Designation: A623M – 22

Standard Specification for Tin Mill Products, General Requirements [Metric][1]

This standard is issued under the fixed designation A623M; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

INTRODUCTION

This specification is the metric counterpart of Specification A623. It is not intended to replace A623. Users of the standard should note several very significant differences in how the product is produced and marketed.

(1) The metric product does not carry the overrun associated with tin mill products produced to customary units. Metric tin mill products are produced to ordered size.

(2) The metric product is designated in units of 100 m^2 called a SITA (System International Tinplate Area), rather than in base boxes.

(3) The metric product is designated by thickness in millimetres rather than by basis weight.

(4) Coating weights are given in grams per square metre, not pounds per base box.

(5) Thickness tolerances are given in absolute figures instead of a ± percentage.

(6) Each package of metric tin mill products contains 100 sheets, not the 112 of customary unit packages.

All of the above significant differences, as well as others of lesser consequence, should be considered when switching from Specification A623 to Specification A623M.

1. Scope

1.1 This specification covers a group of common requirements, which unless otherwise specified in the purchase order or in an individual specification, shall apply to tin mill products.

1.2 In case of conflict in requirements, the requirements of the purchase order, the individual material specification, and this general specification shall prevail in the sequence named.

NOTE 1—A complete inch-pound companion to Specification A623M has been developed—Specification A623; therefore, no inch-pound equivalents are presented.

1.3 The following safety hazards caveat covers Annex A1 through Annex A8 of this specification: *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A370 Test Methods and Definitions for Mechanical Testing of Steel Products

A623 Specification for Tin Mill Products, General Requirements

A700 Guide for Packaging, Marking, and Loading Methods for Steel Products for Shipment

A987 Practice for Measuring Shape Characteristics of Tin Mill Products

E18 Test Methods for Rockwell Hardness of Metallic Materials

E112 Test Methods for Determining Average Grain Size

2.2 *Military Standards:*[3]

MIL-STD-129 Marking for Shipment and Storage

[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] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

MIL-STD-163 Steel Mill Products, Preparation for Marking and Storage

2.3 *Federal Standard:*[3]

Fed. Std. No. 123 Marking for Shipment (Civil Agencies)

3. Terminology

3.1 *Definitions:*

3.1.1 *black plate, n*—light-gage, low-carbon, cold-reduced steel intended for use in the untinned state or for the production of other tin mill products. It is supplied only in a dry or oiled condition.

3.1.2 *box annealing, n*—a process involving slow heating of coils to a subcritical temperature, holding, and cooling therefrom, to recrystallize the grain, and thus, relieve stresses produced during cold reduction. It is accomplished in a sealed container. By introducing and maintaining an inert or slightly reducing atmosphere during the cycle, a relatively bright surface is obtained.

3.1.3 *bright finish, n*—a surface that has a lustrous appearance.

3.1.4 *burr, n*—metal displaced beyond the plane of the surface by slitting or shearing (see 9.1.7 and 9.2.6).

3.1.5 *camber, n*—the greatest deviation of a coil edge from a straight line; the measurement is taken on the concave side and is the perpendicular distance from a straight line to the point of maximum deviation (see 9.1.9 and 9.2.7).

3.1.6 *chemical treatment, electrolytic tin plate, n*—a passivating chemical treatment applied to the surface of electrolytic tin plate to stabilize the plate surface characteristics compatible with a specified end use (see Annex A7).

3.1.7 *chemically treated steel, n*—light-gage, low-carbon, cold-reduced steel that has a passivating or chemical treatment applied to the surface to provide rust resistance or retard underfilm corrosion, or both.

3.1.8 *cold reduction, n*—the process of reducing the thickness of the strip cold, generally accomplished by one rolling through a series of four-high mills arranged in tandem.

3.1.9 *continuous annealing, n*—a process consisting of passing the cold-reduced strip continuously and in a single thickness through a series of vertical passes within a furnace consisting of heating, soaking, and cooling zones to recrystallize the grain and thus relieve stresses produced during cold reduction. An inert or slightly reducing atmosphere is maintained in the furnace to obtain a relatively bright strip.

3.1.10 *differentially coated tin plate, n*—electrolytic tin plate with a different weight of tin coating on each surface.

3.1.11 *double-reduced plate, n*—plate given a second major cold reduction following annealing. Some double-reduced products are produced to achieve a minimum level of ductility (% elongation) in the material. These products carry the designation of High Elongation Double-Reduced, or HEDR.

3.1.12 *electrolytic chromium-coated steel, n*—light-gage, low-carbon, cold-reduced steel on which chromium and chromium oxides have been electrodeposited.

3.1.13 *electrolytic tin plate, n*—light-gage, low-carbon, cold-reduced steel on which tin has been electrodeposited by an acid or alkaline process.

3.1.13.1 *J Plate, n*—electrolytic tin plate, 5.6/2.8 g/m^2 or heavier tin coating, with improved corrosion performance for some galvanic detinning food products as specified in 3.1.13.2 and as measured by the Special Property Tests for Pickle Lag (PL) (see Annex A2), Iron Solution Values (ISV) (see Annex A4), Tin Crystal Size (TCS) (see Annex A3). The alloy layer is normally light in color, characteristic of the acid tinning process.

3.1.13.2 *K Plate, n*—electrolytic tin plate, 5.6/2.8 g/m^2 or heavier tin coating, with improved corrosion performance for some galvanic detinning food products as specified in the following table and as measured by the Special Property Tests for Pickle Lag (PL) (see Annex A2), Iron Solution Value (ISV) (see Annex A4), Tin Crystal Size (TCS) (see Annex A3), Alloy Tin Couple (ATC) (see Annex A5) and Aerated Media Polarization Test (AMP) (see Annex A8).

	Special Properties Aims
Pickle Lag[A]	10 s max
Iron Solution Value	20 µg iron max
Tin Crystal Size	ASTM No. 9 or larger
Alloy Tin Couple[B]	0.12 µA/cm^2 max

[A] The Pickle Lag test is not necessary if the product is processed using an anneal atmosphere gas of HNX or H_2.

[B] Good mill practice has demonstrated the ability to average 0.05 µA/cm^2 or less over an extended period of production.

3.1.13.3 *Discussion*—The production of J Plate and K Plate require special processing and testing. In order to receive J Plate or K Plate, this requirement must be specified on the order.

3.1.14 *length dimension, n*—the longer dimension of a cut size (see 9.2.9).

3.1.15 *lot, n*—each 20 000 sheets or part thereof or the equivalent in coils, of an item in a specific shipment having the same order specifications.

3.1.16 *matte finish, n*—a surface that has an unmelted tin coating, generally on a shot-blast finish (SBF) base steel.

3.1.17 *mechanical designation, n*—an arbitrary number to designate Rockwell hardness and ultimate tensile strength characteristics for double-reduced plate (see 8.2).

3.1.18 *oiling, n*—a lubricant film applied to both surfaces of the plate.

3.1.19 *package, n*—a quantity of 100 sheets.

3.1.20 *passivating treatment, n*—a surface chemical treatment (see 3.1.6).

3.1.21 *Rockwell hardness test, n*—a test for determining hardness (see Annex A1).

3.1.22 *rolling width, n*—the dimension of the sheet perpendicular to the rolling direction.

3.1.23 *single-reduced plate, n*—plate produced with one major cold reduction.

3.1.24 *SITA, n*—100 square metres.

Formula for cut lengths:

TABLE 1 Chemical Requirements for Tin Mill Products

Element	Cast Composition, max %		
	Type D	Type L	Type MR
Carbon	0.12	0.13	0.13
Manganese	0.60	0.60	0.60
Phosphorous	0.020	0.015	0.020
Sulfur	0.03	0.03	0.03
Silicon[A,B]	0.020	0.020	0.020
Copper	0.20	0.06	0.20
Nickel	0.15	0.04	0.15
Chromium	0.10	0.06	0.10
Molybdenum	0.05	0.05	0.05
Aluminum[C]	0.20	0.10	0.20
Other elements, each	0.02	0.02	0.02

[A] When steel produced by the silicon killed method is ordered, the silicon maximum may be increased to 0.080 %.
[B] When strand cast steel produced by the aluminum killed method is ordered or furnished, the silicon maximum may be increased to 0.030 % when approved by the purchaser.
[C] Types L and MR may be supplied as non-killed or killed, which would respectively be produced without and with aluminum additions. Minimum aluminum level for Type D is usually 0.02 %.

$$\text{SITA} = \frac{\text{width (mm)}}{1000} \times \frac{\text{length (mm)}}{1000} \times \text{number of packages}$$

Formula for coils:

$$\text{SITA} = \frac{\frac{\text{width (mm)}}{1000} \times \text{length (m)}}{100\text{m}^2}$$

3.1.25 *steel Type D, n*—base-metal steel aluminum killed, sometimes required to minimize severe fluting and stretcher-strain hazards or for severe drawing applications (see Table 1).

3.1.26 *steel Type L, n*—base-metal steel, low in metalloids and residual elements, sometimes used for improved internal corrosion resistance for certain food-product containers (see Table 1).

3.1.27 *steel Type MR, n*—base-metal steel, similar in metalloid content to Type L but less restrictive in residual elements, commonly used for most tin mill products (see Table 1).

3.1.28 *surface appearance, n*—visual characteristics determined primarily by the steel surface finish; for electrolytic tin plate, the appearance is also influenced by the weight of coating and by melting or not melting the tin coating.

3.1.29 *surface finishes, n*—steel surface finishes for tin mill products imparted by the finishing-mill work rolls; these may be either ground, blasted, or etched roll finishes.

3.1.30 *temper designation, n*—an arbitrary number to designate a Rockwell hardness range for single-reduced products, which indicates the forming properties of the plate (see Section 8 and Table 2 and Table 3).

3.1.31 *temper mill, n*—a mill for rolling base metal steel after annealing to obtain proper temper, flatness, and surface finish; it may consist of one stand or two stands arranged in tandem.

3.1.32 *tin coating weight, n*—the weight of tin applied to the steel surface, usually stated as grams per square metre distributed evenly over both surfaces. The coating is usually referred to by designation numbers, referring separately to the nominal tin weight on each surface, but omitting the units. Thus, 2.8/2.8 designates tin plate with a coating of 2.8 g/m^2 on each of the two surfaces. For differential coatings, the same system is applied. Thus, 1.1/2.2 has a coating of 1.1 g/m^2 on one surface and 2.2 g/m^2 on the other surface.

3.1.33 *width dimension, n*—the shorter dimension of a cut size (see 9.2.9).

TABLE 2 Temper Designations and Hardness Values Single Reduces Tin Mill Products—Box Annealed

NOTE 1—Thinner plate (0.21 mm ordered thickness and thinner) is normally tested using the Rockwell 15TS scale and the results converted to the Rockwell 30TS scale (see Annex A1 and Table A1.1).

Temper Designation	Rockwell Hardness Values All Thickness HR30TS[A]		Characteristics and Typical End Uses
	Nominal	Rang[B] e	
T-1 (T49)	49	45-53	soft for drawing parts such as nozzles, spouts, and oil filter shells
T-2 (T53)	53	49-57	moderately soft for drawing shallow parts such as rings, plugs, and pie pans
T-3 (T57)	57	53-61	Fairly stiff for parts such as can ends and bodies, closures, and crown caps
T-4 (T61)	61	57-65	Increased stiffness for can ends and bodies, crown caps, and large closures

[A] These ranges are based on the use of the diamond spot anvil and a 1.588 mm hardened steel ball indenter.
[B] The hardness ranges are requirements unless otherwise agreed upon between producer and user.
Test conditions:
1. For referee purposes, samples of blackplate, unreflowed ETP, and ECCS shall be aged prior to testing by holding at 400 °F for 10 min.
2. For referee purposes, the hardness test area on material produced with SBF or equivalent rolls shall be sanded smooth on both surfaces.
3. To avoid incorrect results due to the cantilever effect, samples shall have an area no larger than 4 in.^{2} and the point of testing shall be no more than ½ in. off the center of the samples.

4. Base Metal

4.1 The steel shall be made by the open-hearth, electric furnace, or basic-oxygen process.

5. Chemical Composition

5.1 The steel shall conform to the chemical composition requirements as prescribed in Table 1 except as otherwise agreed upon between the manufacturer and the purchaser.

6. Cast or Heat Analysis

6.1 For Type D, MR, and L an analysis of each heat of steel shall be made by the supplier to determine the percentage of carbon, manganese, phosphorus, sulfur, silicon, and residual elements shown in Table 1. Other elements, unless agreed upon between the manufacturer and the purchaser, individually shall not exceed 0.02 %, maximum and while not necessarily analyzed are dependent on the suppliers' practices and controls.

TABLE 3 Temper Designations and Hardness Values Single-Reduced Tin Mill Products—Continuously Annealed

NOTE 1—Thinner plate (0.21 mm ordered thickness and thinner) is normally tested using the Rockwell 15TS and the results converted to the Rockwell 30TS scale (see Annex A1 and Table A1.1).

Temper Designation	Rockwell Hardness Value All Thicknesses HR30TS[A]		Characteristics and Typical End Uses
	Nominal	Range[B]	
T-1 (T49)	49	45–53	soft for drawing parts such as nozzles, spouts, and oil filter shells
T-2 (T53)	53	49–57	moderately soft for drawing shallow parts such as rings, plugs, and pie pans
T-3 (T57)	57	53-61	moderate stiffness for parts such as can ends and bodies, drawn and ironed can bodies closures, and crown caps
T-4 (T61)	61	57-65	increased stiffness for can ends, drawn (and ironed) can bodies, and large closure
T-5 (T65)	65	61-69	moderately high stiffness for can ends and bodies

[A] These ranges are based on the use of the diamond spot anvil and a 1.588 mm hardened steel ball indenter.
[B] The hardness ranges are requirements unless otherwise agreed upon between producer and user.
Test conditions:
1. For referee purposes, samples of blackplate, unreflowed ETP, and ECCS shall be aged prior to testing by holding at 400 °F for 10 min.
2. For referee purposes, the hardness test area on material produced with SBF or equivalent rolls shall be sanded smooth on both surfaces.
3. To avoid incorrect results due to the cantilever effect, samples shall have an area no larger than 4 in.^{2} and the point of testing shall be no more than ½ in. off the center of the samples.

7. Product Analysis

7.1 Rimmed or capped steels are characterized by a lack of uniformity in their chemical composition, and for this reason, product analysis is not technologically appropriate unless misapplication is clearly indicated.

8. Mechanical Requirements

8.1 *Single-Reduced Tin Mill Products, Temper*—The term *temper*, when applied to single-reduced tin mill products, summarizes a combination of interrelated mechanical properties. No single mechanical test can measure all the various factors that contribute to the fabrication characteristics of the material. The Rockwell 30TS hardness value is a quick test, which serves as a guide to the properties of the plate. This test forms the basis for a system of temper designations as shown in Table 2 and Table 3. A given temper shall have hardness values meeting the limits shown. The mechanical properties of continuously annealed plate and batch annealed plate of the same Rockwell 30TS temper designation are not identical. It is important to keep in mind, that the Rockwell 30TS test does not measure all the various factors, which contribute to the fabrication characteristics of the plate.

8.2 *Double-Reduced Tin Mill Products, Mechanical Characteristics*—No test or group of tests have been developed that adequately predict the fabricating performance of double-reduced tin mill products. Some double-reduced products are produced to achieve a minimum level of ductility (% elongation) in the material. These products carry the designation High Elongation Double-Reduced, or HEDR. The required minimum elongation for HEDR products will be at the discretion of the producer and the user. No targets for HEDR products will be referenced aside from the UTS and hardness values in Table 4. Designations for mechanical properties showing typical applications are arranged in generally ascending level of strength as shown in Table 4.

8.3 Rockwell testing shall be in accordance with the latest revision of Test Methods and Definitions A370 (see Annex A1) and Test Methods E18.

9. Permissible Variation in Dimensions

9.1 *Dimensional Characteristics, Coils:*

9.1.1 *Thickness, Method for Determination*—When the purchaser wishes to make tests to ascertain compliance with the requirements of this specification for thickness of an item in a specific shipment of tin mill products in coils having the same order specification, the following procedure shall be used: Random and representative measurements using a hand micrometer must be made throughout the coil length. Measurements may be made at any location across the coil width except 10 mm from the mill-trimmed edge. The hand micrometers are assumed to be accurate to ±0.003 mm. No measurements are to be made within 1.0 m of a weld.

9.1.2 *Thickness Tolerances* shall conform to those prescribed in Table 5 (also see Table 6).

9.1.3 *Transverse Thickness Profile* is the change in sheet thickness from strip center to edge at right angles to the rolling direction. Thickness measured near the edge is normally less than the center thickness. The gauge measured 6 mm in from

TABLE 4 Mechanical Designations Double-Reduced Tin Mill Products

NOTE 1—Thinner plate (0.21 mm ordered thickness and thinner) is normally tested using Rockwell 15TS scale and the results converted to the Rockwell 30TS scale (see Annex A1 and Table A1.1).

Designation[B]	Nominal Longitudinal (L) Ultimate Tensile Strength, MPa	Nominal Rockwell Hardness HR30-TS[A]	Examples of Usage
DR-7.5	520	71	can bodies
DR-8	550	72	can bodies and ends
DR-8.5	580	73	can bodies and ends
DR-9	620	75	can bodies and ends
DR-9.5	660	76	can ends

[A] These values are based on the use of the diamond spot anvil and a 1.588 mm steel ball indenter. Testing will be in accordance with Test Methods and Definitions A370. Rockwell values are too varied to permit establishment of ranges. For details see *AISI Contributions to the Metallurgy of Steel*, "Survey of Mechanical Properties of Double Reduced Tin Plate," January 1966.
[B] Double-reduced products requiring a minimum % elongation or ductility will be designated as HEDR (for example, HEDR-8 temper). The specified amount of minimum elongation for a specific temper designation shall be agreed upon between the producer and the user.

TABLE 5 Thickness Tolerances

NOTE 1—When weld-free coils are specified, this does not afford the supplier the opportunity to discard off-gage product, and for that reason the above thickness tolerances are not applicable.

Lot Size, Mg (metric tons)	Tolerance
0 to 5.5	95 % of the product of the coils shall be within the tolerances slated in Table 6.
Over 5.5 to 13.6	97.5 % of the product of the coils shall be within the tolerances stated in Table 6.
Over 13.6 to 68.0	99.0 % of the product of the coils shall be within the tolerances stated in Table 6.
Over 68.0	99.5 % of the product of the coils shall be within the tolerances stated in Table 6.

the mill trimmed edge shall be no more than either 13 % below the ordered thickness or 10 % less than the center thickness of the individual sheet being measured. Common components of transverse thickness profile are crown and feather edge.

9.1.4 *Crown* is the difference in strip thickness from the center of roll width and the location 25 mm in from the mill-trimmed edge.

9.1.5 *Feather Edge* is the maximum difference in thickness across the strip width between points measured at 6 mm and 25 mm from both mill-trimmed edges. The thickness 6 mm from an edge is usually less than the thickness measured 25 mm or more from the same edge.

9.1.6 *Width*—Coils are trimmed to ordered width. The slit dimension shall not vary by more than −0, +3 mm.

9.1.7 *Burr*—A maximum of 0.05 mm is permissible. Burr may be estimated by using a micrometer with a flat anvil and spindle and measuring the difference between strip thickness adjacent to the edge and strip thickness at the edge, which includes the displaced metal. Care must be taken during that measurement to avoid deforming the displaced metal.

9.1.8 *Coil Length*—Variation between the measured length by the purchaser versus the supplier's billed length shall not exceed the limits prescribed in Table 7.

TABLE 6 Ordered Thickness and Thickness Tolerances

NOTE 1—Thickness tolerances are +5 % and -8 % from the ordered thickness

Ordered Thickness, mm	Thickness Tolerance, Over, mm	Thickness Tolerance, Under, mm
0.140	0.007	0.011
0.150	0.008	0.012
0.160	0.008	0.013
0.170	0.008	0.014
0.180	0.009	0.014
0.190	0.010	0.015
0.200	0.010	0.016
0.210	0.010	0.017
0.220	0.011	0.018
0.230	0.012	0.018
0.240	0.012	0.019
0.250	0.012	0.020
0.260	0.013	0.021
0.270	0.014	0.022
0.280	0.014	0.022
0.290	0.014	0.023
0.300	0.015	0.024
0.310	0.016	0.025
0.320	0.016	0.026
0.330	0.016	0.026
0.340	0.017	0.027
0.350	0.018	0.028
0.360	0.018	0.029
0.370	0.018	0.030
0.380	0.019	0.030

TABLE 7 Coil Length Variation

No. of Coils	Variation, ±, %
1	3
100	0.1

9.1.8.1 Since it is a common practice for each consumer's shearing operation to keep a running measurement of their supplier's coil shipments, any length variation in small lots (1 to 5 coils) for a given period will automatically be included in this summary. Before concluding there is a length variation in these small lots, the total length received from the supplier, regardless of thickness, over periods of one month or one quarter, or both should be checked.

9.1.9 *Camber* is limited to a maximum of 6 mm in 6 m or fraction thereof of length, in accordance with the latest version of measuring methods and definitions in Test Method A987.

9.1.10 *Inside Coil Diameters*—The standard inside diameter produced is approximately 410 mm.

9.2 *Dimensional Characteristics, Cut Sizes:*

9.2.1 *Thickness, Method for Determination*—Random measurements must be made at least 25 mm from the slit edge of the sheet using a hand micrometer. The hand micrometers are assumed to be accurate to ±0.003 mm.

9.2.2 *Thickness Tolerances*—Tin mill products in cut sizes are produced within thickness tolerances of +5 %, -8 % of the ordered thickness, see (Table 6). Any sheets not meeting this requirement are subject to rejection.

9.2.3 *Transverse Thickness Profile* is the change in sheet thickness from strip center to edge at right angles to the rolling direction. Thickness measured near the edge is normally less than the center thickness. The gauge measured 6 mm in from

the mill trimmed edge shall be no more than either 13 % below the ordered thickness or 10 % less than the center thickness of the individual sheet being measured. Common components of transverse thickness profile are crown and feather edge.

9.2.4 *Crown* is the difference in strip thickness from the center of roll width and the location 25 mm in from the mill-trimmed edge.

9.2.5 *Feather Edge* is the maximum difference in thickness across the strip width between points measured at 6 mm and 25 mm from both mill-trimmed edges. The thickness 6 mm from an edge is usually less than the thickness measured 25 mm or more from the same edge.

9.2.6 *Burr*—A maximum of 0.05 mm is permissible. Burr may be estimated by using a micrometer with a flat anvil and spindle and measuring the difference between strip thickness adjacent to the edge and strip thickness at the edge, which includes the displaced metal. Care must be taken during that measurement to avoid deforming the displaced metal.

9.2.7 *Camber*—The maximum permissible deviation is 1.3 mm for each 1 m of length or fraction thereof, in accordance with the latest version of measuring methods and definitions in Test Method A987.

9.2.8 *Out-of-Square* is the deviation of an end edge from a straight line, which is placed at a right angle to the side of the plate, touching one corner and extending to the opposite side. The amount of deviation is customarily limited to 1.5 mm for any edge measurement, except that a multiple-package lift may contain a maximum of four sheets with a deviation up to 3 mm.

9.2.9 *Shearing Practice*—Tin mill products are generally ordered to even-numbered millimetres and sheared to ordered size. The greater dimension is considered length. The slit dimension shall not vary by more than –0, +3 mm and the drum cut dimension shall not vary by more than –0, +6 mm.

10. Special Requirements

10.1 *Welds*—Coils may contain lap or mesh welds, the locations of which are marked. A hole may be punched adjacent to the weld for automatic rejection of the weld during shearing. The leading ends of lap welds shall not exceed 25 mm.

10.2 *Cores*—If coil centers must be supported to minimize damage, this requirement should be so stated on the order as a special requirement.

11. Sheet Count—Cut Sizes

11.1 Small variations in sheet count of a multiple-package lift should average out to at least the proper exact count in quantities of 450 packages or more.

12. Retest Procedure

12.1 In the event the material fails to meet the specified requirements, two further series of samples are to be selected by the purchaser in accordance with the applicable procedures. Both retests must meet the specification limits to qualify as meeting the requirements.

13. Conditions of Manufacture

13.1 The purchaser should be informed of any alterations in the method of manufacture, which will significantly affect the properties of the purchased product. Similarly, the purchaser should inform the manufacturer of modifications in their fabrication methods, which will significantly affect the way in which the purchased product is used.

14. Inspection

14.1 The inspector representing the purchaser shall have entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works that concern the manufacture of the material ordered. The supplier shall afford the inspector all reasonable facilities to satisfy him that the material is being furnished in accordance with this specification. Unless otherwise specified, all inspection and tests shall be made prior to shipment at the supplier's works and such inspection or sampling shall be made in conjunction with and to the extent of the manufacturer's regular inspection operations.

15. Rejection

15.1 Material that shows excessive number of injurious imperfections subsequent to its acceptance at the manufacturer's works, except as noted in the basis of purchase of the applicable specification, shall be rejected and the supplier notified.

16. Packaging

16.1 Unless otherwise specified, the tinplate shall be packaged and loaded in accordance with Practices A700.

16.2 When specified in the contract or order, and for direct procurement by or direct shipment to the government, when Level A is specified, preservation, packaging, and packing shall be in accordance with the Level A requirements of MIL-STD-163.

16.3 The standard method of shipping coils is with the eye of the coil vertical.

17. Marking

17.1 As a minimum requirement, the material shall be identified by having the manufacturer's name, ASTM designation, weight, purchaser's order number, and material identification legibly stenciled on top of each lift or shown on a tag attached to each coil or shipping unit.

17.2 When specified in the contract or order, and for direct procurement by or direct shipment to the government, marking for shipment, in addition to requirements specified in the contract or order, shall be in accordance with MIL-STD-129 for military agencies and in accordance with Federal Std. No. 123 for civil agencies.

18. Keywords

18.1 strength; tensile; tin mill products

ANNEXES

(Mandatory Information)

A1. ROCKWELL HARDNESS TESTING OF TIN MILL PRODUCTS

A1.1 Scope

A1.1.1 This annex covers the application to tin mill products of Rockwell superficial hardness tests using the 15TS and 30TS scales. Tests shall be made in accordance with the methods outlined in Test Methods E18 and Test Methods and Definitions A370 with the exceptions given in the following sections.

A1.2 Anvil

A1.2.1 All tests shall be made using the diamond spot anvil and a 1.588 mm hardened steel ball indenter.

A1.3 Specimens

A1.3.1 *Thickness*—The recommendations given in Table 12 of Test Methods E18 shall not apply to tests on tin mill products. The Rockwell superficial scale to be used shall be determined from the nominal thickness of the material as given in the following table:

Nominal Sheet Thickness, mm	Rockwell Superficial Scale	Major Load, kgf
0.212 and less	15TS	15
0.547–0.213	30TS	30

A1.3.2 *Surface Finish*—The surface of the specimen in contact with the diamond spot anvil shall be flat, smooth, and free from dirt or surface irregularities. When necessary, both specimen surfaces shall be sanded smooth to remove surface irregularities that may affect the test results. Sanding debris shall be removed from the sample before testing. Unless otherwise agreed upon, the tin coating shall not be removed from the surface on which the indentation is made.

A1.4 Reports

A1.4.1 *Number of Tests*—The Rockwell scale value to be reported shall be the average of at least three impressions.

TABLE A1.1 Conversion Table (Approximation) Rockwell Hardness Testing

HR30TS	HR15TS	HR30TS	HR15TS
82.0	93.0	65.0	84.0
81.5	92.5	64.0	. . .
81.0	. . .	63.5	83.5
80.5	92.0	62.5	83.0
80.0	. . .	62.0	. . .
79.0	91.5	61.5	82.5
78.5	. . .	60.5	82.0
78.0	91.0	60.0	. . .
77.5	90.5	59.5	81.5
77.0	. . .	58.5	81.0
76.0	90.0	58.0	. . .
75.5	89.5	57.0	80.5
75.0	. . .	56.5	. . .
74.5	89.0	56.0	80.0
74.0	88.5	55.0	79.5
73.5	. . .	54.5	. . .
73.0	88.0	54.0	79.0
72.0	87.5	53.0	78.5
71.5	. . .	52.5	. . .
71.0	87.0	51.5	78.0
70.0	86.5	51.0	77.5
69.5	. . .	50.5	. . .
69.0	86.0	49.5	77.0
68.0	85.5	49.0	76.5
67.5	. . .	48.5	. . .
67.0	85.0	47.5	76.0
66.0	. . .	47.0	75.5
65.5	84.5	46.0	. . .

A1.4.2 *Conversion*—Hardness tests made on the 15TS scale may be converted to the 30TS scale by the use of Table A1.1. It is recognized that such conversions are for convenience in reporting and that conversion, particularly from tests on thin and soft materials, is not an accurate process.

A2. METHOD FOR DETERMINATION OF PICKLE LAG ON STEEL FOR ELECTROLYTIC TIN PLATE

INTRODUCTION

It is not intended that variations in apparatus, sample preparation, or procedures from those described in this standard method be precluded. Suppliers or consumers may employ such variations for control purposes provided test results agree with results obtained by the standard method.

A2.1 Scope

A2.1.1 The rate of pickling test,[4] also called the pickle lag test, is one of four special property tests used to measure certain characteristics of electrolytic tin plate, which affect internal corrosion resistance. The test is applicable to nominal tin coating and heavier electrolytic tin plate (For K-plate, see 3.1.13.2 and J-plate, see 3.1.13.1). It is not applicable to 2.8/2.8 and lighter electrolytic tin plate.

A2.2 Summary of Method

A2.2.1 The time lag for a piece of steel to attain constant dissolution rate in acid under controlled conditions is determined. The change in pressure in a closed system caused by hydrogen evolution from the steel is continuously plotted on a chart through use of an electro-mechanical linkage and mercury manometer.

A2.3 Apparatus

A2.3.1 *Reaction Vessel,*[5,6] consisting of a specially modified 125 mL Erlenmeyer flask. The flask shall have a 10 mm bore stopcock, glass sealed to the mouth and a small-diameter glass tube side arm sealed in the side just below the mouth of the original flask. The bottom of the flask shall be rounded out. A mercury switch shall be attached to the stop-cock plug with a metal band.

A2.3.2 *Constant-Temperature Water Bath,* large enough to accommodate the reaction vessel and maintain a temperature of 90 ± 0.5 °C.

A2.3.3 *Recording Mercury Manometer,*[7,6] to measure the rate of increase in pressure in the vessel generated by hydrogen. Initial setup of the recorder is described in Section 9.

A2.3.4 A381 by 3.17 mm magnetized steel rod for removal of test specimen. (A one-hole rubber stopper may be positioned near the upper end to prevent the bottom of the rod from striking the bottom of the reaction flask.)

A2.3.5 *Coordinate Paper,* 101 by 279 mm, with either 10 or 20 gradations, each 25.4 mm.

A2.4 Reagents and Materials

A2.4.1 *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.[8] 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.

A2.4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A2.4.3 *For Rate of Pickling Test:*

A2.4.3.1 *Hydrochloric Acid* (HCl), (6 *N*).

A2.4.4 *For Sample Preparation:*

A2.4.4.1 *Acetone.*

A2.4.4.2 *Antimony Trichloride Solution* (120 g/L)—Dissolve 120 g of antimony trichloride ($SbCl_3$) in 1 L of concentrated HCl.

A2.4.4.3 *Sodium Carbonate Solution* (Na_2CO_3) (0.5 %).

A2.4.4.4 *Sodium Hydroxide Solution* (NaOH) (10 %).

A2.4.4.5 *Sodium Peroxide* (Na_2O_2), *granulated.*

A2.4.5 *For Water Bath:*

A2.4.5.1 *Paraffin Oil.*

A2.5 Test Specimen Preparation

A2.5.1 *Test Specimen*—A piece of steel 8 by 65 mm with the long dimension perpendicular to the rolling direction of the steel.

A2.5.1.1 Cut a piece of metal 8 by 100 mm or longer. The added length above the 65 mm serves as a handle during preparation.

A2.5.1.2 Remove surface oil and grease by dipping the specimen in acetone and wiping with a cloth or paper towel.

A2.5.1.3 Cathodically clean the specimen in 0.5 % solution of Na_2CO_3, rinse in water, and dry.

A2.5.1.4 Detin the specimen by immersing in $SbCl_3$-HCl solution at room temperature. Allow the specimen to remain in solution 10 to 20 s after bubbling ceases.

A2.5.1.5 Remove the specimen, rinse in tap water, and wipe surface clean of antimony. (A wet cellulose sponge with a little non-ionic detergent has been found effective.)

A2.5.1.6 Immerse specimen in 10 % NaOH solution held at 90 °C for approximately 1 min. During this time add granulated Na_2O_2 slowly to keep solution bubbling freely. This treatment removes the last traces of antimony and any iron-tin alloy not removed during detinning. More than one specimen may be treated at one time. A stainless steel beaker with specimens contacting the beaker appears to facilitate removal of the antimony and iron-tin alloy.

A2.5.1.7 Rinse specimen successively in tap water, distilled or deionized water and acetone. Alternatively rinse specimen in tap water and wipe dry with a clean towel.

A2.5.1.8 Trim specimen to 8 by 65 mm.

A2.5.1.9 Handle the specimen with forceps as touching with the fingers may produce erratic test results.

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[4] Willey, A. R., Krickl, J. L., and Hartwell, R. R., "Steel Surface Properties Affect Internal Corrosion Performance of Tin Plate Containers," *Corrosion*, Vol 12, No. 9, 1956, p. 433.

[5] The sole source of supply of the apparatus known to the committee at this time is Wilkens-Anderson Co., 5626 W. Division St., Chicago, IL 60651. Such apparatus or its equivalent has been found satisfactory.

[6] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[7] The sole source of supply of the apparatus known to the committee at this time is Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, PA 19154. Such apparatus or its equivalent has been found satisfactory.

[8] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

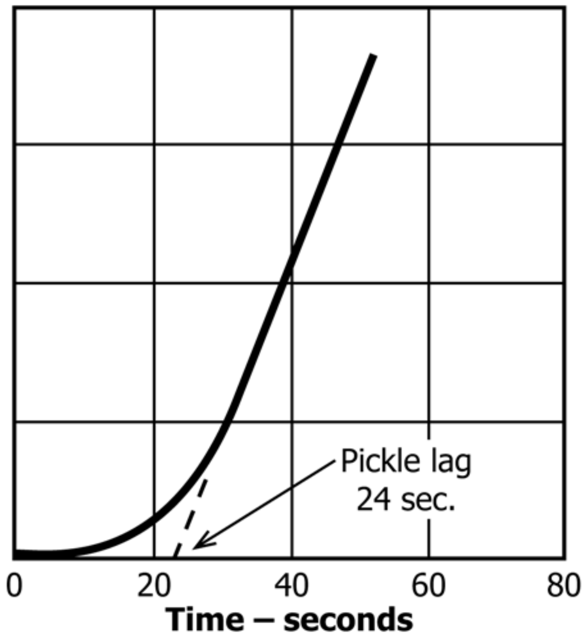

FIG. A2.1 Pickle Lag

A2.6 Procedure

A2.6.1 Bring the constant-temperature water bath to 90 ± 0.5 °C, making certain the 6 *N* HCl in the reaction vessel has also reached 90 °C, if it has been freshly transferred.

A2.6.2 Start recorder and place the pen against the graph paper near the bottom.

A2.6.3 Drop the specimen into the reaction vessel and immediately close the stopcock. The mercury switch will start the recorder drum turning. The pressure generated by reaction of the acid on the specimen will cause the pen to rise.

A2.6.4 Allow approximately 51 to 635 mm of vertical pen travel. Remove pen from paper and immediately open stopcock.

A2.6.5 Remove the specimen with a magnetized rod.

A2.6.6 Reposition the pen for the next determination and repeat the procedure.

A2.6.7 Change acid after every ten specimens.

A2.7 Calculation

A2.7.1 Extrapolate the upper straight-line portion of the curve to the horizontal base line.

A2.7.2 Measure the time in seconds along the horizontal base line between the origin of the curve and the point where the extrapolation intersects the base line. This time in seconds is defined as the *pickle lag*. A typical curve is shown in Fig. A2.1.

A2.8 Interferences

A2.8.1 Do not use rubber stoppers and tubing in contact with the acid. Some substance is extracted from the rubber, which acts as an inhibitor and increases lag time.

A2.8.2 Headspace in the vessel affects the slope of the corrosion–time curve. The total volume of headspace in the reaction vessel between the liquid level and the plug of the stopcock should be approximately 40 mL including the volume of the side arm to the manometer. Lag time is not affected by small variation in headspace volume.

A2.8.3 It is essential that the system be gas-tight. A periodic test to check the system is recommended. Attach an aspirator bulb to the reaction vessel inlet. Raise pressure to about 7 kPa. Close the stopcock and start the recording drum and holding pressure in system. If the system is gas-tight, the recording pen will draw a straight horizontal line.

A2.9 Assembly and Preparation of Apparatus

A2.9.1 It has been found convenient to alter the manometer (see A2.3.3) furnished with the equipment to avoid occasional problems of air entrapment in the mercury reservoir. The reservoir may be replaced with a stainless steel U-tube and connected to the two glass tubes with rubber tubing.

A2.9.2 Remove the front panel and the circular plate on top of the recorder (see Annex A2.3.3) to install the mercury manometer. Make an electrical connection from the mercury reservoir or the stainless steel U-tube to the electrical relay. With the traveling rack about 6.35 mm from its bottom position insert the moving electrical contact in the manometer arm with the reservoir trap at top and attach it to the top of the rack. Add mercury to the trap to bring the level up to the bottom of the moving contact. Add a drop of 6*N* HCl to the straight manometer arm to keep the wall clean. The arm should be cleaned or replaced when it becomes coated with mercury compounds.

A2.9.3 Connect the straight manometer arm to the reaction vessel with a 457 mm length of rubber or vinyl tubing, 4.76 mm inside diameter.

A2.9.4 Connect the mercury switch in series with the motor drive for the recorder drum. The switch is adjusted so the motor turns on when the stopcock of the reaction vessel is in the closed position. The rack should oscillate vertically when the switch on the top of the recorder is turned to the *on* position.

A2.9.5 Add a layer of paraffin oil approximately 6.35 mm thick to the water bath in order to minimize evaporation.

A2.9.6 Mount the reaction vessel in the constant-temperature water bath using a corrosion-resistant buret holder so that the side arm is 12.7 mm below the level of the bath. Stopcock grease or equivalent is used to lubricate the stopcock, which is firmly held in place by a 12.7 mm wide rubber band or other means.

A2.9.7 Fill the reaction vessel with 6 *N* HCl to the stopcock. Remove enough acid to provide a constant headspace of 40 mL in the reaction vessel and side arm. This is readily accomplished by lowering a glass tube of convenient bore to a predetermined depth (the glass tube should be marked for this purpose) and connecting it to a water aspirator. Any acid in the side arm should be expelled by squeezing the tubing connected to the side arm.

A3. METHODS FOR TIN CRYSTAL SIZE TEST FOR ELECTROLYTIC TIN PLATE

INTRODUCTION

The three methods described in this annex for estimating tin crystal size on electrolytic tin plate are typical of several possible methods to obtain the same result. Publication of these methods is not intended to preclude any other method that produces the same result.

A3.1 Scope

A3.1.1 The tin crystal size test is one of four special property tests used to measure certain characteristics of electrolytic tin plate, which affect internal corrosion resistance. The test is applicable to nominal tin coating weights 5.6/2.8 g/m^2 and heavier electrolytic tin plate (for K-plate, see 3.1.13.2 and J-plate, see 3.1.13.1). It is not applicable to 2.8/2.8 g/m^2 and lighter electrolytic tin plate.

A3.2 Summary of Method

A3.2.1 The surface of a piece of electrolytic tin plate is chemically etched or examined under polarized light to reveal the tin crystal pattern. The size of the tin crystals is estimated by comparison with ASTM macro-grain size number standards.

A3.3 Apparatus (Required Only for Method No. 3)

A3.3.1 *Polarized Light Source and Analyzer.*[5,6]

A3.4 Reagents and Materials (Required Only for Method No. 1)

A3.4.1 *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.[8] 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.

A3.4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A3.4.3 *Cotton or Soft Cloth.*

A3.4.4 *Ferric Chloride* ($FeCl_3·6H_2O$)—Chemically pure grade.

A3.4.5 *Hydrochloric Acid* (HCl) (1*N*)—Chemically pure grade.

A3.4.6 *Sodium Sulfide* ($Na_2S·9H_2O$) *or Sodium Bisulfate* ($NaHSO_3·H_2O$)—Chemically pure grade.

A3.5 Test Specimen

A3.5.1 The sample consists of any convenient size piece of fused electrolytic tin plate 25.8 cm^2 or larger.

A3.6 Procedure

A3.6.1 *Method No. 1*—Ferric chloride etch.

A3.6.1.1 Prepare etching solution by dissolving 100 g of $FeCl_3·6\ H_2O$ and 1 g of $Na_2S·9\ H_2O$ or $NaHSO_3·H_2O$ in 1000 mL of 1 *N* HCl. Solution is reusable but should be replaced when etching of specimen takes longer than 30 s.

A3.6.1.2 Buff surface of specimen vigorously but with light pressure with cotton or soft cloth. This disrupts the passive film and permits the etching solution to attack the tin readily.

A3.6.1.3 As an alternative to A3.6.1.2 and, if the equipment is available, cathodically clean specimen in 0.5 % sodium carbonate (Na_2CO_3) solution for 30 s. Reversing the polarity of the current for 1 s near the beginning of the cleaning cycle assists in removal of the passive layer. Rinse in tap water.

A3.6.1.4 Immerse specimen in etching solution for 5 to 15 s or until a crystal pattern develops. Remove, rinse in tap water, and dry. (Do not allow the specimen to remain in the etching solution too long as complete detinning will occur.)

A3.6.1.5 Estimate the tin crystal size number by comparing the specimen with ASTM macro-grain size number standards. (See Test Methods E112.) For routine testing, it is convenient to use a set of secondary standards consisting of actual tin plate specimens or photographs thereof at 1 × magnification.

A3.6.2 *Method No. 2*—Iron solution value disk.

A3.6.2.1 Examine the specimen after completion of the ISV test (see Annex A4) as it will already be suitably etched.

A3.6.2.2 Estimate tin crystal size same as in Method No. 1.

A3.6.3 *Method No. 3*—Polarized light.

A3.6.3.1 This is a rapid nondestructive method.

A3.6.3.2 Place the specimen in a beam of polarized light so the beam strikes the surface obliquely.

A3.6.3.3 Examine the reflected light beam through an analyzer. Rotate the analyzer to obtain best definition of tin crystal pattern.

A3.6.3.4 Estimate tin crystal size same as in Method No. 1.

A4. METHOD FOR DETERMINATION OF IRON SOLUTION VALUE ON ELECTROLYTIC PLATE

INTRODUCTION

It is not intended that variations in apparatus, sample preparation, or procedures from those described in this standard method be precluded. Suppliers or consumers may employ such variations for control purposes provided results agree with those obtained by the standard method.

A4.1 Scope

A4.1.1 The iron solution test,[4] also called the ISV test, is one of four special property tests used to measure certain characteristics of electrolytic tin plate, which affect internal corrosion resistance. The test is applicable to nominal tin coating weights 5.6/2.8 g⁄m^2, and heavier electrolytic tin plate (for K-plate, see 3.1.13.2 and J-plate, see 3.1.13.1). It is not applicable to 2.8/2.8 and lighter electrolytic tin plate.

A4.2 Summary of Method

A4.2.1 The iron solution test involves the colorimetric determination of the total amount of iron dissolved when 20.3 cm^2 of tin plate surface area are exposed for 2 h at 27 ± 0.5 °C to 50 mL of a mixture of dilute sulfuric acid (H_2SO_4), hydrogen peroxide (H_2O_2), and ammonium thiocyanate (NH_4SCN). The amount of iron dissolved expressed as micrograms is arbitrarily called the *iron solution value* (ISV).

A4.3 Apparatus[5,6]

A4.3.1 *Cabinet, Room, or Other Means* of maintaining 27 ± 0.5 °C during the test run.

A4.3.2 *Test Vessels,* round, tall-form, wide-mouth, approximately 236 mL glass bottles with 63 mm diameter plastic caps.

A4.3.3 *Gaskets* made from 1.59 mm thick vinyl sheeting. Gaskets have 51 mm inside diameter (ID) and 61.5 mm outside diameter (OD).

A4.3.4 *Burets*—Two 25 mL automatic filling rapid dispensing burets.

A4.3.5 *Equipment for Cathodically Cleaning Test Specimens*—The power source should be capable of supplying 1 to 1½ A per test specimen (26 cm^2 disk). A stainless steel beaker or tank is recommended as the cleaning vessel as it may also serve as the anode.

A4.3.6 *Spectrophotometer and Cuvettes.*

A4.4 Reagents and Materials

A4.4.1 *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.[8] 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.

A4.4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A4.4.3 *For Cleaning Test Specimen:*

A4.4.3.1 *Acetone.*

A4.4.3.2 *Sodium Carbonate Solution* (Na_2CO_3) (0.5 %).

A4.4.4 *For Iron Solution Test:*

A4.4.4.1 *Ammonium Thiocyanate Solution (Iron-Free)* (NH_4SCN).

A4.4.4.2 *Hydrogen Peroxide Solution* (H_2O_2) (30 %).

A4.4.4.3 *Sulfuric Acid* (H_2SO_4) (2.18 *N*).

A4.4.5 *For Calibration:*

A4.4.5.1 *Iron Wire, Analytical.*

A4.4.5.2 *Sulfuric Acid* (H_2SO_4) (10 *N*).

A4.5 Procedure

A4.5.1 *Test Solutions:*

A4.5.1.1 Prepare a 3 % solution of H_2O_2 by dilution of the 30 % grade.

A4.5.1.2 Prepare acid-peroxide stock solution by mixing in following proportions: 23 mL of H_2SO_4 (2.18 *N*) to 2 mL of H_2O_2 (3 %). (This mixture remains stable for several weeks.) Connect acid-peroxide stock solution bottle to one of the 25 mL automatic filling rapid-dispensing burets.

A4.5.1.3 Prepare a stock solution of NH_4SCN (40 g⁄L) and connect the stock bottle to the other buret.

A4.5.2 *Sample Preparation:*

A4.5.2.1 The specimen consists of a flat-circular piece of tin plate 57.33 ± 0.03 mm in diameter. This is equivalent to 25.8 cm^2. The specimen must be typical of the plate being tested and free of incidental deep scratches and surface conditions that are not representative of the tin plate under test.

A4.5.2.2 Cathodically clean the specimen in 0.5 % Na_2CO_3 solution for 30 s. Near the beginning of the cleaning cycle reverse the polarity of the current for 1 s. This 1 s anodic flash assists in removal of the oxides on the surface.

A4.5.2.3 Rinse the specimen successively in tap water and distilled or deionized water. Dry in acetone vapors. Do not touch the test surface.

A4.5.3 *Iron Solution Test:*

A4.5.3.1 Place the cleaned specimen, test surface up, in the plastic cap. (Paper liner should previously have been removed. To facilitate seating of gasket, the last 1.59 mm of cap thread may be removed by machining on a lathe.)

A4.5.3.2 Place the vinyl gasket over the specimen, seating it so that the gasket lies flat and holds the specimen firmly in place.

A4.5.3.3 Add 25 mL of the H_2SO_4-H_2O_2 stock solution and 25 mL of the NH_4SCN solution to the test vessel. Swirl to assure thorough mixing.