Designation: A 657/A 657M – 03

Standard Specification for Tin Mill Products, Black Plate Electrolytic Chromium-Coated, Single and Double Reduced[1]

This standard is issued under the fixed designation A 657/A 657M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers single- and double-reduced tin mill black plate electrolytically coated with chromium and chromium oxide. The steel is furnished in coils and cut lengths for use in the manufacture of cans, closures, crowns, and other products.

1.2 This specification is applicable to orders in either inch-pound units (as A 657) which is supplied in thicknesses from 0.0050 to 0.0149 in. or in SI units [as A 657M] which is supplied in thicknesses from 0.127 to 0.378 mm.

1.3 The values stated in either inch-pound or SI units are to be regarded as standard. Within the text, the SI units are shown in brackets. The values stated in each system are not exact equivalents. Therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with this specification.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

A 623 Specification for Tin Mill Products, General Requirements[2]

A 623M Specification for Tin Mill Products General Requirements [Metric][2]

A 700 Practices for Packaging, Marking, and Loading Methods for Steel Products for Domestic Shipment[3]

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water[4]

3. Ordering Information

3.1 Orders for product under this specification shall include the following information, as required and applicable, to adequately describe the desired product:

3.1.1 Name of product (include whether single- or double-reduced),

3.1.2 Thickness (must be consistent with intended application),

3.1.3 Oiling (Section 6),

3.1.4 Steel type (MR, L, etc.),

3.1.5 Temper designation (T-3, T-4, etc. for single-reduced) or mechanical designation (DR-8, DR-9, etc., double-reduced),

3.1.6 Surface finish (7B, 7C, etc.).

3.1.7 Coil width or cut size,

3.1.8 Rolling direction must be specified on cut sizes and should be indicated by underlining the slit (rolling width) dimension,

3.1.9 On coils, specify minimum or range of acceptable inside diameters. The standard inside diameter is approximately 16 in. if ordered as Specification A 657 [410 mm if ordered as Specification A 657M]. Coils should be specified to a maximum coil weight if ordered as Specification A 657 [mass if ordered as Specification A 657M], or maximum outside diameter, or both.

3.1.10 Packaging,

3.1.11 Special requirements where applicable, and,

3.1.12 ASTM specification designation and year of issue.

NOTE 1—A typical ordering description for coils is as follows: 1250 base boxes, single reduced, black plate electrolytic chromium coated, DOS, (0.0093 in.) MR, T-3 BA, 35⅜ in. by coil, 16 in. inside diameter, 59 in. maximum outside diameter, 25 000 lb maximum coil weight, for regular crowns in accordance with Specification A 657/A 657M - XX. [250 Sitas, single reduced, black plate electrolytic chromium coated, DOS, 0.235 mm thickness, MR, T-3 BA, 513, 900 mm by coil, 410 mm inside diameter, 1500 mm maximum outside diameter, 11 500 kg maximum coil mass for regular crowns in accordance with Specification A 657/A 657M - XX].

NOTE 2—A typical ordering description of cut sizes is as follows: 1000 base boxes, double reduced black plated electrolytic chromium coated, BSC, 0.0061 in., MR, DR CA 8, 31 by 32 in., for cap closures in accordance with Specification A 657/A 657M - XX. [200 SITAS, double

[1] This specification is under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel, and Related Alloys and is the direct responsibility of Subcommittee A01.20 on Tin Mill Products.

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[2] *Annual Book of ASTM Standards*, Vol 01.06.

[3] *Annual Book of ASTM Standards*, Vol 01.05.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

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reduced, black plate electrolytic chromium coated, DOS, 0.155 mm thickness, MR, DR8 CA, 788 by 813 mm, cap closures in accordance with Specification A 657/A 657M - XX].

NOTE 3—The production of coils does not afford the same opportunity for inspection, grading, and sorting as does the production of cut sizes. Accordingly, appropriate processing and quality-control procedures are required by the purchaser to obtain optimum utilization of the material. Cut sizes are line inspected visually and mechanically during production. Sheets having surface imperfections that will not interfere with their utilization are included.

NOTE 4—Tin mill products are supplied on an area basis expressed in base boxes. In coils, the number of base boxes is computed from the measured length and the specified width. In cut sizes, the number of base boxes is computed from the specified length and width dimensions and sheet count. For calculating weight, the density of steel for tin mill products is 0.2836 lb/in.^{3} [Tin mill products are supplied on an area basis expressed in SITAS. In coils, the number of SITAS is computed from the measured length and the specified width. In cut sizes, the number of SITAS is computed from the specified length and width dimensions and sheet count. For calculating mass, the density of steel for tin mill products is 7850 kg/m^3].

4. Chromium Coating Weights [Masses]

4.1 *Total Coating* is made up of chromium metal and chromium oxides. These are determined separately.

4.1.1 *Metallic Chromium* is expressed as milligrams of chromium metal per square foot [milligrams of chromium per square metre] on each side of the sheet or strip.

	Average	
Nominal	Min	Max
mg/ft^2[mg/m^2]	mg/ft^2 [mg/m^2]	mg/ft^2 [mg/m^2]
5 [54]	3 [32]	13 [140]

4.1.2 *Chromium Oxide* is expressed as milligrams of chromium in the oxide per square foot [milligrams of chromium per square metre] as shipped on each side of the sheet or strip.

Average	
Min,	Max,
mg/ft^2 [mg/m^2]	mg/ft^2 [mg/m^2]
0.7 [8]	2.5 [27]

4.2 *Sampling for Chromium Coating Weights [Masses]*:

4.2.1 *Coils*—When the purchaser wishes to make tests to ascertain compliance with the requirements of this specification for metallic chromium and chromium oxide weights [masses] on a lot of any specific item of electrolytic chromium-coated steel in coils, use the following procedure:

4.2.1.1 Take one test strip at random from a representative section of each coil across its width. Select a minimum of three test strips, each taken from different coils from any one item of a specific lot. Spot test each test strip at three positions (edge, center, edge) across the rolling direction of the strip. Spot samples are customarily disks or squares, each of 4 in.^{2} [25.8 cm^2] in area. In order to secure representative samples, take samples so that the outer edge of the sample is at least 1 in. [25 mm] from either edge of the test strip. The average value of all spot tests on all test strips represents the average coating-weight [mass] test value.

4.2.1.2 From one- or two-coil lots, sample each coil. A minimum of three randomly selected test strips shall represent the lot.

4.2.2 *Cut Lengths*—When the purchaser wishes to make tests to ascertain compliance with the requirements of this specification for metallic chromium and chromium oxide weights [mass] on a specification of electrolytic chromium-coated steel in cut sizes, use the following procedure:

4.2.2.1 Take one sheet at random from each 50 packages, or part thereof, with a minimum of three different sample sheets, each taken from different bundles from any one item of a specific shipment. Spot test each sample at three positions across the rolling direction of the sheet (edge, center, edge). Spot samples are customarily disks or squares, each of 4 in.^{2} [25.8 cm^2] in area. In order to secure representative samples, take edge specimens so that the outer edge of the sample is at least 1 in. [25 mm] from either edge of the sheet. The average value of all spot tests on all sheets tested represents the average coating-weight (mass) test value.

4.3 *Determination of Chromium Coating Weights [Masses]*—Typical method for determining metallic chromium and chromium oxide weights [masses] for electrolytic chromium-coated steel is described in Annex A1, Annex A2, Annex A3, and Fig. A1.1 and Fig. A3.1.

5. Surface Appearance and Finish

5.1 *Single-Reduced Base Metal Finish*—Single-reduced electrolytic chromium coated steel is produced with ground roll finishes and shot-blasted roll finishes as follows:

5.1.1 *Ground Roll Finishes*:

5.1.1.1 *7B*—A smooth finish that may contain fine grit lines. This finish is specified for special applications.

5.1.1.2 *7C*—A smooth finish with grit lines. The surface roughness will range from 10 to 25 µin. Ra [0.254 to 0.635 µm Ra]. This is the standard finish for tin mill products.

5.1.2 *Blasted and/or Otherwise Textured Roll Finishes*:

5.1.2.1 *5C*—A shot-blasted finish (SBF) for general applications. The surface roughness will range from 30 to 60 µin. Ra [0.760 to 1.52 µm Ra].

5.1.2.2 *5D*—A shot-blasted finish for special applications.

5.2 *Double-Reduced Base Metal Finish*—Double-reduced electrolytic chromium-coated steel is produced only with ground-roll finishes.

5.2.1 *Ground Roll Finishes*:

5.2.1.1 *7C*—A smooth finish with grit lines. The surface roughness will range from 6 to 25 µin. Ra [0.152 to 0.635 µm Ra]. This is the standard finish for tin mill products.

6. Oiling

6.1 *Electrolytic Chromium-Coated Steel* is furnished with an extremely thin oil film on both surfaces to minimize abrasion in shearing, coiling or uncoiling, shipping, and handling. Acetyl tributylcitrate, butyl stearate or dioctyl (di(2-ethylhexyl) sebacate) is used. Surface active agents such as glycerol monooleate may be incorporated into the oil film. Oil film other than normal should be negotiated with the producer. The oil film is not a drawing lubricant.

6.1.1 Unless indicated by the purchaser, the special oiling treatment will be left to the discretion of the plate supplier.

6.1.2 Weight [mass] of oil film as applied by the plate supplier normally ranges from approximately 0.10 g/base box to 0.40 g/base box [0.50 to 200 g/SITA (5 to 20 mg/m^2)].

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Specific aim ranges required for particular end uses and handling practices may be furnished as agreed upon by the purchaser and supplier.

7. Workmanship and Quality Level Requirements

7.1 The production of coils does not afford the same opportunity for inspection, grading, and sorting as does the production of cut lengths. Accordingly, appropriate processing and quality-control procedures are required by the purchaser to obtain optimum utilization of the material. Cut lengths are line inspected visually and mechanically during production. Sheets having surface imperfections that will not interfere with their utilization are included.

8. General Requirements for Delivery

8.1 Product furnished under this specification shall conform to the applicable requirements of the latest edition of Specification A 623 [A 623M], unless otherwise provided herein.

9. Keywords

9.1 chromium coated; coated steel sheet; tin mill products

ANNEXES

(Mandatory Information)

A1. METHOD FOR DETERMINATION OF METALLIC-CHROMIUM COATING WEIGHT (MASS) ON CHROMIUM-PLATED TIN-FREE STEEL

A1.1 Scope

A1.1.1 This method is applicable to the determination of from 1 to 15 mg Cr/ft^2 [10.8 to 161 mg Cr/m^2] of surface in chromium-plated tinfree steel (TFS).

A1.2 Principle

A1.2.1 The oxide layer is removed chemically with a hot sodium hydroxide (NaOH) solution. The metallic chromium is then electrostripped at a current of from 10 to 20 mA with 1 *N* NaOH solution as the electrolyte. The stripping solution after acidification is then analyzed for chromium by oxidation with ammonium persulfate ($(NH_4)_2S_2O_8$) followed by photometric determination with diphenylcarbazide.

A1.3 Reagents

A1.3.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A1.3.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A1.3.3 *Ammonium Persulfate Solution (30 %)*—Dissolve 30 g of ammonium persulfate ($(NH_4)_2S_2O_8$) in 100 mL of water. This solution should be prepared fresh each day.

A1.3.4 *Chromium, Standard Solution* (1 mL = 0.02 mg Cr)—Dissolve 0.2828 g of dry, primary standard grade potassium dichromate ($K_2Cr_2O_7$) in distilled water and dilute to 500 mL in a volumetric flask. This solution contains 0.2 mg Cr/mL. Pipet a 100-mL aliquot of this stock solution to a 1-L volumetric flask and dilute to volume with distilled water. This dilute standard contains 0.02 mg Cr/mL.

A1.3.5 *Diphenylcarbazide Solution (0.24 %)*—Dissolve 0.25 g of diphenylcarbazide in 95 mL of acetone. Add 5 mL of glacial acetic acid, mix well, and store in a refrigerator until used.

A1.3.6 *Oxide Stripping Solution (Hot (90°C) 10 N NaOH Solution)*—Dissolve 200 g of NaOH pellets in 400 mL of water and dilute to 500 mL. Prepare as needed.

A1.3.7 *Silver Nitrate Solution (2 %)*—Dissolve 2 g of silver nitrate ($AgNO_3$) in 100 mL of water. Keep protected from light.

A1.3.8 *Sodium Hydroxide (Electrolyte) Solution (1 N)*—Dissolve 40 g of NaOH pellets in 1000 mL of distilled water.

A1.3.9 *Sulfuric-Phosphoric Acid Mixture*— Cautiously add 200 mL of sulfuric acid (H_2SO_4) to 500 mL of distilled water. Mix well and cool. Slowly add 300 mL of 85 % phosphoric acid (H_3PO_4), mix well, and store in a borosilicate bottle.

A1.4 Apparatus

A1.4.1 *Cell and Electrodes*—The cell is constructed of TFE-fluorocarbon with a TFE-fluorocarbon gasket to define the area to be stripped. A platinum rod is used as the cathode. This is the same cell as used for oxide determinations as shown in determination of chromium in the oxide.

A1.4.2 *Power Supply*—Philbrick Researches PR-30, 15-V regulated constant-current power supply or equivalent.

A1.4.3 *Milliammeter*, 0–50 mA full scale.

A1.4.4 *Voltmeter*, 0–2 V full scale.

A1.4.5 *Spectrophotometer*—Beckman Model B or equivalent.

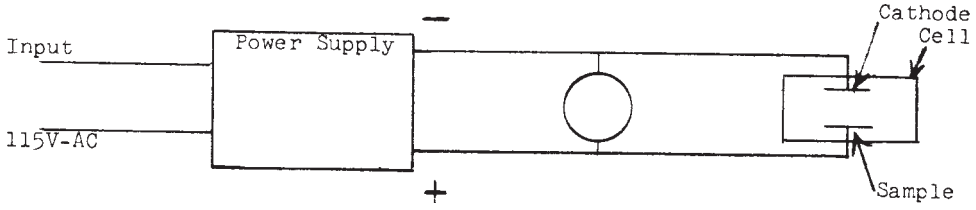
A1.4.6 *Vacuum Pump* for specimen hold down.

A1.5 Preparation of Chromium Calibration Curve

A1.5.1 Pipet 0 (blank), 5, 10, 15, and 20-mL aliquots of standard chromium solution (1 mL = 0.02 mg Cr) to 400-mL beakers. Add the same volume of electrolyte as is used in the stripping cell, and neutralize with H_2SO_4(1+3).

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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V = Voltmeter (0–2 V F.S.)
Cell = as described in method for chromium oxide determination

FIG. A1.1 Circuit Diagram for Stripping

A1.5.2 Add 25 mL of H_2SO_4-H_3PO_4 solution, 2 mL of $AgNO_3$ solution, and 5 mL of $(NH_4)_2S_3O_8$ solution. Heat the solutions to boiling and continue the boiling for about 10 min after the persulfate has been decomposed. Cool, and then transfer the solutions to 500-mL volumetric flasks. Add 5 mL of diphenylcarbazide solution and dilute to volume. Measure the absorbance after 2 min at 560 nm in a 1-cm cell using water as a reference. Correct for the absorbance of the reagent blank, and plot absorbance versus milligrams of chromium per 500 mL.

A1.6 Procedure

A1.6.1 Remove the surface oxides from the sample with 90°C 10 *N* NaOH solution for 10 min. Clamp the sample into the cell and introduce enough 1 *N* NaOH solution electrolyte to cover the sample completely. Connect the positive lead of the power supply to sample and the negative lead to the platinum cathode. Strip the metallic chromium coating with a current of 10 to 20 mA. The completion of the stripping is indicated by a pronounced sharp rise in cell voltage (=0.5 V). During the stripping operation the voltmeter will read several tenths of a volt. At the completion of stripping the voltage will rise rapidly from this value to a value approximately 0.5 V higher, a noticeable change in the 0 to 2-V instrument.

A1.6.2 After stripping, transfer the electrolyte solution to a 400-mL beaker, and with H_2SO_4(1+3) neutralize to pH 7 using a pH meter or pH paper to determine neutrality. Then add 25 mL of H_2SO_4-H_3PO_4 acid solution. Then add 2 mL of $AgNO_3$ solution, 5 mL of $(NH_4)_2S_2O_8$ solution, and heat to boiling. Continue boiling for about 10 min after the excess persulfate is decomposed as evidenced by lack of formation of small bubbles. Cool the solution to room temperature and transfer to a 500-mL volumetric flask. Add 5 mL of diphenylcarbazide solution, and dilute to volume. Measure the absorbance after 2 min in a spectrophotometer at 560 nm in a 1-cm cell using water as the reference. After correcting for the reagent blank, determine the milligrams of chromium from a calibration curve previously prepared with solutions containing known amounts of chromium. Calculate the coating weight [mass] in milligrams per square foot [per square metre].

A1.7 Calculations

A1.7.1 Calculate the coating weight [mass] in milligrams per square foot [per square metre] as follows:

$$\text{Cr, mg/ft}^2 \text{ of surface} = W \times 144/A$$

$$\left[\text{Cr, mg/m}^2 \text{ of surface} = W \times \frac{1}{A}\right]$$

where:

W = chromium obtained from calibration curve, milligrams, and
A = area stripped, in.^{2} [m^2].

A2. PREFERRED TEST METHOD FOR DETERMINATION OF CHROMIUM IN THE OXIDE ON THE SURFACE OF ELECTROLYTIC CHROMIUM COATED STEEL

A2.1 Scope

A2.1.1 This test method covers the determination of chromium present as oxide on the surface of electrolytic chromium coated steel.

A2.2 Summary Of Test Method

A2.2.1 The chromium oxide layer on the surface of the chromium coated steel is dissolved with 7.5 *N* sodium hydroxide (NaOH) solution, diluted to specific volume and aspirated into an air-acetylene flame (yellow-reducing). The absorbance at 357.9 nm is compared to the absorbances obtained from a series of standard chromium solutions and the chromium present as oxide is calculated in milligrams per square foot [per square metre].

A2.3 Reagents

A2.3.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A2.3.2 *Purity of Water*—Deionized or distilled water having a volume resistivity greater than 1 MΩcm at 25°C as determined by Method B of Test Methods D 1125.

A2.3.3 *Sodium Hydroxide Solution* (7.5*N*)—Dissolve 300 g of NaOH pellets in distilled water, cool, and then dilute to 1 L.

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A2.3.4 *Standard Solution, Chromium*—Dissolve 0.2828 µg of dry, primary standard grade potassium dichromate ($K_2Cr_2O_7$) in distilled water and dilute to 1000 mL in a volumetric flask. This solution contains 0.1 mg Cr/mL.

A2.3.5 *Standard Solution* (blank, 1.0, 2.0, and 5.0 µg Cr/mL levels)—Pipet into four 100 mL volumetric flasks, the following amounts of the chromium standard solution (A2.3.4). First flask, no solution. Second flask, 1.0 mL. Third flask, 2.0 mL. Fourth flask, 5.0 mL. Add 50 mL of 7.5 *N* NaOH solution to each flask. Dilute each flask to 100 mL with distilled water.

A2.4 Apparatus

A2.4.1 *Atomic Absorption Spectrometer*—Perkin Elmer 403 equipped with a standard Perkin-Elmer nebulizer-burner assembly with a 4-in. [10.2 cm] single slot head (or equivalent instrumentation).

A2.4.2 Hollow Cathode Discharge Source—Westinghouse, chromium, WL-2293A (or equivalent).

A2.4.3 *Cutter*, to blank 2.257 in. [5.73 cm] diameter discs or a size of known area greater than 4 in.^{2} [10.2 cm^2].

A2.5 Preparation of Chromium Calibration Curve

A2.5.1 Set the following settings on the atomic absorption spectrometer: lamp current, 15.0 mA; wave length, 357.9 nm; spectral slit, 0.7 nm; flame, air/acetylene.

A2.5.2 Ignite the air-acetylene flame (yellow-reducing) and allow the instrument to warm up for five minutes. While aspirating the 5.0 µg/mL standard solution (A2.3.5), optimize the flame composition and burner position for maximum absorbance (approximately 0.200 absorbance unit). Aspirate the other standard solutions (blank, 1.0, 2.0, and 5.0) and chart the four standard solution values. Plot absorbance versus µg/mL of chromium in solution.

A2.6 Use of Blank Disc or Sample Size

A2.6.1 Blank disc or sample size to be used. (If one side test is desired, spray one side with high-temperature clear Krylon lacquer and let dry. This prevents the sprayed side from being dissolved).

A2.6.2 Clean disc with chloroform or acetone.

A2.6.3 The uncoated side(s) are analyzed for chromium present as chromium oxide as follows. Place 50 mL of 7.5 *N* NaOH in a 600-mL beaker and heat to boiling. Remove from hot plate and allow to stand until boiling ceases. Add the sample disc to the beaker and strip for 5 min while swirling to maintain contact of solution over the sample. Prevent sample from lying flat on the bottom of the beaker by bending the disc slightly through the center. Remove sample after 5 min with a stirring bar retriever, rinse with distilled water, and allow the solution to cool to room temperature. Transfer the NaOH solution into a 100-mL volumetric flask, dilute to volume and mix thoroughly.

A2.7 Calculation

A2.7.1 Calculate the amount of chromium present in the oxide as follows:

$$C = A \times B \times \frac{144 \text{ in.}^2/\text{ft}^2}{D} \times \frac{1}{1000}$$

where:

A = concentration of Cr in µg/mL from chromium calibration curve,
B = dilution factor, millilitres (100),
C = chromium in the oxide per square foot, milligrams, and
D = area in in.^{2} of surface dissolved.

A3. METHOD FOR DETERMINATION OF CHROMIUM IN THE OXIDE ON THE SURFACE OF ELECTROLYTIC CHROMIUM-COATED STEEL

A3.1 Scope

A3.1.1 This method covers the determination of chromium present as oxide on the surface of electrolytic chromium coated steel.

A3.2 Summary of Method

A3.2.1 The oxide layer on the surface of the sheet is dissolved with a hot 10 *N* sodium hydroxide (NaOH) solution. After acidifying the solution, the chromium is oxidized with ammonium persulfate ($(NH_4)_2S_2O_8$) and then determined photometrically with diphenylcarbazide.

A3.3 Reagents

A3.3.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A3.3.2 *Purity of Water*—Deionized or distilled water having a volume resistivity greater than 1 MΩ cm at 25° C as determined by Method B of Test Methods D 1125.

A3.3.3 *Ammonium Persulfate Solution (30 %)*—Dissolve 30 g of ammonium persulfate ($(NH_4)_2S_2O_8$) in 100 mL of distilled water. This solution should be prepared freshly each day.

A3.3.4 *Chromium Standard Solution* (*1 mL = 0.01 mg Cr*)—Dissolve 0.2828 g of dry, primary standard grade potassium dichromate ($K_2Cr_2O_7$) in distilled water and dilute to 1000 mL in a volumetric flask. This solution contains 0.1 mg Cr/mL. Pipet a 100-mL aliquot of this stock solution to a 1-L volumetric flask, and dilute to volume with distilled water. This dilute standard solution contains 0.01 mg Cr/mL.

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A3.3.5 *Diphenylcarbazide Solution (0.25 %)*—Dissolve 0.25 g of diphenylcarbazide in 95 mL of acetone. Add 5 mL of glacial acetic acid, mix well, and store in a refrigerator until used.

A3.3.6 *Silver Nitrate Solution* (*2 %*)—Dissolve 2 g of silver nitrate ($AgNO_3$) in 100 mL of distilled water. Keep protected from light.

A3.3.7 *Sodium Hydroxide Solution* (*10 N*)—Dissolve 400 g of NaOH pellets in distilled water, cool, and then dilute to 1 L.

A3.3.8 *Sulfuric-Phosphoric Acid Mixture*—Cautiously add 200 mL of sulfuric acid (H_2SO_4) to 500 mL of distilled water. Mix well and cool. Slowly add 300 mL of 85 % phosphoric acid (H_3PO_4), mix well, and store in a borosilicate bottle.

A3.4 Apparatus

A3.4.1 *Spectrophotometer*—Bausch and Lomb Spectronic 20 or equivalent.

A3.4.2 *Sample Holder*, for holding sample while stripping oxide surface film from only one surface of the sample.

A3.4.3 *Vacuum Pump*, for attachment of sample holder to test surface.

A3.5 Preparation of Chromium Calibration Curve

A3.5.1 Pipet 0 (blank), 1.0, 2.0, 5.0, 8.0, and 10.0-mL aliquots of standard chromium solution (1 mL = 0.01 mg Cr) to 250-mL beakers and dilute to approximately 100 mL with distilled water. Add 5 mL of H_2SO_4-H_3PO_4 solution 2 mL of $AgNO_3$ solution, and 5 mL of $(NH_4)_2S_2O_8$ solution. Heat the solutions to boiling and continue the boiling for about 10 min to decompose the excess persulfate. Cool, and then transfer the solutions to 100-mL volumetric flasks. Add 3 mL of diphenylcarbazide solution and dilute to volume. Measure the absorbance after 2 min at 560 nm in a 1-cm cell using water as a

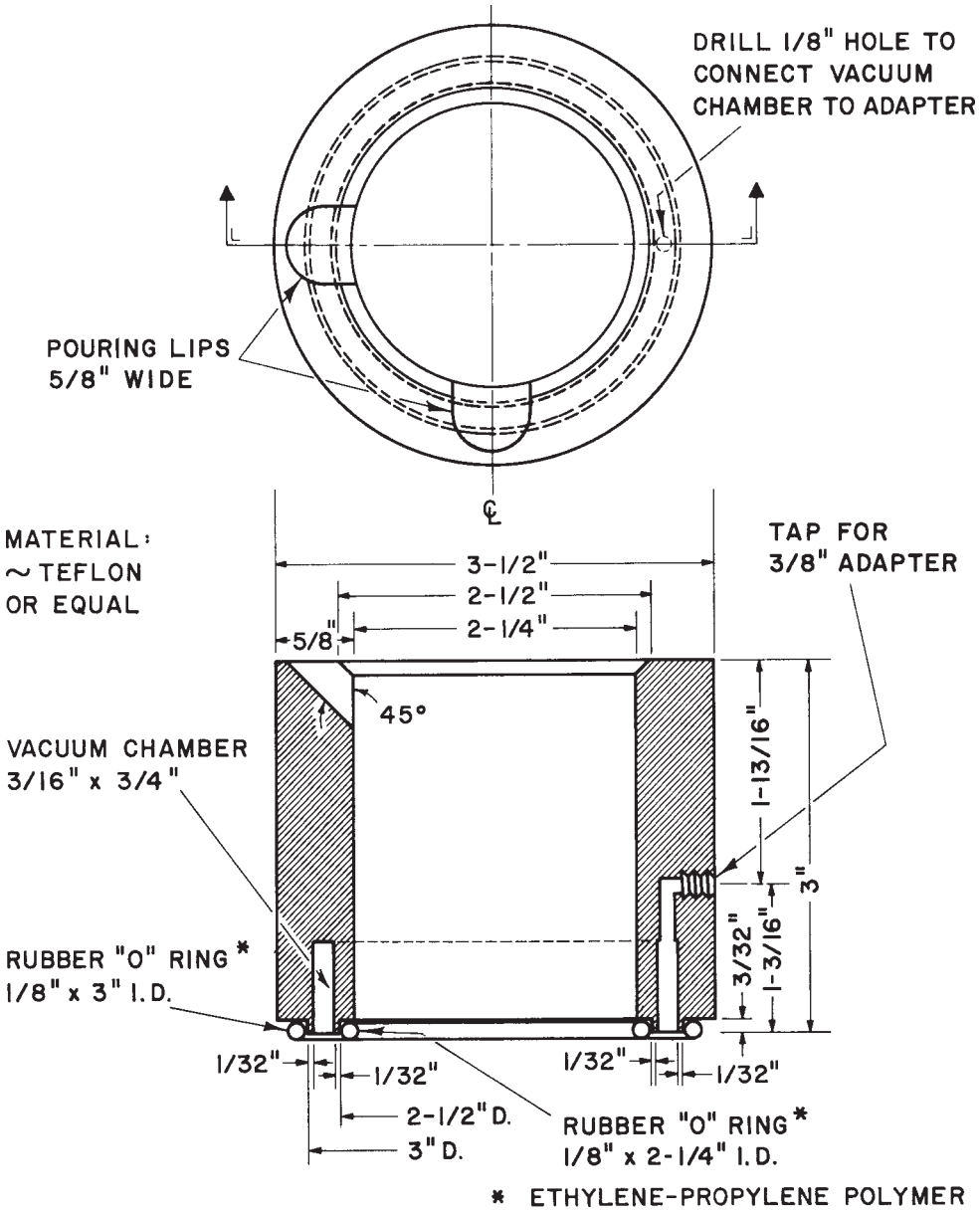


FIG. A3.1 Sample Holder

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reference. Correct for the absorbance of the blank, and plot absorbance versus milligrams of chromium per 100 mL.

A3.6 Procedure

A3.6.1 To remove the oxide surface film on each side of the sample separately, attach an appropriate size sample to the sample holder. The inner O-ring of the sample holder will define an area of 4.18-in.^{2}[10.6 cm] when the sample holder is attached to a vacuum pump. Place the sample, with cell attached, on a hot plate that is regulated to maintain the temperature of the stripping solution at 90°C. Add 15 mL of hot 10 *N* NaOH solution to the cell. After 10-min, transfer the stripping solution to a 250-mL beaker, rinsing the cell thoroughly. Cool, neutralize to litmus with (1+1) H_2SO_4, and add 5 mL of H_2SO_4-H_3PO_4 solution. Add 2 mL of $AgNO_3$ solution, and 5 mL of $(NH_4)_2S_2O_8$ solution. Heat the solution to boiling and continue boiling for about 10 min to decompose the excess persulfate. Cool the solution to room temperature and transfer to a 100-mL volumetric flask. Add 3 mL of diphenylcarbazide solution and dilute to volume. Measure the absorbance after 2 min at 560 nm in a 1-cm cell using distilled water as a reference. Correct for the absorbance of the reagent blank that is carried through the procedure and obtain the milligrams of chromium from the calibration curve.

A3.7 Calculation

A3.7.1 Calculate the coating weight in milligrams per square foot [square metre] as follows:

$$\text{Cr, mg/ft}^2 \text{ of surface} = W \times 144/A$$

$$\left[\text{Cr, mg/m}^2 \text{ of surface} = W \times \frac{1}{A}\right]$$

where:

W = chromium from the calibration curve, milligrams, and
A = area stripped, in.^{2} [m ^{2}].

NOTE A3.1—A 4-in.^{2} [25.8 cm^2] disk may be stripped in a 250-mL beaker when the analysis of each separate side is not required.

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