.1 This method describes a recommended practice for the qualitative and quantitative analysis of lacquers for plasticizer content by direct separation through the application of programmed temperature gas chromatography.

2. APPARATUS

2.1 Gas chromatograph equipped with a suitable recorder and a flow-control assembly to provide constant mass flow-rate of helium gas through the detector during programmed temperature operation, and separate controls for injection port, detector and column temperatures. The detector cell temperature should be maintained at 300 °C, injection port temperature 330 °C, detector cell current 160 ma, and the gas flow adjusted to 120 cc per minute at exit.

2.2 Column. Six feet of 1/4-inch diameter copper tubing packed with 20 percent by weight of silicone grease on acid and alkali washed chromosorb W, 60- to 80-mesh.

2.3 Syringe. Fixed needle, 50 microliter capacity.

2.4 Magnetic stirrer and bar.

3. REAGENTS

3.1 Acetone. A.C.S. reagent grade.

3.2 Hexanes. A.C.S. reagent grade.

3.3 Petroleum ether.

3.4 Butyl sebacate.

4. PROCEDURE

4.1 Weigh accurately about 100 mg of butyl sebacate or butyl adipate (preferably the former) as an internal standard into a 25-mL flask followed by 2 or 3 g of the lacquer vehicle which is also accurately weighed. Add 1 mL of

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acetone and insert a small magnetic stirring bar. With rapid stirring, add 3 mL of hexanes dropwise at the rate of 80-100 drops per minute, from a buret. Continue the stirring and add 3 mL of petroleum ether in the same manner. Filter through a rapid filter paper into a 25 mL beaker, add an antibumping stone and place in a 70 °C water bath. Remove the beaker as soon as boiling subsides.

4.2 Heat the chromatographic column to 210 °C, set the attenuation at a point of low sensitivity and inject 10 to 20 microliters of sample onto the column. Immediately engage the temperature programming mechanism for a column heating rate of 4 °C per minute. After the solvents have emerged, reset the sensitivity according to the type and amount of plasticizer anticipated. When the maximum temperature of 290 °C has been reached, maintain until the chromatogram has completely developed.

4.3 Identification.

4.3.1 Identify the peaks of the chromatogram by calculating their retention times, relative to the internal standard and compare to the calibration chart (table I).

TABLE I. Relative retention data for plasticizers.

Plasticizer	Relative retention time (Dibutyl sebacate = 1)
Dimethyl phthalate	0.16
Diethyl phthalate	.26
Dibutyl phthalate	.67
Butyl benzyl phthalate	1.32
Di-(2-ethylhexyl) phthalate (DOP)	1.65
Tricresyl phosphate	1/1.78-1.978
Diocetyl sebacate	2.20

1/ TCP is usually a mixture of isomers and will emerge as several peaks within the range indicated.

4.4 Calculation.

4.4.1 Determine the detector response correction factor for each plasticizer likely to be encountered in lacquer analysis by chromatographing known mixtures of each with the internal standard, and retain the data for future reference.

4.4.2 Measure the area of each peak obtained from chromatographing the unknown sample (see 4.2), correct for detector response, and calculate the amount of plasticizer by comparison to the internal standard.

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5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 7375
 January 24, 1986

SOLVENT CONTENT (INCLUDING AMINES) OF WATER BASED
 PAINTS BY GAS CHROMATOGRAPHY

1. SCOPE

1.1 This method describes the quantitative determination of volatile organics in water based paints. It is believed to be applicable to a broad range of volatile components including alkoxy ethanols, alcohols, and amines.

2. APPARATUS

2.1 Gas chromatograph equipped with a thermal conductivity detector.

2.2. Chromatographic column. A glass column 2 meters by 3 millimeters diameter packed with an amine-suitable material. The packing consisting of 28 percent Pennwalt 223 plus 4 percent potassium hydroxide on 80-100 mesh gas chrom R has been found satisfactory. Other satisfactory columns may be used.

2.3 Syringe, 10 microliter capacity.

3. REAGENTS

3.1 None.

4. PROCEDURE

4.1 Operate the gas chromatograph under the following conditions:

Detector temperature, ° C.	250
Injection port temperature, ° C.	230
Column oven	
Initial temperature, ° C.	100
Final temperature, ° C.	150
Rate of temperature increase beginning at time of injection, ° C/minute.	10
Time at 150° C, minutes.	20
Carrier (helium), milliliters per minute.	30

Accurately weigh about 3 grams of the paint into a 120 mm by 17 mm centrifuge tube. Add about 0.3 grams, accurately weighed, of a suitable internal standard.^{1/} Mix thoroughly and centrifuge until most of the pigment has

^{1/} Select a non-interfering internal standard by comparing its retention time with those of a preliminary run of the paint sample without an internal standard.

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settled. Inject 2 microliters of the supernatant into the sample port of the gas chromatograph and develop the chromatogram under the conditions described in 3.1. Identify the solvent components from retention times given in table I. These retention times were determined for the column described in 2.2.

TABLE I. Retention data.

Solvent	Retention time, minutes
Ethyl alcohol	1.9
Triethylamine	2.7
Methyl cellosolve	5.2
Cellosolve	7.4
Cellosolve acetate	13.5
Butyl cellosolve	17.5

Concentration C_x of an individual solvent component is calculated in weight/weight percent from the equation.

$$C_x = \frac{A_x W_{IS} F \times 100}{A_{IS} W_s}$$

where: A_x is the peak area of the solvent component.
 A_{IS} is the peak area of the internal standard
 W_{IS} is the weight of the internal standard.
 W_s is the weight of the paint.
 F is a predetermined correction factor.

5. NOTES

5.1 None

6. PRECISION

6.1 No data.

METHOD 7381
January 24, 1986

HYDROXYL NUMBER

1. SCOPE

1.1 This method is to be used to determine the hydroxyl value of the polyester portion of two-package urethane coating systems. The result is affected by acid value of the sample and a correction must be made after the acid number of the sample has been obtained.

2. APPARATUS

2.1 Magnetic stirrer and bar.

3. REAGENTS

3.1 Acetic anhydride in ethylacetate, 2 N. Add 7.2 g of reagent grade p-toluenesulfonic acid monohydrate to 180 ml of A.C.S. reagent grade ethyl acetate in a clean, dry 250-ml amber-colored reagent bottle. Insert a magnetic stirring bar and agitate until the acid is completely dissolved. Add slowly, 60 ml of A.C.S. reagent grade acetic anhydride, maintaining the agitation during the addition. This reagent is good for several days.

3.2 Potassium hydroxide in methanol, 0.56 N. Dissolve 37 grams of reagent grade potassium hydroxide in 225 ml of absolute methanol by warming. Cool and filter and dilute to one liter with absolute methanol. Standardize against potassium acid phthalate.

3.3 Mixed indicator. Mix 1 part of 0.1 percent neutralized cresol red with 3 parts of 0.1 percent neutralized thymol blue.

4. PROCEDURE

4.1 Weigh accurately a 1- to 2-g sample of the vehicle from a dropping bottle into a 250-ml Erlenmeyer flask. Pipette 5 ml of the 2 N acetic anhydride in ethyl acetate into the sample flask and into a separate flask for use as a blank. Cover with a watchglass and immerse to just above the liquid level in a constant temperature water bath maintained at 50° C. After 5 minutes, remove the flask and swirl gently, then return it to the bath and allow the reaction to proceed for an additional 10 minutes. Add 2 ml of water and swirl vigorously. Add 10 ml of a 3-to-1 pyridine-water-mixture, rinsing the sides of the flask during the addition. Allow the flask to stand 5 minutes at room temperature. Add indicator to a distinct coloration, then titrate both sample and blank with the 0.56 N alkali, taking the color change from yellow to blue-violet as the end point.

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4.2 Calculation.

4.2.1 Correct the difference between the volume of alkali used for the sample and blank for any free acidity as determined in the acid number method, then apply the calculation for hydroxyl number as follows:

$$\text{Hydroxyl number} = \frac{\text{Corrected volume of alkali (ml)} \times \text{normality} \times 56.1}{\text{Weight of sample in grams} \times \text{nonvolatile fraction}}$$

5. NOTES

5.1 None.

6. Precision

6.1 No data.

METHOD 7391
January 24, 1986

AMINE NITROGEN CONTENT

1. SCOPE

1.1 This method is used to measure the amine and polyamine content of the activator portion of two-package epoxy coating systems.

2. APPARATUS

2.1 Flask. A 250-ml Erlenmeyer flask.

2.2 Stirring apparatus. Magnetic with a Teflon covered stirring bar.

2.3 Buret. Twenty-five ml.

2.4 Drying tube.

3. REAGENTS

3.1 Perchloric acid in acetic acid, 0.1 N. Prepare by adding 24 ml of acetic anhydride and 8.9 ml of 70 percent perchloric acid to 1,000 ml of glacial acetic acid. Mix, cool, and standardize against weighed quantities of potassium acid phthalate dissolved in glacial acetic acid using methyl violet as indicator.

) Correct the titration, if necessary, by running a blank on the same volume of acetic acid used to dissolve the standard. Calculate the normality of the reagent.

$$\text{Normality} = \frac{\text{grams of potassium biphthalate} \times 1,000}{(\text{ml of reagent for standard} - \text{ml for blank}) \times 204.22}$$

4. PROCEDURE

4.1 Weigh accurately about 0.5 g of the activator into a 250-ml Erlenmeyer flask and add 25 ml glacial acetic acid followed by 50 ml of methyl isobutyl ketone. Protect the sample, solvents and reagents from exposure to moisture of the atmosphere. Add a few crystals of methyl violet as indicator, sufficient to give a strong purple color. Insert a dry teflon-covered magnetic stirring bar. Attach a vented rubber stopper to the tip of a dry, 25-ml buret in such a manner that the sample will be protected from moisture during titration. When the buret is filled with reagent, attach a drying tube to the upper end. Attach the sample flask to the stopper on the buret, stir slowly and titrate with a perchloric acid reagent to a blue-green end point.

4.2 Run a blank titration on a mixture of 25 ml of the acetic acid and 50 ml of methyl isobutyl ketone.

4.3 Calculation.

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4.3.1 Calculate the amine nitrogen content:

Percent nitrogen = $\frac{(\text{ml of reagent for sample} - \text{ml for blank}) \times \text{normality} \times 14.0}{10 \times \text{weight of sample}}$

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 7401
January 24, 1986

EPOXY RESIN IN COATING VEHICLES

1. SCOPE

1.1 This method describes a procedure for determining epoxy resins of the bisphenolepichlorohydrin type in coating vehicles including amine, polyamide, and phenol-catalyzed enamels, fatty acid esters and simple mixtures. Silicone resins are known to interfere, causing high yields.

2. APPARATUS

2.1 Volumetric flask. A 50- or 100 ml- ± 0.1 ml.

2.2 Volumetric flask. A 125- or 250-ml ± 0.2 ml.

2.3 Water bath. Capable of maintaining temperatures of 60° C and $75^{\circ} \pm 1^{\circ}$ C.

2.4 Filter. A 50-ml fritted glass crucible of medium porosity.

3. REAGENTS

3.1 Nitric acid, fuming, 90 percent.

3.2 Sodium hydroxide, aqueous, 0.1 N. Four grams per liter.

3.3 Sodium hydroxide, aqueous, 1.0 N. Forty grams per liter.

4. PROCEDURE

4.1 Weigh a sample of the vehicle or resin solution of not more than one gram to the nearest milligram and dissolve in a low-boiling solvent such as acetone, methyl ethyl ketone, or a 1:1 mixture of benzene:ethanol and dilute to a convenient volume such as 50 ml or 100 ml in a volumetric flask. Withdraw an aliquot that is estimated to contain not more than 50 milligrams of epoxy resin and transfer to a glass-stoppered Erlenmeyer flask of 125- or 250-ml capacity. Evaporate the solvents with a gentle current of air by placing the flask in a water bath at 60° C so that the sample dries in a uniform thin film on the bottom of the flask. Add about 3 ml of acetone and redry the sample.

4.2 Add 5 ml of 90 percent fuming nitric acid to the flask, stopper and swirl frequently until most of the sample appears digested. Vent the stopper and place the flask in a water bath and increase the temperature to 75° C. After 90 minutes at the 75° temperature, cool to room temperature and cautiously add 100 ml of water from a graduate. Stopper the flask and swirl the sample for a full minute. If, upon settling of the precipitate, the supernatant liquid appears very cloudy, agitate repeatedly until maximum clarity is obtained. Filter the sample through a large, 50 ml fritted glass crucible of medium porosity and wash

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thoroughly with water. Wash the collected precipitate alternately with 25-ml portions of aqueous alkali and with water, using the 0.1 N alkali for the first two washings and the 1.0 N alkali for the second two washings. Finally, wash thoroughly with water to remove all traces of alkali.

4.3 Dry the crucible in an oven at 60° C for several hours, cool in a desiccator and weigh.

4.4 If styrene is known to be present in the original sample or has been detected qualitatively, wash the crucible under vacuum with acetone until all acetone soluble matter is removed, redry and weigh, calculating the soluble portion as the epoxy resin.

4.5 Calculation.

Percent epoxy resin = $\frac{\text{weight of precipitate} \times 0.606 \times 100}{\text{Weight of aliquot sample} \times \text{nonvolatile fraction}}$

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

METHOD 7409
January 24, 1986

EPOXY RESIN CONTENT IN PAINTS CONTAINING AN
ACRYLIC LATEX RESIN

1. SCOPE

1.1 This method determines the amount of epoxy resin in paints in the presence of an acrylic latex resin.

2. APPARATUS

2.1 Soxhlet extraction assembly.

2.2 Conventional laboratory equipment.

3. REAGENTS

3.1 Sodium chloride

3.2 Benzene

4. PROCEDURE

4.1 Accurately weigh by difference 5 to 7 grams of the well mixed paint into a mortar containing 30 grams of sodium chloride moistened with 2 ml of water. Mix well and place in a vacuum desiccator over Drierite for 16 hours with vacuum pump running.

4.2 Grind the salt paint mixture, transfer to a 43 x 123 mm Soxhlet extraction thimble, and cover with glass wool. Extract with 400 ml of benzene under vigorous reflux for 4 hours. The Soxhlet extraction apparatus should be equipped with a calcium chloride tube and a few Berl saddles in the 250 ml Erlenmeyer flask.

4.3 At the conclusion of the extraction period remove the thimble and rinse with benzene. Transfer the extract to a tared 250 ml beaker containing 2 Berl saddles. Evaporate the extract on a steam plate under a light stream of nitrogen. When all of the solvent has evaporated, dry overnight in a vacuum oven at 70° C. Cool in a desiccator and weigh.

4.4 Calculation.

$$\text{Epoxy resin percent} = \frac{\text{net weight of benzene extract}}{\text{weight of paint sample}} \times 100$$

5. NOTES

5.1 None.

6. PRECISION

6.1 No data.

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METHOD 7427
January 24, 1986

FREE DIISOCYANATE IN URETHANE MATERIALS OR PREPOLYMERS
(Volumetric Distillation Method)

1. SCOPE

1.1 This method describes a procedure for the determination of the free toluene diisocyanate or hexamethylenediisocyanate content of low molecular weight urethanes. It covers products containing no more than 2.0 percent of the free isocyanates mentioned above. Higher concentrations can be dealt with by altering the sample size. Interference from higher molecular weight diisocyanates is not encountered since they are not separated from the polymer by the distillation. The method is not applicable when the material contains dibutyl tin dilaurate or other catalysts.

2. APPARATUS

- 2.1 Buret, 10 ml, graduated to .05 ml divisions.
- 2.2 Distillation flask, 50 ml (see figure 1).
- 2.3 Oil bath, constant temperature, for 150° C \pm 2°.
- 2.4 Test tube with side arm, 20 mm x 150 mm (see figure 1).
- 2.5 Vacuum pump, with manometer and teflon needle-valve control.

3. REAGENTS

- 3.1 Bromphenol blue indicator, 0.1 percent in ethyl alcohol.
- 3.2 Cellosolve acetate (ethylene glycol monoethyl ether acetate), 99 percent polyurethane grade^{1/}. (see note 1).

NOTE 1. Unless a fresh supply of known purity of cellosolve acetate and diphenyl ether is available, it is advisable to dry over molecular sieve, type 4A, for a minimum of 24 hours and withdraw by pipet as needed.

3.3 Dibutylamine reagent, approximately 0.25 N. Add 10 ml of high purity dibutylamine^{2/} to high purity chlorobenzene^{3/} and dilute to 250 ml with chlorobenzene. Store in a brown glass-stoppered bottle. If materials of the purity indicated are not available, the dibutylamine should be freshly distilled and the chlorobenzene should be dried over calcium chloride and redistilled.

- ^{1/} Union Carbide Chemicals Co. product has been found satisfactory.
- ^{2/} Eastman Organic Chemical list No. 1260 has been found satisfactory.
- ^{3/} Fisher Scientific Co. Index No. B-255 having certified analysis and containing 0.02 percent maximum water content, is satisfactory.

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3.4 Diphenyl ether^{4/} (see note 1.).

3.5 Hydrochloric acid ethyl alcohol, 0.1 N, Standardized.

3.6 Methanol, absolute.

4. PROCEDURE

4.1 Oven-dry and cool all glassware used in this method including pipets shortly before being used.

4.2 Weigh accurately about 5 grams of the urethane sample in the 50 ml distillation flask containing Berl saddles or pieces of porous plate. As soon as possible, add from a pipet, 5 ml of a 1:1 mixture of cellosolve acetate and diphenyl ether. Insert a tight fitting rubber stopper and swirl the flask to dissolve the sample. Attach the distillation assembly and immerse the receiver in a dry ice-acetone bath. Raise the oil bath which has previously been preheated to 150° C around the flask. Evacuate the system cautiously by means of a vacuum pump, over a 5 to 10 minute period, to 1 - 3 mm of mercury pressure, then maintain these conditions for 10 minutes.

4.3 Remove the distillation flask from the oil bath, release the vacuum, disconnect the apparatus at the neoprene tubing and remove from the dry ice-acetone bath, but keep the delivery tube in the receiver. Raise the stopper slightly off the receiver and add from a pipet, exactly 5 ml of the dibutylamine reagent into the receiver. Rinse the delivery tube through the upper end with a small amount of cellosolve acetate using a medicine dropper. While the receiver is warming to room temperature, allow the reagent to contact all of the solid particles in the delivery tube by drawing up some of the mixture with a rubber bulb or a pipet control. After all solids are dissolved, remove the delivery tube and transfer the entire sample quantitatively to a 250 ml Erlenmeyer flask using absolute methanol for washing. Dilute to approximately 100 ml volume with absolute methanol. Add several drops of the bromphenol blue indicator and titrate with the 0.1 N hydrochloric acid from a 10 ml buret, to a permanent yellow color. Titrate 5 ml of the dibutylamine reagent as a blank.

4.4 Calculation.

4.4.1 Percent free toluene diisocyanate =

$$\frac{(\text{ml of acid for blank} - \text{ml for sample}) \times \text{normality of acid} \times 8.7}{\text{weight of sample}}$$

4.4.2 Percent free hexamethylenediisocyanate =

$$\frac{(\text{ml of acid for blank} - \text{ml for sample}) \times \text{normality of acid} \times 8.4}{\text{weight of sample}}$$

^{4/} Eastman No. 104 and Fisher Scientific index D-89 have been found satisfactory.

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5. NOTES

5.1 None.

6. PRECISION

6.1 Collaborative studies have been carried out over a 2 year period involving four samples and ten laboratories. Total degrees of freedom were 50 for repeatability and 38 for reproducibility.

6.1.1 Repeatability. At the 95 percent confidence level the precision is 0.13.

6.1.2 Reproducibility. At the 95 percent confidence level the precision is 0.30.

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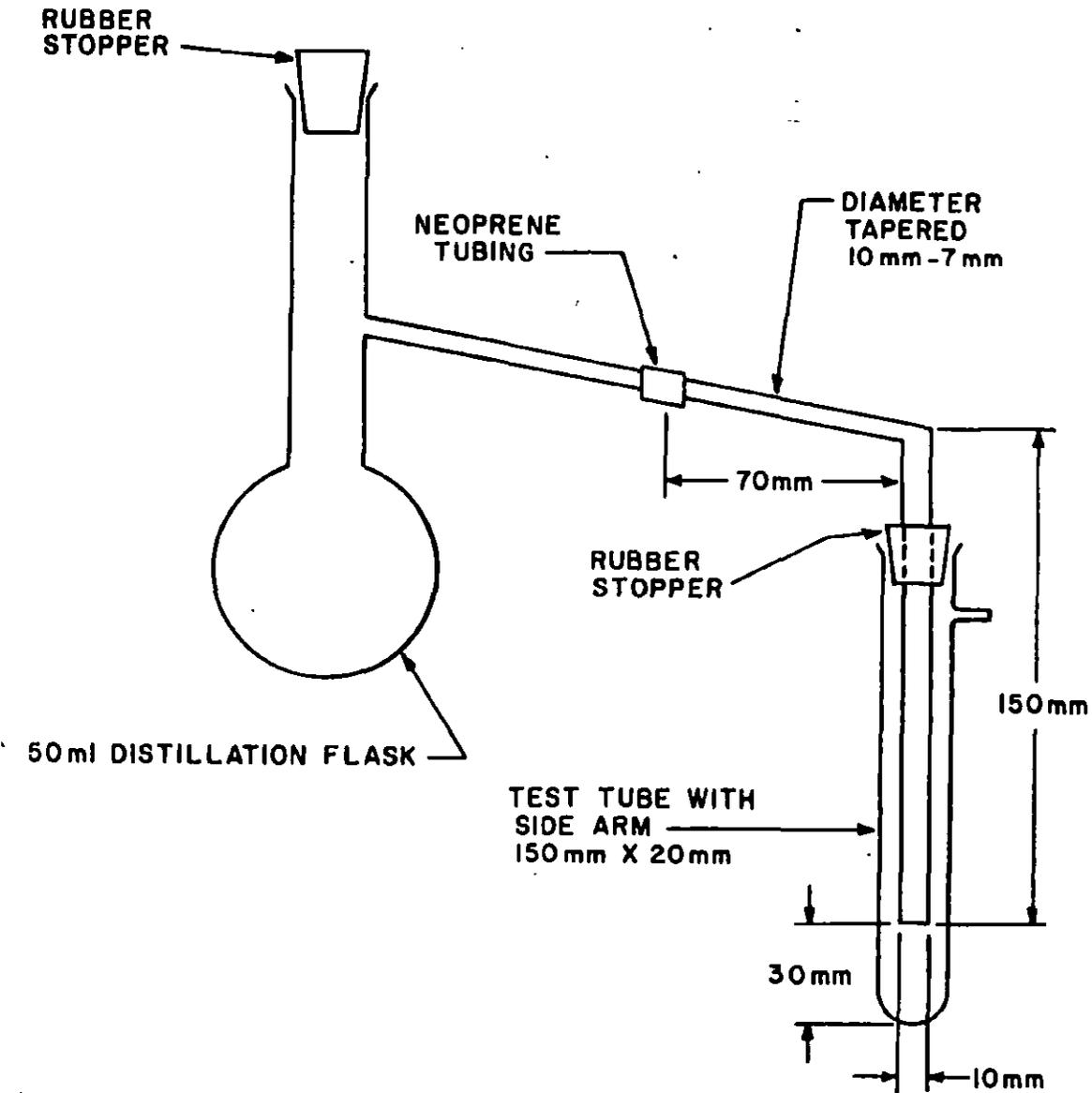


FIGURE 1. Distillation assembly.

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