

# Standard Specification for Tin Mill Products, General Requirements<sup>1</sup>

This standard is issued under the fixed designation A623; 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  $(\varepsilon)$  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.

# 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 any 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 metric companion to Specification A623 has been developed—Specification A623M; therefore no metric equivalents are presented in this specification.

1.3 The following safety caveat covers Annex A3 through Annex A10 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 and health practices and determine the applicability of regulatory limitations prior to use.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

A370 Test Methods and Definitions for Mechanical Testing of Steel Products

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

A987 Practice for Measuring Shape Characteristics of Tin Mill Products

D1125 Test Methods for Electrical Conductivity and Resistivity of Water

E18 Test Methods for Rockwell Hardness of Metallic Materials

E112 Test Methods for Determining Average Grain Size

2.2 U.S. Military Standards:<sup>3</sup>

MIL-STD-129 Marking for Shipment and Storage
MIL-STD-163 Steel Mill Products, Preparation for Marking
and Storage

2.3 U.S. Federal Standard:<sup>3</sup>

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

# 3. Terminology

- 3.1 Definitions:
- 3.1.1 *base box*, *n*—a unit of area equivalent to 112 sheets 14 by 20 in. or 31 360 in.<sup>2</sup> (217.78 ft<sup>2</sup>) (see Annex A1).
- 3.1.2 base weight, n—a term used to describe the thickness of tin mill products. The designated base weight multiplied by a factor of 0.00011 is the nominal decimal thickness, in inches of the material. Although it is customary industry-wide to use the term "pound" (for example, 75 lb), following the base weight designation, base weight is correctly used only to define nominal material thickness, and is not a measure of the weight of a base box.
- 3.1.3 *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.
- 3.1.4 box annealing, n—a process involving slow heating of coils to a subcritical temperature, holding, and cooling therefrom, to soften the strip and 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.
- 3.1.5 *bright finish*, n—a surface that has a melted tin coating.
- 3.1.6 *bundle*, *n*—a unit containing two or more packages of a cut size, supported by a platform, generally consisting of ten or more packages. (Also commonly referred to as a multiple-package lift containing two or more packages.)
- 3.1.7 *burr*, *n*—metal displaced beyond the plane of the surface by slitting or shearing (see 9.1.7 and 9.2.6).

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

- 3.1.8 *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).
- 3.1.9 *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 A8 and Annex A10).
- 3.1.10 *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.
- 3.1.11 *cold reduction*—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.
- 3.1.12 continuous annealing—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 soften the strip and relieve stresses produced during cold reduction. An inert or slightly reducing atmosphere is maintained in the furnace to obtain a relatively bright strip.
- 3.1.13 *differentially coated tin plate*—electrolytic tin plate with a different weight of tin coating on each surface.
- 3.1.14 *double-reduced plate*—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.
- 3.1.15 *electrolytic chromium-coated steel*—light-gage, low-carbon, cold-reduced steel on which chromium and chromium oxides have been electrodeposited.
- 3.1.16 *electrolytic tin plate*—light-gage, low-carbon, cold-reduced steel on which tin has been electrodeposited by an acid or alkaline process.
- 3.1.16.1 *J Plate*—electrolytic tin plate, No. 50 or heavier tin coating, with improved corrosion performance for some galvanic detinning food products as specified in the table following 3.1.16.2 and as measured by the Special Property Tests for Pickle Lag (*PL*) (see Annex A3), Iron Solution Values (*ISV*) (see Annex A5), Tin Crystal Size (*TCS*) (see Annex A4). The alloy layer is normally light in color, characteristic of the acid tinning process.
- 3.1.16.2 *K Plate*—electrolytic tin plate, No. 50 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 A3), Iron Solution Value (*ISV*) (see Annex A5), Tin Crystal Size (*TCS*) (see Annex A4), and Alloy Tin Couple (*ATC*) (see Annex A6) and Aerated Media Polarization (AMP) (see Annex A9).

Pickle Lag<sup>A</sup> Iron Solution Value Tin Crystal Size Alloy Tin Couple<sup>B</sup> Special Properties Aims 10 s max 20 µg Iron max ASTM No. 9 or larger 0.12 µA/cm² max

- <sup>A</sup> The Pickle Lag test is not necessary if the product is processed using an anneal atmosphere gas of HNX or H<sub>2</sub>.
- $^{B}$  Good mill practice has demonstrated the ability to average 0.05  $\mu\text{A/cm}^{\,2}$  or less over an extended period of production.
- 3.1.16.3 *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
- 3.1.17 *length dimension*—the longer dimension of a cut size.
- 3.1.18 *lot*—each 20 000 sheets or part thereof or the equivalent in coils, of an item in a specific shipment having the same order specifications.
- 3.1.19 *matte finish*—a surface that has an unmelted tin coating, generally on a shot-blast finish (SBF) base steel.
- 3.1.20 *mechanical designation*—an arbitrary number to designate Rockwell hardness and ultimate tensile strength characteristics for double-reduced plate (see 8.2).
- 3.1.21 *oiling*—a lubricant film applied to both surfaces of the plate.
  - 3.1.22 package—a unit quantity of 112 sheets.
- 3.1.23 *passivating treatment*—a surface chemical treatment (see 3.1.9).
- 3.1.24 *ratio*—the number of base boxes in a package of a given size (see 3.1.1 and Annex A1).
- 3.1.25 *Rockwell hardness test*—a test for determining hardness (see Annex A2).
- 3.1.26 *rolling width*—the dimension of the sheet perpendicular to the rolling direction.
- 3.1.27 *single-reduced plate*—plate produced with one major cold reduction.
- 3.1.28 *steel Type D*—base-metal steel aluminum killed, sometimes required to minimize severe fluting and stretcherstrain hazards or for severe drawing applications (see Table 1).

**TABLE 1 Chemical Requirements for Tin Mill Products** 

Element	Cast	Composition, m	ax %
Liement	Type D	Type L	Type MR
Carbon	0.12	0.13	0.13
Manganese	0.60	0.60	0.60
Phosphorus	0.020	0.015	0.020
Sulfur	0.03	0.03	0.03
Silicon <sup>A,B</sup>	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 <sup>C</sup>	0.20	0.10	0.20
Other elements, each	0.02	0.02	0.02

 $^{A}\!W$ hen steel produced by the silicon killed method is ordered, the silicon maximum may be increased to 0.080 %.

<sup>B</sup>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.

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 %.



- 3.1.29 *steel Type L*—base-metal steel, low in metalloids and residual elements, sometimes used for improved internal corrosion resistance for certain food-product containers (see Table 1).
- 3.1.30 *steel Type MR*—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).
- 3.1.31 *surface appearance*—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.
- 3.1.32 *surface finishes*—steel surface finishes for tin mill products imparted by the finishing-mill work rolls. These may be either ground or blasted-roll finishes.
- 3.1.33 *temper designation*—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 Tables 2 and 3).
- 3.1.34 *temper mill*—a mill for rolling basemetal steel after annealing to obtain proper temper, flatness, and surface finish. It may consist of one stand or two stands arranged in tandem.
- 3.1.35 tin coating weight—the weight of tin applied to the steel surface, usually stated as pounds per base box, distributed evenly over both surfaces of a base box, the total coated area being 62 720 in.<sup>2</sup> Thus 0.25 lb/bb has a nominal weight of 0.125 lb on each of the two surfaces. Frequently, the coating is referred to as a designation number, and the decimal point is omitted. Thus, 0.25 lb/bb is 25.

#### TABLE 2 Temper Designations and Hardness Values Single-Reduced Tin Mill Products—Box Annealed

Note 1—Thinner plate (0.0083 in. ordered thickness and lighter) is normally tested using the Rockwell 15TS scale and the results converted to the Rockwell 30TS scale (see Annex A2 and Table A2.1).

Temper Desig-	Rockwell Hardr All Thicknesses		Characteristics and Typical
nation	Nominal	Range <sup>B</sup>	End Uses
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

 $<sup>^{</sup>A}\text{These}$  ranges are based on the use of the diamond spot anvil and a  $^{1}\!/_{16}$  in. hardened steel ball indenter.

Test conditions:

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

Note 1—Thinner plate (0.0083-in. ordered thickness and lighter) is normally tested using the Rockwell 15TS scale and the results converted to the Rockwell 30TS scale (see Annex A2 and Table A2.1).

Temper Designation	Rockwell Hardne All Thicknesses Nominal		Characteristics and Typical - End Uses
T-1	49	45–53	soft for drawing parts such as nozzles, spouts, and oil filter shells
T-2	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, closures, and crown caps
T-4 (T61)	61	57–65	increased stiffness for can ends, drawn (and ironed) can bodies, and large closures
T-5 (T65)	65	61–69	moderately high stiffness for can ends and bodies

 $<sup>^{</sup>A}\,$  These ranges are based on the use of the diamond spot anvil and a  $1\!/\!\!16$  in. hardened steel ball indenter.

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  $\frac{1}{2}$  in. off the center of the samples.
- 3.1.35.1 For differentially coated tin plate, twice the nominal coating weight on each side is designated, usually by the number method; hence, 10/25 designates the nominal weight of 0.05 lb/bb on one side and 0.125 lb/bb on the other side.
- 3.1.36 *vapor vacuum deposition*—the condensation and solidification of the metal or metal containing vapors, under high vacuum, to form deposits onto a steel surface.
  - 3.1.37 *width dimension*—the shorter dimension of a cut size.

# 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.

 $<sup>^{</sup>B}$ The hardness ranges are requirements unless otherwise agreed upon between producer and user.

<sup>1.</sup> For referee purposes, samples of blackplate, unreflowed ETP, and ECCS shall be aged prior to testing by holding at 400°F for 10 min.

<sup>2.</sup> For referee purposes, the hardness test area on material produced with SBF or equivalent rolls shall be sanded smooth on both surfaces.

<sup>3.</sup> To avoid incorrect results due to the cantilever effect, samples shall have an area no larger than 4 in.<sup>2</sup> and the point of testing shall be no more than  $\frac{1}{2}$  in. off the center of the samples.

 $<sup>^{\</sup>rm B}\!{\rm The}$  hardness ranges are requirements unless otherwise agreed upon between producer and user.



# 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 Tables 2 and 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.

# TABLE 4 Mechanical Designations Double-Reduced Tin Mill Products

Note 1—Thinner plate (0.0083 in. ordered thickness and lighter) is normally tested using Rockwell 15TS scale and the results converted to the Rockwell 30TS scale (see Annex A2 and Table A2.1).

Designa- tion <sup>B</sup>	Nominal Longitudinal Ultimate Tensile Strengh psi	Nominal Rockwell Hardness HR30- TS <sup>A</sup>	Examples of Usage
DR-7.5	75 000	71	can bodies
DR-8	80 000	72	can bodies and ends
DR-8.5	85 000	73	can bodies and ends
DR-9	90 000	75	can bodies and ends
DR-9.5	95 000	76	can ends

<sup>&</sup>lt;sup>A</sup> These values are based on the use of the diamond spot anvil and a ½6 in. hardened 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.

8.3 Rockwell testing shall be in accordance with the latest revision of Test Methods and Definitions A370 and Test Methods E18 (see Annex A2).

#### 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 within 1 in. from the mill trimmed edge. The hand micrometers are assumed to be accurate to  $\pm 0.0001$  in. No measurements are to be made within 3 ft 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 ½ in. 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.
- 9.1.4 *Crown* is the difference in strip thickness from the center of roll width and the locations 1 in. in from both mill-trimmed edges.
- 9.1.5 Feather Edge is the maximum difference in thickness across the strip width between points measured at ½ in. and 1 in. from both mill-trimmed edges. The thickness ¼ in. from an edge is usually less than the thickness measured 1 in. or more from the same edge.
- 9.1.6 *Width*—Coils are trimmed to the ordered width. The slit dimension shall not vary over width by more than -0,  $+\frac{1}{8}$  in.
- 9.1.7 *Burr*—A maximum of 0.002 in. 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.

#### **TABLE 5 Thickness Tolerances**

Note 1—When coils are specified, this does not afford the supplier the opportunity to discard all off-gage product and for that reason the following thickness tolerances are applicable for various lot sizes.

Lot Size, lb	Tolerance
0 to 12 000	95 % of the product of the coils shall be within the tolerances stated in Table 6
Over 12 000 to 30 000	97.5 % of the product of the coils shall be within the tolerances stated in Table 6
Over 30 000 to 150 000	99.0 % of the product of the coils shall be within the tolerances stated in Table 6
Over 150 000	99.5 % of the product of the coils shall be within the tolerances stated in Table 6

<sup>&</sup>lt;sup>B</sup>Double-reduced products requiring a minimum % elongation or ductility will be designated as HEDR (e.g., 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 6 Ordered Thickness and Thickness Tolerances** 

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

Ordered	Thickness	Thickness
Thickness,	Tolerance,	Tolerances
in.	Over, in.	Under, in.
0.0050	0.0003	0.0004
0.0055	0.0003	0.0004
0.0061	0.0003	0.0005
0.0066	0.0003	0.0005
0.0072	0.0004	0.0006
0.0077	0.0004	0.0006
0.0083	0.0004	0.0007
0.0088	0.0004	0.0007
0.0094	0.0005	0.0008
0.0099	0.0005	0.0008
0.0105	0.0005	0.0008
0.0110	0.0006	0.0009
0.0113	0.0006	0.0009
0.0118	0.0006	0.0009
0.0123	0.0006	0.0010
0.0130	0.0007	0.0010
0.0141	0.0007	0.0011
0.0149	0.0007	0.0012

- 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.
- 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 base weight, over periods of one month or one quarter, or both should be checked.
- 9.1.9 *Camber* is limited to a maximum of ½ in. in 20 ft of length, in accordance with the latest revision of measuring methods and definitions in Test Method A987.
- 9.1.10 *Inside Coil Diameters*—The standard inside diameter produced is approximately 16 in.
  - 9.2 Dimensional Characteristics, Cut Sizes:
- 9.2.1 Thickness, Method for Determination—Random measurements must be made at least 1 in. from the edge of the sheet using a hand micrometer. The hand micrometers are assumed to be accurate to  $\pm 0.0001$  in.
- 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 ¼ in. 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

**TABLE 7 Coil Length Variation** 

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

- 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 locations 1 in. in from both mill-trimmed edges.
- 9.2.5 Feather Edge is the maximum difference in thickness across the strip width between points measured at  $\frac{1}{4}$  in. and 1 in. from both mill-trimmed edges. The thickness  $\frac{1}{4}$  in. from an edge is usually less than the thickness measured 1 in. or more from the same edge.
- 9.2.6 *Burr*—A maximum of 0.002 in. 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 ½16 in. for each 48 in. of length or fraction thereof, in accordance with the latest revision 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 ½6 in. for any edge measurement, except that a bundle may contain a maximum of four sheets with a deviation up to ½ in.
- 9.2.9 Shearing Practice—Tin mill products are sheared to the ordered width and to the ordered length. The greater dimension is considered length. The slit dimension shall not vary over the designated overrun by more than -0,  $+\frac{1}{8}$  in. and the drum cut dimension shall not vary over the designated overrun by more than -0,  $+\frac{1}{4}$  in.

#### 10. Special Requirements

- 10.1 Welds—Coils may contain lap or mash 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 1 in.
- 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/bundle should average out to at least the proper exact count in quantities of 400 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 tin plate 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 tin mill products

# ANNEXES

(Mandatory Information)

#### A1. ABBREVIATED RATIO TABLES FOR TIN MILL PRODUCTS

A1.1 The base box is the unit of area of 112 sheets 14 by 20 in. or 31 360 in. $^2$  (217.78 ft $^2$ ).

A1.2 To determine the number of base boxes represented by 112 sheets of any other dimensions, a computation is necessary. The computation is carried out using ratio tables.

A1.2.1 Tables A1.1-A1.6 $^4$  are an abbreviated set of such ratio tables, which can be used to determine the number of base boxes represented by 112 and 1000 sheets in sizes from  $\frac{1}{16}$  in. square to 50 in. square.

A1.2.2 The following example demonstrates the use of these tables. The example applies to various sheet dimensions as follows:

A1.2.2.1 Sheet with No Fractional Dimensions—Step 1 only.

A1.2.2.2 Sheet with Fractional Dimensions on Only One Dimension—Steps 1 and 2.

A1.2.2.3 Sheet with Fractional Dimensions on Both Dimensions—Steps 1, 2, 3, and 4.

A1.2.3 An example of the use of abbreviated ratio tables to develop the number of base boxes represented by 112 and 1000 sheets with specified dimensions  $28\frac{1}{16}$  by  $34\frac{1}{2}$  in. is given in Table A1.7.

<sup>&</sup>lt;sup>4</sup> These tables are reproduced, by permission of the American Iron and Steel Institute, from "Tin Mill Products," *Steel Products Manual*, AISI, 1963.

TABLE A1.1 Tin Plate Ratios—Base Boxes per 112 Sheets Full-Inch Widths

Full-Inch Lengths	0	1	2	3	4	5	6	7	8	9
1 2 3 4 5	.0000 .0000 .0000 .0000	.0036 .0071 .0107 .0143 .0179	.0071 .0143 .0214 .0286 .0357	.0107 .0214 .0321 .0429 .0536	.0143 .0286 .0429 .0571 .0714	.0179 .0357 .0536 .0714 .0893	.0214 .0429 .0643 .0857 .1071	.0250 .0500 .0750 .1000 .1250	.0286 .0571 .0857 .1143 .1429	.0321 .0643 .0964 .1286 .1607
6 7 8 9 10	.0000 .0000 .0000 .0000	.0214 .0250 .0286 .0321 .0357	.0429 .0500 .0571 .0643 .0714	.0643 .0750 .0857 .0964 .1071	.0857 .1000 .1143 .1286 .1429	.1071 .1250 .1429 .1607 .1786	.1286 .1500 .1714 .1929 .2143	.1500 .1750 .2000 .2250 .2500	.1714 .2000 .2286 .2571 .2857	.1929 .2250 .2571 .2893 .3214
11 12 13 14 15	.0000 .0000 .0000 .0000	.0393 .0429 .0464 .0500 .0536	.0786 .0857 .0929 .1000 .1071	.1179 .1286 .1393 .1500 .1607	.1571 .1714 .1857 .2000 .2143	.1964 .2143 .2321 .2500 .2679	.2357 .2571 .2786 .3000 .3214	.2750 .3000 .3250 .3500 .3750	.3143 .3429 .3714 .4000 .4286	.3536 .3857 .4179 .4500 .4821
16 17 18 19 20	.0000 .0000 .0000 .0000	.0571 .0607 .0643 .0679 .0714	.1143 .1214 .1286 .1357 .1429	.1714 .1821 .1929 .2036 .2143	.2286 .2429 .2571 .2714 .2857	.2857 .3036 .3214 .3393 .3571	.3429 .3643 .3857 .4071 .4286	.4000 .4250 .4500 .4750 .5000	.4571 .4857 .5143 .5429 .5714	.5143 .5464 .5786 .6107 .6429
21 22 23 24 25	.0000 .0000 .0000 .0000	.0750 .0786 .0821 .0857 .0893	.1500 .1571 .1643 .1714 .1786	.2250 .2357 .2464 .2571 .2679	.3000 .3143 .3286 .3429 .3571	.3750 .3929 .4107 .4286 .4464	.4500 .4714 .4929 .5143 .5357	.5250 .5500 .5750 .6000 .6250	.6000 .6286 .6571 .6857 .7143	.6750 .7071 .7393 .7714 .8036
26 27 28 29 30	.0000 .0000 .0000 .0000	.0929 .0964 .1000 .1036 .1071	.1857 .1929 .2000 .2071 .2143	.2786 .2893 .3000 .3107 .3214	.3714 .3857 .4000 .4143 .4286	.4643 .4821 .5000 .5179 .5357	.5571 .5786 .6000 .6214 .6429	.6500 .6750 .7000 .7250 .7500	.7429 .7714 .8000 .8286 .8571	.8357 .8679 .9000 .9321 .9643
31 32 33 34 35	.0000 .0000 .0000 .0000	.1107 .1143 .1179 .1214 .1250	.2214 .2286 .2357 .2429 .2500	.3321 .3429 .3536 .3643 .3750	.4429 .4571 .4714 .4857 .5000	.5536 .5714 .5893 .6071 .6250	.6643 .6857 .7071 .7286 .7500	.7750 .8000 .8250 .8500 .8750	.8857 .9143 .9429 .9714 1.0000	.9964 1.0286 1.0607 1.0929 1.1250
36 37 38 39 40	.0000 .0000 .0000 .0000	.1286 .1321 .1357 .1393 .1429	.2571 .2643 .2714 .2786 .2857	.3857 .3964 .4071 .4179 .4286	.5143 .5286 .5429 .5571 .5714	.6429 .6607 .6786 .6964 .7143	.7714 .7929 .8143 .8357 .8571	.9000 .9250 .9500 .9750 1.0000	1.0286 1.0571 1.0857 1.1143 1.1429	1.1571 1.1893 1.2214 1.2536 1.2857
41 42 43 44 45	.0000 .0000 .0000 .0000	.1464 .1500 .1536 .1571 .1607	.2929 .3000 .3071 .3143 .3214	.4393 .4500 .4607 .4714 .4821	.5857 .6000 .6143 .6286 .6429	.7321 .7500 .7679 .7857 .8036	.8786 .9000 .9214 .9429 .9643	1.0250 1.0500 1.0750 1.1000 1.1250	1.1714 1.2000 1.2286 1.2571 1.2857	1.3179 1.3500 1.3821 1.4143 1.4464
46 47 48 49 50	.0000 .0000 .0000 .0000	.1643 .1679 .1714 .1750 .1786	.3286 .3357 .3429 .3500 .3571	.4929 .5036 .5143 .5250 .5357	.6571 .6714 .6857 .7000 .7143	.8214 .8393 .8571 .8750 .8929	.9857 1.0071 1.0286 1.0500 1.0714	1.1500 1.1750 1.2000 1.2250 1.2500	1.3143 1.3429 1.3714 1.4000 1.4286	1.4786 1.5107 1.5429 1.5750 1.6071



# TABLE A1.1 Continued

Full-Inch Lengths										
	10	11	12	13	14	15	16	17	18	19
10	.3571	.3929	.4286	.4643	.5000	.5357	.5714	.6071	.6429	.6786
11	.3929	.4321	.4714	.5107	.5500	.5893	.6286	.6679	.7071	.7464
12	.4286	.4714	.5143	.5571	.6000	.6429	.6857	.7286	.7714	.8143
13	.4643	.5107	.5571	.6036	.6500	.6964	.7429	.7893	.8357	.8821
14	.5000	.5500	.6000	.6500	.7000	.7500	.8000	.8500	.9000	.9500
15	.5357	.5893	.6429	.6964	.7500	.8036	.8571	.9107	.9643	1.0179
16	.5714	.6286	.6857	.7429	.8000	.8571	.9143	.9714	1.0286	1.0857
17	.6071	.6679	.7286	.7893	.8500	.9107	.9714	1.0321	1.0929	1.1536
18	.6429	.7071	.7714	.8357	.9000	.9643	1.0286	1.0929	1.1571	1.2214
19	.6786	.7464	.8143	.8821	.9500	1.0179	1.0857	1.1536	1.2214	1.2893
20	.7143	.7857	.8571	.9286	1.0000	1.0714	1.1429	1.2143	1.2857	1.3571
21	.7500	.8250	.9000	.9750	1.0500	1.1250	1.2000	1.2750	1.3500	1.4250
22	.7857	.8643	.9429	1.0214	1.1000	1.1786	1.2571	1.3357	1.4143	1.4929
23	.8214	.9036	.9857	1.0679	1.1500	1.2321	1.3143	1.3964	1.4786	1.5607
24	.8571	.9429	1.0286	1.1143	1.2000	1.2857	1.3714	1.4571	1.5429	1.6286
25	.8929	.9821	1.0714	1.1607	1.2500	1.3393	1.4286	1.5179	1.6071	1.6964
26	.9286	1.0214	1.1143	1.2071	1.3000	1.3929	1.4857	1.5786	1.6714	1.7643
27	.9643	1.0607	1.1571	1.2536	1.3500	1.4464	1.5429	1.6393	1.7357	1.8321
28	1.0000	1.1000	1.2000	1.3000	1.4000	1.5000	1.6000	1.7000	1.8000	1.9000
29	1.0357	1.1393	1.2429	1.3464	1.4500	1.5536	1.6571	1.7607	1.8643	1.9679
30	1.0714	1.1786	1.2857	1.3929	1.5000	1.6071	1.7143	1.8214	1.9286	2.0357
31	1.1071	1.2179	1.3286	1.4393	1.5500	1.6607	1.7714	1.8821	1.9929	2.1036
32	1.1429	1.2571	1.3714	1.4857	1.6000	1.7143	1.8286	1.9429	2.0571	2.1714
33	1.1786	1.2964	1.4143	1.5321	1.6500	1.7679	1.8857	2.0036	2.1214	2.2393
34	1.2143	1.3357	1.4571	1.5786	1.7000	1.8214	1.9429	2.0643	2.1857	2.3071
35	1.2500	1.3750	1.5000	1.6250	1.7500	1.8750	2.0000	2.1250	2.2500	2.3750
36	1.2857	1.4143	1.5429	1.6714	1.8000	1.9286	2.0571	2.1857	2.3143	2.4429
37	1.3214	1.4536	1.5857	1.7179	1.8500	1.9821	2.1143	2.2464	2.3786	2.5107
38	1.3571	1.4929	1.6286	1.7643	1.9000	2.0357	2.1714	2.3071	2.4429	2.5786
39	1.3929	1.5321	1.6714	1.8107	1.9500	2.0893	2.2286	2.3679	2.5071	2.6464
40	1.4286	1.5714	1.7143	1.8571	2.0000	2.1429	2.2857	2.4286	2.5714	2.7143
41	1.4643	1.6107	1.7571	1.9036	2.0500	2.1964	2.3429	2.4893	2.6357	2.7821
42	1.5000	1.6500	1.8000	1.9500	2.1000	2.2500	2.4000	2.5500	2.7000	2.8500
43	1.5357	1.6893	1.8429	1.9964	2.1500	2.3036	2.4571	2.6107	2.7643	2.9179
44	1.5714	1.7286	1.8857	2.0429	2.2000	2.3571	2.5143	2.6714	2.8286	2.9857
45	1.6071	1.7679	1.9286	2.0893	2.2500	2.4107	2.5714	2.7321	2.8929	3.0536
46	1.6429	1.8071	1.9714	2.1357	2.3000	2.4643	2.6286	2.7929	2.9571	3.1214
47	1.6786	1.8464	2.0143	2.1821	2.3500	2.5179	2.6857	2.8536	3.0214	3.1893
48	1.7143	1.8857	2.0571	2.2286	2.4000	2.5714	2.7429	2.9143	3.0857	3.2571
49	1.7500	1.9250	2.1000	2.2750	2.4500	2.6250	2.8000	2.9750	3.1500	3.3250
50	1.7857	1.9643	2.1429	2.3214	2.5000	2.6786	2.8571	3.0357	3.2143	3.3929



# TABLE A1.1 Continued

Full-Inch Lengths										
22	20	21	22	23	24	25	26	27	28	29
20	1.4286	1.5000	1.5714	1.6429	1.7143	1.7857	1.8571	1.9286	2.0000	2.0714
21	1.5000	1.5750	1.6500	1.7250	1.8000	1.8750	1.9500	2.0250	2.1000	2.1750
22	1.5714	1.6500	1.7286	1.8071	1.8857	1.9643	2.0429	2.1214	2.2000	2.2786
23	1.6429	1.7250	1.8071	1.8893	1.9714	2.0536	2.1357	2.2179	2.3000	2.3821
24	1.7143	1.8000	1.8857	1.9714	2.0571	2.1429	2.2286	2.3143	2.4000	2.4857
25	1.7857	1.8750	1.9643	2.0536	2.1429	2.2321	2.3214	2.4107	2.5000	2.5893
26	1.8571	1.9500	2.0429	2.1357	2.2286	2.3214	2.4143	2.5071	2.6000	2.6929
27	1.9286	2.0250	2.1214	2.2179	2.3143	2.4107	2.5071	2.6036	2.7000	2.7964
28	2.0000	2.1000	2.2000	2.3000	2.4000	2.5000	2.6000	2.7000	2.8000	2.9000
29	2.0714	2.1750	2.2786	2.3821	2.4857	2.5893	2.6929	2.7964	2.9000	3.0036
30	2.1429	2.2500	2.3571	2.4643	2.5714	2.6786	2.7857	2.8929	3.0000	3.1071
31	2.2143	2.3250	2.4357	2.5464	2.6571	2.7679	2.8786	2.9893	3.1000	3.2107
32	2.2857	2.4000	2.5143	2.6286	2.7429	2.8571	2.9714	3.0857	3.2000	3.3143
33	2.3571	2.4750	2.5929	2.7107	2.8286	2.9464	3.0643	3.1821	3.3000	3.4179
34	2.4286	2.5500	2.6714	2.7929	2.9143	3.0357	3.1571	3.2786	3.4000	3.5214
35	2.5000	2.6250	2.7500	2.8750	3.0000	3.1250	3.2500	3.3750	3.5000	3.6250
36	2.5714	2.7000	2.8286	2.9571	3.0857	3.2143	3.3429	3.4714	3.6000	3.7286
37	2.6429	2.7750	2.9071	3.0393	3.1714	3.3036	3.4357	3.5679	3.7000	3.8321
38	2.7143	2.8500	2.9857	3.1214	3.2571	3.3929	3.5286	3.6643	3.8000	3.9357
39	2.7857	2.9250	3.0643	3.2036	3.3429	3.4821	3.6214	3.7607	3.9000	4.0393
40	2.8571	3.0000	3.1429	3.2857	3.4286	3.5714	3.7143	3.8571	4.0000	4.1429
41	2.9286	3.0750	3.2214	3.3679	3.5143	3.6607	3.8071	3.9536	4.1000	4.2464
42	3.0000	3.1500	3.3000	3.4500	3.6000	3.7500	3.9000	4.0500	4.2000	4.3500
43	3.0714	3.2250	3.3786	3.5321	3.6857	3.8393	3.9929	4.1464	4.3000	4.4536
44	3.1429	3.3000	3.4571	3.6143	3.7714	3.9286	4.0857	4.2429	4.4000	4.5571
45	3.2143	3.3750	3.5357	3.6964	3.8571	4.0179	4.1786	4.3393	4.5000	4.6607
46	3.2857	3.4500	3.6143	3.7786	3.9429	4.1071	4.2714	4.4357	4.6000	4.7643
47	3.3571	3.5250	3.6929	3.8607	4.0286	4.1964	4.3643	4.5321	4.7000	4.8679
48	3.4286	3.6000	3.7714	3.9429	4.1143	4.2857	4.4571	4.6286	4.8000	4.9714
49	3.5000	3.6750	3.8500	4.0250	4.2000	4.3750	4.5500	4.7250	4.9000	5.0750
50	3.5714	3.7500	3.9286	4.1071	4.2857	4.4643	4.6429	4.8214	5.0000	5.1786

# TABLE A1.1 Continued

Full-Inch Lengths	30	31	32	33	34	35	36	37	38	39
30	3.2143	3.3214	3.4286	3.5357	3.6429	3.7500	3.8571	3.9643	4.0714	4.1786
31	3.3214	3.4321	3.5429	3.6536	3.7643	3.8750	3.9857	4.0964	4.2071	4.3179
32	3.4286	3.5429	3.6571	3.7714	3.8857	4.0000	4.1143	4.2286	4.3429	4.4571
33	3.5357	3.6536	3.7714	3.8893	4.0071	4.1250	4.2429	4.3607	4.4786	4.5964
34	3.6429	3.7643	3.8857	4.0071	4.1286	4.2500	4.3714	4.4929	4.6143	4.7357
35	3.7500	3.8750	4.0000	4.1250	4.2500	4.3750	4.5000	4.6250	4.7500	4.8750
36	3.8571	3.9857	4.1143	4.2429	4.3714	4.5000	4.6286	4.7571	4.8857	5.0143
37	3.9643	4.0964	4.2286	4.3607	4.4929	4.6250	4.7571	4.8893	5.0214	5.1536
38	4.0714	4.2071	4.3429	4.4786	4.6143	4.7500	4.8857	5.0214	5.1571	5.2929
39	4.1786	4.3179	4.4571	4.5964	4.7357	4.8750	5.0143	5.1536	5.2929	5.4321
40	4.2857	4.4286	4.5714	4.7143	4.8571	5.0000	5.1429	5.2857	5.4286	5.5714
41	4.3929	4.5393	4.6857	4.8321	4.9786	5.1250	5.2714	5.4179	5.5643	5.7107
42	4.5000	4.6500	4.8000	4.9500	5.1000	5.2500	5.4000	5.5500	5.7000	5.8500
43	4.6071	4.7607	4.9143	5.0679	5.2214	5.3750	5.5286	5.6821	5.8357	5.9893
44	4.7143	4.8714	5.0286	5.1857	5.3429	5.5000	5.6571	5.8143	5.9714	6.1286
45	4.8214	4.9821	5.1429	5.3036	5.4643	5.6250	5.7857	5.9464	6.1071	6.2679
46	4.9286	5.0929	5.2571	5.4214	5.5857	5.7500	5.9143	6.0786	6.2429	6.4071
47	5.0357	5.2036	5.3714	5.5393	5.7071	5.8750	6.0429	6.2107	6.3786	6.5464
48	5.1429	5.3143	5.4857	5.6571	5.8286	6.0000	6.1714	6.3429	6.5143	6.6857
49	5.2500	5.4250	5.6000	5.7750	5.9500	6.1250	6.3000	6.4750	6.6500	6.8250
50	5.3571	5.5357	5.7143	5.8929	6.0714	6.2500	6.4286	6.6071	6.7857	6.9643
I-Inch ngths										

Full-Inch Lengths	40		42	42		4-	4.5		40	40
27	40	41	42	43	44	45	46	47	48	49
40	5.7143	5.8571	6.0000	6.1429	6.2857	6.4286	6.5714	6.7143	6.8571	7.0000
41 42 43 44 45	5.8571 6.0000 6.1429 6.2857 6.4286	6.0036 6.1500 6.2964 6.4429 6.5893	6.1500 6.3000 6.4500 6.6000 6.7500	6.2964 6.4500 6.6036 6.7571 6.9107	6.4429 6.6000 6.7571 6.9143 7.0714	6.5893 6.7500 6.9107 7.0714 7.2321	6.7357 6.9000 7.0643 7.2286 7.3929	6.8821 7.0500 7.2179 7.3857 7.5536	7.0286 7.2000 7.3714 7.5429 7.7143	7.1750 7.3500 7.5250 7.7000 7.8750
46 47 48 49 50	6.5714 6.7143 6.8571 7.0000 7.1429	6.7357 6.8821 7.0286 7.1750 7.3214	6.9000 7.0500 7.2000 7.3500 7.5000	7.0643 7.2179 7.3714 7.5250 7.6786	7.2286 7.3857 7.5429 7.7000 7.8571	7.3929 7.5536 7.7143 7.8750 8.0357	7.5571 7.7214 7.8857 8.0500 8.2143	7.7214 7.8893 8.0571 8.2250 8.3929	7.8857 8.0571 8.2286 8.4000 8.5714	8.0500 8.2250 8.4000 8.5750 8.7500

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**50** 8.9286

TABLE A1.2 Tin Plate Ratios—Base Boxes per 112 Sheets Full-Inch Widths

Full-Inch Lengths	1/16	1/8	3/16	1/4	5/16	3/8	7/16	1/2	9/16	5/8	11/16	3/4	13/16	7/8	15/16
1 2 3 4 5	.0002 .0004 .0007 .0009	.0004 .0009 .0013 .0018 .0022	.0007 .0013 .0020 .0027 .0033	.0009 .0018 .0027 .0036 .0045	.0011 .0022 .0033 .0045 .0056	.0013 .0027 .0040 .0054 .0067	.0016 .0031 .0047 .0063 .0078	.0018 .0036 .0054 .0071 .0089	.0020 .0040 .0060 .0080 .0100	.0022 .0045 .0067 .0089 .0112	.0025 .0049 .0074 .0098 .0123	.0027 .0054 .0080 .0107 .0134	.0029 .0058 .0087 .0116 .0145	.0031 .0063 .0094 .0125 .0156	.0033 .0067 .0100 .0134 .0167
6	.0013	.0027	.0040	.0054	.0067	.0080	.0094	.0107	.0121	.0134	.0147	.0161	.0174	.0188	.0201
7	.0016	.0031	.0047	.0063	.0078	.0094	.0109	.0125	.0141	.0156	.0172	.0188	.0203	.0219	.0234
8	.0018	.0036	.0054	.0071	.0089	.0107	.0125	.0143	.0161	.0179	.0196	.0214	.0232	.0250	.0268
9	.0020	.0040	.0060	.0080	.0100	.0121	.0141	.0161	.0181	.0201	.0221	.0241	.0261	.0281	.0301
10	.0022	.0045	.0067	.0089	.0112	.0134	.0156	.0179	.0201	.0223	.0246	.0268	.0290	.0313	.0335
11	.0025	.0049	.0074	.0098	.0123	.0147	.0172	.0196	.0221	.0246	.0270	.0295	.0319	.0344	.0368
12	.0027	.0054	.0080	.0107	.0134	.0161	.0188	.0214	.0241	.0268	.0295	.0321	.0348	.0375	.0402
13	.0029	.0058	.0087	.0116	.0145	.0174	.0203	.0232	.0261	.0290	.0319	.0348	.0377	.0406	.0435
14	.0031	.0063	.0094	.0125	.0156	.0188	.0219	.0250	.0281	.0313	.0344	.0375	.0406	.0438	.0469
15	.0033	.0067	.0100	.0134	.0167	.0201	.0234	.0268	.0301	.0335	.0368	.0402	.0435	.0469	.0502
16	.0036	.0071	.0107	.0143	.0179	.0214	.0250	.0286	.0321	.0357	.0393	.0429	.0464	.0500	.0536
17	.0038	.0076	.0114	.0152	.0190	.0228	.0266	.0304	.0342	.0379	.0417	.0455	.0493	.0531	.0569
18	.0040	0080	.0121	.0161	.0201	.0241	.0281	.0321	.0362	.0402	.0442	.0482	.0522	.0563	.0603
19	.0042	.0085	.0127	.0170	.0212	.0254	.0297	.0339	.0382	.0424	.0467	.0509	.0551	.0594	.0636
20	.0045	.0089	.0134	.0179	.0223	.0268	.0313	.0357	.0402	.0446	.0491	.0536	.0580	.0625	.0670
21	.0047	.0094	.0141	.0188	.0234	.0281	.0328	.0375	.0422	.0469	.0516	.0563	.0609	.0656	.0703
22	.0049	.0098	.0147	.0196	.0246	.0295	.0344	.0393	.0442	.0491	.0540	.0589	.0638	.0688	.0737
23	.0051	.0103	.0154	.0205	.0257	.0308	.0359	.0411	.0462	.0513	.0565	.0616	.0667	.0719	.0770
24	.0054	.0107	.0161	.0214	.0268	.0321	.0375	.0429	.0482	.0536	.0589	.0643	.0696	.0750	.0804
25	.0056	.0112	.0167	.0223	.0279	.0335	.0391	.0446	.0502	.0558	.0614	.0670	.0725	.0781	.0837
26	.0058	.0116	.0174	.0232	.0290	.0348	.0406	.0464	.0522	.0580	.0638	.0696	.0754	.0813	.0871
27	.0060	.0121	.0181	.0241	.0301	.0362	.0422	.0482	.0542	.0603	.0663	.0723	.0783	.0844	.0904
28	.0063	.0125	.0188	.0250	.0313	.0375	.0438	.0500	.0563	.0625	.0688	.0750	.0813	.0875	.0938
29	.0065	.0129	.0194	.0259	.0324	.0388	.0453	.0518	.0583	.0647	.0712	.0777	.0842	.0906	.0971
30	.0067	.0134	.0201	.0268	.0335	.0402	.0469	.0536	.0603	.0670	.0737	.0804	.0871	.0938	.1004
31	.0069	.0138	.0208	.0277	.0346	.0415	.0484	.0554	.0623	.0692	.0761	.0830	.0900	.0969	.1038
32	.0071	.0143	.0214	.0286	.0357	.0429	.0500	.0571	.0643	.0714	.0786	.0857	.0929	.1000	.1071
33	.0074	.0147	.0221	.0295	.0368	.0442	.0516	.0589	.0663	.0737	.0810	.0884	.0958	.1031	.1105
34	.0076	.0152	.0228	.0304	.0379	.0455	.0531	.0607	.0683	.0759	.0835	.0911	.0987	.1063	.1138
35	.0078	.0156	.0234	.0313	.0391	.0469	.0547	.0625	.0703	.0781	.0859	.0938	.1016	.1094	.1172
36	.0080	.0161	.0241	.0321	.0402	.0482	.0563	.0643	.0723	.0804	.0884	.0964	.1045	.1125	.1205
37	.0083	.0165	.0248	.0330	.0413	.0496	.0578	.0661	.0743	.0826	.0908	.0991	.1074	.1156	.1239
38	.0085	.0170	.0254	.0339	.0424	.0509	.0594	.0679	.0763	.0848	.0933	.1018	.1103	.1188	.1272
39	.0087	.0174	.0261	.0348	.0435	.0522	.0609	.0696	.0783	.0871	.0958	.1045	.1132	.1219	.1306
40	.0089	.0179	.0268	.0357	.0446	.0536	.0625	.0714	.0804	.0893	.0982	.1071	.1161	.1250	.1339
41	.0092	.0183	.0275	.0366	.0458	.0549	.0641	.0732	.0824	.0915	.1007	.1098	.1190	.1281	.1373
42	.0094	.0188	.0281	.0375	.0469	.0563	.0656	.0750	.0844	.0938	.1031	.1125	.1219	.1313	.1406
43	.0096	.0192	.0288	.0384	.0480	.0576	.0672	.0768	.0864	.0960	.1056	.1152	.1248	.1344	.1440
44	.0098	.0196	.0295	.0393	.0491	.0589	.0688	.0786	.0884	.0982	.1080	.1179	.1277	.1375	.1473
45	.0100	.0201	.0301	.0402	.0502	.0603	.0703	.0804	.0904	.1004	.1105	.1205	.1306	.1406	.1507
46	.0103	.0205	.0308	.0411	.0513	.0616	.0719	.0821	.0924	.1027	.1129	.1232	.1335	.1438	.1540
47	.0105	.0210	.0315	.0420	.0525	.0629	.0734	.0839	.0944	.1049	.1154	.1259	.1364	.1469	.1574
48	.0107	.0214	.0321	.0429	.0536	.0643	.0750	.0857	.0964	.1071	.1179	.1286	.1393	.1500	.1607
49	.0109	.0219	.0328	.0438	.0547	.0656	.0766	.0875	.0984	.1094	.1203	.1313	.1422	.1531	.1641
50	.0112	.0223	.0335	.0446	.0558	.0670	.0781	.0893	.1004	.1116	.1228	.1339	.1451	.1563	.1674

TABLE A1.3 Tin Plate Ratios—Base Boxes per 112 Sheets Fractional Widths and Length

15/16	.000 .000 .000 .000 .000 .000	.0010 .0013 .0015	.0019 .0021 .0023	.0027 .0029 .0031
2/8		.0010 .0012 .0014 .0016	.0018 .0020 .0021 .0023	.0025 .0027 .0029
13/16	.0002 .0004 .0005 .0007	.0009 .0011 .0013 .0015	.0016 .0018 .0020 .0020	.0024 .0025 .0027
3/4	.0002 .0003 .0005 .0007	.0008 .0010 .0012 .0013	.0015 .0017 .0018 .0020	.0022 .0023 .0025
11/16	.0002	.0008 .0009 .0011 .0012	.0015 .0015 .0017 .0018	.0020 .0021 .0023
8/8	.000. .0003 .0004	.0007 .0008 .00100.	.0013 .0014 .0015	.0018 .0020 .0021
9/16	.0001 .0003 .0005	.0006 .0009 .0009	.0011 .0013 .0014	.0016 .0018 .0019
1/2	.000.	.0000 .0000 .0009	.0010 .0011 .0012 .0013	.0015
7/16	.000. .0002 .0003 .0004	.0005 .0006 .0007 .0008	.0009 .0010 .0011 .0012	.0013 .0014 .0015
3/8	.0001	.0005 .0005 .0006	.0008	.0011
5/16	.000 .000 .000 .000 .0003	.0003	.0006 .0007 .0008 .0008	.0009
1/4	.0001	.0003	.0005	.0007 .0008 .0008
3/16	.0000.	00003	0000.	.0005
1/8			.000 .000 .000 .000 .000	.000 .000 .000 .000
1/16	00000.		.0001	
	\frac{1}{2} \frac{1}{8} \frac{1}{2} \frac{1}{4}	5/16 3/8 7/16 1/2	2 2 2 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	13/1 7/8 15/1

TABLE A1.4 Tin Plate Ratios—Base Boxes per 1000 Sheets Full-Inch Widths

Full-Inch Lengths	0	1	2	3	4	5	6	7	8	9
1 2 3 4 5	.00 .00 .00 .00	.03 .06 .10 .13	.06 .13 .19 .26 .32	.10 .19 .29 .38 .48	.13 .26 .38 .51 .64	.16 .32 .48 .64 .80	.19 .38 .57 .77 .96	.22 .45 .67 .89 1.12	.26 .51 .77 1.02 1.28	.29 .57 .86 1.15 1.43
6 7 8 9 10	.00 .00 .00 .00	.19 .22 .26 .29 .32	.38 .45 .51 .57 .64	.57 .67 .77 .86 .96	.77 .89 1.02 1.15 1.28	.96 1.12 1.28 1.43 1.59	1.15 1.34 1.53 1.72 1.91	1.34 1.56 1.79 2.01 2.23	1.53 1.79 2.04 2.30 2.55	1.72 2.01 2.30 2.58 2.87
11 12 13 14 15	.00 .00 .00 .00	.35 .38 .41 .45 .48	.70 .77 .83 .89 .96	1.05 1.15 1.24 1.34 1.43	1.40 1.53 1.66 1.79 1.91	1.75 1.91 2.07 2.23 2.39	2.10 2.30 2.49 2.68 2.87	2.46 2.68 2.90 3.13 3.35	2.81 3.06 3.32 3.57 3.83	3.16 3.44 3.73 4.02 4.30
16 17 18 19 20	.00 .00 .00 .00	.51 .54 .57 .61 .64	1.02 1.08 1.15 1.21 1.28	1.53 1.63 1.72 1.82 1.91	2.04 2.17 2.30 2.42 2.55	2.55 2.71 2.87 3.03 3.19	3.06 3.25 3.44 3.64 3.83	3.57 3.79 4.02 4.24 4.46	4.08 4.34 4.59 4.85 5.10	4.59 4.88 5.17 5.45 5.74
21 22 23 24 25	.00 .00 .00 .00	.67 .70 .73 .77	1.34 1.40 1.47 1.53 1.59	2.01 2.10 2.20 2.30 2.39	2.68 2.81 2.93 3.06 3.19	3.35 3.51 3.67 3.83 3.99	4.02 4.21 4.40 4.59 4.78	4.69 4.91 5.13 5.36 5.58	5.36 5.61 5.87 6.12 6.38	6.03 6.31 6.60 6.89 7.17
26 27 28 29 30	.00 .00 .00 .00	.83 .86 .89 .92 .96	1.66 1.72 1.79 1.85 1.91	2.49 2.58 2.68 2.77 2.87	3.32 3.44 3.57 3.70 3.83	4.15 4.30 4.46 4.62 4.78	4.97 5.17 5.36 5.55 5.74	5.80 6.03 6.25 6.47 6.70	6.63 6.89 7.14 7.40 7.65	7.46 7.75 8.04 8.32 8.61
31 32 33 34 35	.00 .00 .00 .00	.99 1.02 1.05 1.08 1.12	1.98 2.04 2.10 2.17 2.23	2.97 3.06 3.16 3.25 3.35	3.95 4.08 4.21 4.34 4.46	4.94 5.10 5.26 5.42 5.58	5.93 6.12 6.31 6.51 6.70	6.92 7.14 7.37 7.59 7.81	7.91 8.16 8.42 8.67 8.93	8.90 9.18 9.47 9.76 10.04
36 37 38 39 40	.00 .00 .00 .00	1.15 1.18 1.21 1.24 1.28	2.30 2.36 2.42 2.49 2.55	3.44 3.54 3.64 3.73 3.83	4.59 4.72 4.85 4.97 5.10	5.74 5.90 6.06 6.22 6.38	6.89 7.08 7.27 7.46 7.65	8.04 8.26 8.48 8.71 8.93	9.18 9.44 9.69 9.95 10.20	10.33 10.62 10.91 11.19 11.48
41 42 43 44 45	.00 .00 .00 .00	1.31 1.34 1.37 1.40 1.43	2.61 2.68 2.74 2.81 2.87	3.92 4.02 4.11 4.21 4.30	5.23 5.36 5.48 5.61 5.74	6.54 6.70 6.86 7.02 7.17	7.84 8.04 8.23 8.42 8.61	9.15 9.38 9.60 9.82 10.04	10.46 10.71 10.97 11.22 11.48	11.77 12.05 12.34 12.63 12.91
46 47 48 49 50	.00 .00 .00 .00	1.47 1.50 1.53 1.56 1.59	2.93 3.00 3.06 3.13 3.19	4.40 4.50 4.59 4.69 4.78	5.87 5.99 6.12 6.25 6.38	7.33 7.49 7.65 7.81 7.97	8.80 8.99 9.18 9.38 9.57	10.27 10.49 10.71 10.94 11.16	11.73 11.99 12.24 12.50 12.76	13.20 13.49 13.78 14.06 14.35



# TABLE A1.4 Continued

Full-Inch Lengths										
돌	10	11	12	13	14	15	16	17	18	19
10	3.19	3.51	3.83	4.15	4.46	4.78	5.10	5.42	5.74	6.06
11	3.51	3.86	4.21	4.56	4.91	5.26	5.61	5.96	6.31	6.66
12	3.83	4.21	4.59	4.97	5.36	5.74	6.12	6.51	6.89	7.27
13	4.15	4.56	4.97	5.39	5.80	6.22	6.63	7.05	7.46	7.88
14	4.46	4.91	5.36	5.80	6.25	6.70	7.14	7.59	8.04	8.48
15	4.78	5.26	5.74	6.22	6.70	7.17	7.65	8.13	8.61	9.09
16	5.10	5.61	6.12	6.63	7.14	7.65	8.16	8.67	9.18	9.69
17	5.42	5.96	6.51	7.05	7.59	8.13	8.67	9.22	9.76	10.30
18	5.74	6.31	6.89	7.46	8.04	8.61	9.18	9.76	10.33	10.91
19	6.06	6.66	7.27	7.88	8.48	9.09	9.69	10.30	10.91	11.51
20	6.38	7.02	7.65	8.29	8.93	9.57	10.20	10.84	11.48	12.12
21	6.70	7.37	8.04	8.71	9.38	10.04	10.71	11.38	12.05	12.72
22	7.02	7.72	8.42	9.12	9.82	10.52	11.22	11.93	12.63	13.33
23	7.33	8.07	8.80	9.53	10.27	11.00	11.73	12.47	13.20	13.93
24	7.65	8.42	9.18	9.95	10.71	11.48	12.24	13.01	13.78	14.54
25	7.97	8.77	9.57	10.36	11.16	11.96	12.76	13.55	14.35	15.15
26	8.29	9.12	9.95	10.78	11.61	12.44	13.27	14.09	14.92	15.75
27	8.61	9.47	10.33	11.19	12.05	12.91	13.78	14.64	15.50	16.36
28	8.93	9.82	10.71	11.61	12.50	13.39	14.29	15.18	16.07	16.96
29	9.25	10.17	11.10	12.02	12.95	13.87	14.80	15.72	16.65	17.57
30	9.57	10.52	11.48	12.44	13.39	14.35	15.31	16.26	17.22	18.18
31	9.89	10.87	11.86	12.85	13.84	14.83	15.82	16.80	17.79	18.78
32	10.20	11.22	12.24	13.27	14.29	15.31	16.33	17.35	18.37	19.39
33	10.52	11.58	12.63	13.68	14.73	15.78	16.84	17.89	18.94	19.99
34	10.84	11.93	13.01	14.09	15.18	16.26	17.35	18.43	19.52	20.60
35	11.16	12.28	13.39	14.51	15.63	16.74	17.86	18.97	20.09	21.21
36	11.48	12.63	13.78	14.92	16.07	17.22	18.37	19.52	20.66	21.81
37	11.80	12.98	14.16	15.34	16.52	17.70	18.88	20.06	21.24	22.42
38	12.12	13.33	14.54	15.75	16.96	18.18	19.39	20.60	21.81	23.02
39	12.44	13.68	14.92	16.17	17.41	18.65	19.90	21.14	22.39	23.63
40	12.76	14.03	15.31	16.58	17.86	19.13	20.41	21.68	22.96	24.23
41	13.07	14.38	15.69	17.00	18.30	19.61	20.92	22.23	23.53	24.84
42	13.39	14.73	16.07	17.41	18.75	20.09	21.43	22.77	24.11	25.45
43	13.71	15.08	16.45	17.83	19.20	20.57	21.94	23.31	24.68	26.05
44	14.03	15.43	16.84	18.24	19.64	21.05	22.45	23.85	25.26	26.66
45	14.35	15.78	17.22	18.65	20.09	21.52	22.96	24.39	25.83	27.26
46	14.67	16.14	17.60	19.07	20.54	22.00	23.47	24.94	26.40	27.87
47	14.99	16.49	17.98	19.48	20.98	22.48	23.98	25.48	26.98	28.48
48	15.31	16.84	18.37	19.90	21.43	22.96	24.49	26.02	27.55	29.08
49	15.63	17.19	18.75	20.31	21.88	23.44	25.00	26.56	28.13	29.69
50	15.94	17.54	19.13	20.73	22.32	23.92	25.51	27.10	28.70	30.29



# TABLE A1.4 Continued

ths										
Full-Inch Lengths	20	21	22	23	24	25	26	27	28	29
20	12.76	13.39	14.03	14.67	15.31	15.94	16.58	17.22	17.86	18.49
21	13.39	14.06	14.73	15.40	16.07	16.74	17.41	18.08	18.75	19.42
22	14.03	14.73	15.43	16.14	16.84	17.54	18.24	18.94	19.64	20.34
23	14.67	15.40	16.14	16.87	17.60	18.34	19.07	19.80	20.54	21.27
24	15.31	16.07	16.84	17.60	18.37	19.13	19.90	20.66	21.43	22.19
25	15.94	16.74	17.54	18.34	19.13	19.93	20.73	21.52	22.32	23.12
26	16.58	17.41	18.24	19.07	19.90	20.73	21.56	22.39	23.21	24.04
27	17.22	18.08	18.94	19.80	20.66	21.52	22.39	23.25	24.11	24.97
28	17.86	18.75	19.64	20.54	21.43	22.32	23.21	24.11	25.00	25.89
29	18.49	19.42	20.34	21.27	22.19	23.12	24.04	24.97	25.89	26.82
30	19.13	20.09	21.05	22.00	22.96	23.92	24.87	25.83	26.79	27.74
31	19.77	20.76	21.75	22.74	23.72	24.71	25.70	26.69	27.68	28.67
32	20.41	21.43	22.45	23.47	24.49	25.51	26.53	27.55	28.57	29.59
33	21.05	22.10	23.15	24.20	25.26	26.31	27.36	28.41	29.46	30.52
34	21.68	22.77	23.85	24.94	26.02	27.10	28.19	29.27	30.36	31.44
35	22.32	23.44	24.55	25.67	26.79	27.90	29.02	30.13	31.25	32.37
36	22.96	24.11	25.26	26.40	27.55	28.70	29.85	30.99	32.14	33.29
37	23.60	24.78	25.96	27.14	28.32	29.50	30.68	31.86	33.04	34.22
38	24.23	25.45	26.66	27.87	29.08	30.29	31.51	32.72	33.93	35.14
39	24.87	26.12	27.36	28.60	29.85	31.09	32.33	33.58	34.82	36.07
40	25.51	26.79	28.06	29.34	30.61	31.89	33.16	34.44	35.71	36.99
41	26.15	27.46	28.76	30.07	31.38	32.68	33.99	35.30	36.61	37.91
42	26.79	28.13	29.46	30.80	32.14	33.48	34.82	36.16	37.50	38.84
43	27.42	28.79	30.17	31.54	32.91	34.28	35.65	37.02	38.39	39.76
44	28.06	29.46	30.87	32.27	33.67	35.08	36.48	37.88	39.29	40.69
45	28.70	30.13	31.57	33.00	34.44	35.87	37.31	38.74	40.18	41.61
46	29.34	30.80	32.27	33.74	35.20	36.67	38.14	39.60	41.07	42.54
47	29.97	31.47	32.97	34.47	35.97	37.47	38.97	40.47	41.96	43.46
48	30.61	32.14	33.67	35.20	36.73	38.27	39.80	41.33	42.86	44.39
49	31.25	32.81	34.38	35.94	37.50	39.06	40.63	42.19	43.75	45.31
50	31.89	33.48	35.08	36.67	38.27	39.86	41.45	43.05	44.64	46.24

# TABLE A1.4 Continued

Full-Inch Lengths										
돌골	30	31	32	33	34	35	36	37	38	39
30	28.70	29.66	30.61	31.57	32.53	33.48	34.44	35.40	36.35	37.31
31	29.66	30.64	31.63	32.62	33.61	34.60	35.59	36.58	37.56	38.55
32	30.61	31.63	32.65	33.67	34.69	35.71	36.73	37.76	38.78	39.80
33	31.57	32.62	33.67	34.73	35.78	36.83	37.88	38.93	39.99	41.04
34	32.53	33.61	34.69	35.78	36.86	37.95	39.03	40.11	41.20	42.28
35	33.48	34.60	35.71	36.83	37.95	39.06	40.18	41.29	42.41	43.53
36	34.44	35.59	36.73	37.88	39.03	40.18	41.33	42.47	43.62	44.77
37	35.40	36.58	37.76	38.93	40.11	41.29	42.47	43.65	44.83	46.01
38	36.35	37.56	38.78	39.99	41.20	42.41	43.62	44.83	46.05	47.26
39	37.31	38.55	39.80	41.04	42.28	43.53	44.77	46.01	47.26	48.50
40	38.27	39.54	40.82	42.09	43.37	44.64	45.92	47.19	48.47	49.74
41	39.22	40.53	41.84	43.14	44.45	45.76	47.07	48.37	49.68	50.99
42	40.18	41.52	42.86	44.20	45.54	46.88	48.21	49.55	50.89	52.23
43	41.14	42.51	43.88	45.25	46.62	47.99	49.36	50.73	52.10	53.48
44	42.09	43.49	44.90	46.30	47.70	49.11	50.51	51.91	53.32	54.72
45	43.05	44.48	45.92	47.35	48.79	50.22	51.66	53.09	54.53	55.96
46	44.01	45.47	46.94	48.41	49.87	51.34	52.81	54.27	55.74	57.21
47	44.96	46.46	47.96	49.46	50.96	52.46	53.95	55.45	56.95	58.45
48	45.92	47.45	48.98	50.51	52.04	53.57	55.10	56.63	58.16	59.69
49	46.88	48.44	50.00	51.56	53.13	54.69	56.25	57.81	59.38	60.94
50	47.83	49.43	51.02	52.61	54.21	55.80	57.40	58.99	60.59	62.18
Full-Inch Lengths	40	41	42	43	44	45	46	47	48	49
40	51.02	52.30	53.57	54.85	56.12	57.40	58.67	59.95	61.22	62.50
41	52.30	53.60	54.91	56.22	57.53	58.83	60.14	61.45	62.76	64.06
42	53.57	54.91	56.25	57.59	58.93	60.27	61.61	62.95	64.29	65.63
43	54.85	56.22	57.59	58.96	60.33	61.70	63.07	64.45	65.82	67.19
44	56.12	57.53	58.93	60.33	61.73	63.14	64.54	65.94	67.35	68.75
45	57.40	58.83	60.27	61.70	63.14	64.57	66.01	67.44	68.88	70.31
46	58.67	60.14	61.61	63.07	64.54	66.01	67.47	68.94	70.41	71.88
47	59.95	61.45	62.95	64.45	65.94	67.44	68.94	70.44	71.94	73.44
48	61.22	62.76	64.29	65.82	67.35	68.88	70.41	71.94	73.47	75.00
49	62.50	64.06	65.63	67.19	68.75	70.31	71.88	73.44	75.00	76.56
50	63.78	65.37	66.96	68.56	70.15	71.75	73.34	74.94	76.53	78.13
	50									
50	79.72									

**♣** A623 – 16

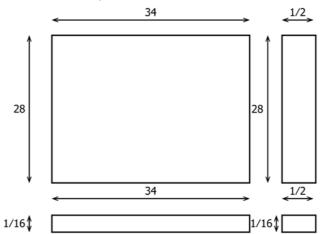
TABLE A1.5 Tin Plate Ratios—Base Boxes per 1000 Sheets Fractional Widths

Full-Inch Lengths		1/8		1/4	_	3/8		1/2		5/8		3/4		7/8	
	1/16		3/16		5/16		7/16		9/16		11/16		13/16		15/16
1 2 3 4	.00 .00	.00 .01	.01 .01	.01 .02	.01 .02	.01 .02	.01 .03	.02 .03	.02 .04	.02 .04	.02 .04	.02 .05	.03 .05	.03 .06	.03 .06
3 4 5	.01 .01 .01	.01 .02 .02	.02 .02 .03	.02 .03 .04	.03 .04 .05	.04 .05 .06	.04 .06 .07	.05 .06 .08	.05 .07 .09	.06 .08 .10	.07 .09 .11	.07 .10 .12	.08 .10 .13	.08 .11 .14	.09 .12 .15
6	.01	.02	.04	.05	.06	.07	.08	.10	.11	.12	.13	.14	.16	.17	
7 8	.01	.03	.04 .05	.06 .06	.07	.08	.10 .11	.11 .13	.13 .14	.14 .16	.15	.17 .19	.18 .21	.20 .22 .25	.18 .21 .24 .27 .30
9 10	.02 .02	.04 .04	.05 .06	.07 .08	.09 .10	.11 .12	.13 .14	.14 .16	.16 .18	.18 .20	.18 .20 .22	.22 .24	.23 .26	.28	.30
11 12	.02 .02	.04 .05	.07 .07	.09 .10	.11 .12	.13 .14	.15 .17	.18 .19	.20 .22	.22 .24	.24 .26	.26 .29	.28 .31	.31 .33 .36	.33 .36
12 13 14 15	.03 .03	.05 .06	.08 .08	.10 .11	.13 .14	.16 .17	.18 .20	.19 .21 .22	.22 .23 .25 .27	.26 .28	.28 .31	.29 .31 .33 .36	.34 .36	.39	.33 .36 .39 .42 .45
16	.03	.06	.10	.12	.15	.18	.21	.24		.30	.33	.36	.39	.42 .45	
17 18	.03 .04	.07 .07	.10 .10	.14 .14	.17 .18	.20	.22 .24 .25 .27 .28	.26 .27 .29 .30 .32	.29 .30 .32	.34 .36	.35 .37 .39	.41 .43	.44 .47	.47 .50	.48 .51 .54 .57 .60
17 18 19 20	.04	.08 .08	.11	.15 .16	.19	.20 .22 .23 .24	.27	.30	.34	.38	.42 .44	.45 .48	.49 .52	.53 .56	.57 .60
21	.04 .04	.08 .09	.13 .13	.17 .18	.21 .22	.25 .26	.29	.33 .35 .37	.38 .39	.42 .44	.46 .48	.50 .53	.54 .57	.59 .61	.63
21 22 23 24 25	.05 .05	.09	.14 .14	.18 .19	.23	.28 .29 .30	.29 .31 .32 .33 .35	.38	.41 .43	.46 .48	.50 .53	.55 .57 .60	.60 .62	.64 .67	.63 .66 .69 .72 .75
	.05	.10	.15	.20	.24			.40	.45	.50	.55		.65	.70	
26 27	.05 .05 .06	.10 .11 .11	.16 .16 .17	.21 .22 .22	.26 .27	.31 .32	.36 .38 .39	.41 .43 .45	.47 .48	.52 .54	.57 .59 .61	.62 .65 .67	.67 .70 .73	.73 .75 .78	.78 .81 .84 .87
28 29 30	.06 .06	.12	.17 .18	.23	.28 .29 .30	.32 .33 .35 .36	.40 .42	.46 .48	.50 .52 .54	.56 .58 .60	.64 .66	.69 .72	.75 .78	.81 .84	.87 .90
31	.06	.12	.19	.25	.31	.37	.43	.49 .51	.56 .57	.62	.68	.74	.80	.87	.93
31 32 33 34 35	.06 .07	.13 .13	.19 .20	.26 .26	.31 .32 .33	.38 .39	.45 .46	.51 .53	.59	.64 .66	.70 .72	.77 .79	.83 .86	.89 .92	.96 .99
	.07 .07	.14 .14	.20 .21	.27 .28	.34 .35	.41 .42	.47 .49	.53 .54 .56	.61 .63	.68 .70	.75 .77	.81 .84	.88 .91	.95 .98	1.02 1.05
36 37 38	.07 .07	.14 .15 .15	.22 .22	.29 .29	.36 .37	.43 .44	.50 .52	.57 .59 .61	.65 .66	.72 .74	.79 .81	.86 .88 .91	.93 .96	1.00 1.03	1.08 1.11
38 39 40	.08 .08	.16	.22	.30 .31	.38 .39	.45 .47	.52 .53 .54	.62	.68 .70	.76 .78	.83 .86	.93	.98 1.01	1.06 1.09	1.11 1.14 1.17 1.20
40 41	.08 .08	.16 .16	.24	.32 33	.40 .41	.48 .49	.56 57	.64 .65	.72 .74	.80 .82	.88 .90	.96 .98	1.04 1.06	1.12	
42 43	.08 .09	.17 .17	.25 .25 .26	.33 .33 .34	.42 .43	.50 .51	.57 .59 .60	.67 .69	.75 .77	.84 .86	.92 .94	1.00 1.03	1.09 1.11	1.14 1.17 1.20	1.26 1.29
44 45	.09	.18 .18	.26 .27	.35 .36	.44 .45	.53 .54	.61 .63	.70 .72	.79 .81	.88	.96 .99	1.05 1.08	1.14 1.17	1.23 1.26	1.23 1.26 1.29 1.32 1.35
46 47	.09	.18 .19	.28 .28	.37 .37	.46 .47	.55 .56	.64 .66	.73 .75	.83 .84	.92 .94	1.01 1.03	1.10 1.12	1.19 1.22	1.28 1.31	1.38 1.41
48 49	.10 .10	.19 .20	.28 .29 .29	.38 .39	.48 .49	.56 .57 .59	.67 .68	.75 .77 .78	.86 .88	.96 .98	1.05 1.07	1.15 1.17	1.24 1.27	1.34 1.37	1.38 1.41 1.43 1.46 1.49
50	.10	.20	.30	.40	.50	.60	.70	.80	.90	1.00	1.10	1.20	1.30	1.40	1.49

TABLE A1.6 Tin Plate Ratios—Base Boxes per 1000 Sheets Fractional Widths and Lengths

		1/8		1/4		3/8		1/2		5/8		3/4		7/8	
	1/16		3/16		5/16		7/16		9/16		11/16		13/16		15/16
1/ <sub>16</sub> 1/ <sub>8</sub> 3/ <sub>16</sub> 1/ <sub>4</sub>	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .01	.00 .00 .01 .01
5/ <sub>16</sub> 3/ <sub>8</sub> 7/ <sub>16</sub> 1/ <sub>2</sub>	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .01	.00 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01
9/16 5/8 11/16 3/4	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .01 .01 .01	.01 .01 .01 .01	.01 .01 .01	.01 .01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .02 .02	.01 .01 .02 .02	.01 .02 .02 .02	.02 .02 .02 .02	.02 .02 .02 .02
13/ <sub>16</sub> 7/ <sub>8</sub> 15/ <sub>16</sub>	.00 .00 .00	.00 .00 .00	.00 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .01 .01	.01 .02 .02	.02 .02 .02	.02 .02 .02	.02 .02 .02	.02 .02 .02	.02 .02 .03	.02 .03 .03

TABLE A1.7 Example of the Use of Abbreviated Ratio Tables



Dimensional Components of a 281/16 by 341/2-in. Sheet for Use in Abbreviated Ratio Tables

Step	Components	112 Sheet	Table	1000 Sheet	Table
		Ratio <sup>A</sup>		Ratio <sup>B</sup>	
1	34 by 28	3.4000	A1.1	30.36	A1.4
2	28 by ½	0.0500	A1.2	0.45	A1.5
3	½16 by 34	0.0076	A1.2	0.07	A1.5
4	½ by ½	0.0001	A1.3	0.00	A1.6
Total		3.4577		30.88	

A One-hundred twelve sheets measuring  $28\frac{1}{16}$  by  $34\frac{1}{2}$  in. represents 3.4577 base boxes. B One-thousand sheets measuring  $28\frac{1}{16}$  by  $34\frac{1}{2}$  in. represents 30.88 base boxes.

#### A2. ROCKWELL HARDNESS TESTING OF TIN MILL PRODUCTS

## A2.1 Scope

A2.1.1 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 latest revision of Test Methods and Definitions A370 and with the methods outlined in Test Methods E18, with the exceptions given in the following sections.

#### A2.2 Anvil

A2.2.1 All tests shall be made using the diamond spot anvil and a ½16 in. hardened steel ball indenter.

# A2.3 Specimens

A2.3.1 *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, in.	Rockwell Superficial Scale	Major Load, kgf
0.0083 and less	15TS	15
0.0215-0.0084	30TS	30

A2.3.2 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.

# **A2.4 Reports**

A2.4.1 *Number of Tests*—The Rockwell scale value to be reported shall be the average of at least three impressions.

A2.4.2 *Conversion*—Hardness tests made on the 15TS scale may be converted to the 30TS scale by the use of Table A2.1.

TABLE A2.1 Conversion Table (Approximation) Rockwell Hardness Testing

Tidi diless resting											
HR 30TS	HR 15TS	HR 30TS	HR 15TS								
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									

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.

# A3. 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 specification method be precluded. Suppliers or consumers may employ such variations for control purposes provided test results agree with results obtained by the standard method.

# A3.1 Scope

A3.1.1 The rate of pickling test,<sup>5</sup> 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 No. 50, No. 50/25, and heavier electrolytic tin plate (for K-plate, see 3.1.16.2 and J-plate, see 3.1.16.1). It is not applicable to No. 25 and lighter electrolytic tin plate.

# A3.2 Summary of Method

A3.2.1 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.

# A3.3 Apparatus

A3.3.1 *Reaction Vessel*, 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.

A3.3.2 Constant-Temperature Water Bath, large enough to accommodate the reaction vessel and maintain a temperature of  $90 \pm 0.5^{\circ}$  C.

A3.3.3 *Recording Mercury Manometer,* to measure the rate of increase in pressure in the vessel generated by hydrogen. Initial setup of the recorder is described in Section 10.

A3.3.4 A15 by ½-in. 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.)

A3.3.5 *Coordinate Paper*, 4 by 11 in., with either 10 or 20 gradations to the inch.

#### A3.4 Reagents and Materials

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.<sup>6</sup> 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 For Rate of Pickling Test:

A3.4.3.1 Hydrochloric Acid (HCl), (6 N).

A3.4.4 For Sample Preparation:

A3.4.4.1 Acetone.

A3.4.4.2 *Antimony Trichloride Solution (120 g/L)*— Dissolve 120 g of antimony trichloride (SbCl<sub>3</sub>) in 1 L of concentrated HCl.

A3.4.4.3 Sodium Carbonate Solution (Na<sub>2</sub>CO<sub>3</sub>) (0.5 %).

A3.4.4.4 Sodium Hydroxide Solution (NaOH) (10 %).

A3.4.4.5 Sodium Peroxide (Na<sub>2</sub>O<sub>2</sub>), granulated.

A3.4.5 For Water Bath:

A3.4.5.1 Paraffin Oil.

# **A3.5** Test Specimen Preparation

A3.5.1 *Test Specimen*— A piece of steel <sup>5</sup>/<sub>16</sub> by 2½ in. with the long dimension perpendicular to the rolling direction of the steel.

A3.5.1.1 Cut a piece of metal  $\frac{5}{16}$  by 4 in. or longer. The added length above the  $2\frac{1}{2}$  in. serves as a handle during preparation.

A3.5.1.2 Remove surface oil and grease by dipping the specimen in acetone and wiping with a cloth or paper towel.

A3.5.1.3 Cathodically clean the specimen in 0.5 % solution of  $Na_2CO_3$ , rinse in water, and dry.

A3.5.1.4 Detin the specimen by immersing in SbCl<sub>3</sub> -HCl solution at room temperature. Allow the specimen to remain in solution 10 to 20 s after bubbling ceases.

A3.5.1.5 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.)

A3.5.1.6 Immerse specimen in 10 % NaOH solution held at 90°C for approximately 1 min. During this time add granulated Na<sub>2</sub>O<sub>2</sub> 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.

A3.5.1.7 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.

A3.5.1.8 Trim specimen to 5/16 by 21/2 in.

A3.5.1.9 Handle the specimen with forceps as touching with the fingers may produce erratic test results.

#### A3.6 Procedure

A3.6.1 Bring the constant-temperature water bath to 90  $\pm$  0.5°C, making certain the 6 N HCl in the reaction vessel has also reached 90°C, if it has been freshly transferred.

A3.6.2 Start recorder and place the pen against the graph paper near the bottom.

A3.6.3 Drop the specimen into the reaction vessel and immediately close the stopcock. The mercury switch will start

<sup>&</sup>lt;sup>5</sup> 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.

<sup>&</sup>lt;sup>6</sup> 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 U.K. Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



the recorder drum turning. The pressure generated by reaction of the acid on the specimen will cause the pen to rise.

A3.6.4 Allow approximately 2 to  $2\frac{1}{2}$  in. of vertical pen travel. Remove pen from paper and immediately open stopcock.

A3.6.5 Remove the specimen with a magnetized rod.

A3.6.6 Reposition the pen for the next determination and repeat the procedure.

A3.6.7 Change acid after every ten specimens.

#### A3.7 Calculation

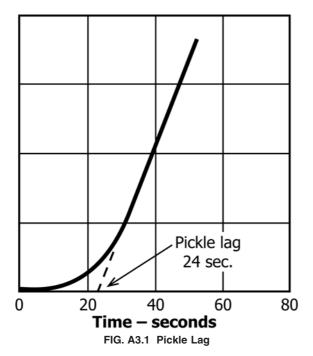
A3.7.1 Extrapolate the upper straight-line portion of the curve to the horizontal base line.

A3.7.2 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. A3.1.

#### A3.8 Interferences

A3.8.1 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.

A3.8.2 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.

A3.8.3 It is essential that the system be gastight. A periodic test to check the system is recommended. Attach an aspirator bulb to the reaction vessel inlet. Raise pressure to about 2 in. Hg. Close the stopcock and start the recording drum and holding pressure in system. If the system is gastight, the recording pen will draw a straight horizontal line.

#### A3.9 Assembly and Preparation of Apparatus

A3.9.1 It has been found convenient to alter the manometer (see A3.3.3) 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.

A3.9.2 Remove the front panel and the circular plate on top of the recorder (see A3.3.3) 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  $\frac{1}{4}$  in. 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.

A3.9.3 Connect the straight manometer arm to the reaction vessel with an 18-in. length of rubber or vinyl tubing,  $\frac{3}{16}$ -in. inside diameter.

A3.9.4 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.

A3.9.5 Add a layer of paraffin oil approximately ½ in. thick to the water bath in order to minimize evaporation.

A3.9.6 Mount the reaction vessel in the constant-temperature water bath using a corrosion-resistant burst holder so that the side arm is ½ in. below the level of the bath. Stopcock grease or equivalent is used to lubricate the stopcock, which is firmly held in place by a ½-in. wide rubber band or other means.

A3.9.7 Fill the reaction vessel with 6 NHCl 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.

#### A4. 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.

## A4.1 Scope

A4.1.1 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 No. 50, No. 50/25, and heavier electrolytic tin plate (for K-plate, see 3.1.16.2 and J-plate, see 3.1.16.1). It is not applicable to No. 25 and lighter electrolytic tin plate.

# A4.2 Summary of Methods

A4.2.1 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.

# A4.3 Apparatus (Required Only for Method No. 3)

A4.3.1 Polarized Light Source and Analyzer.

# A4.4 Reagents and Materials (Required Only for Method No. 1)

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.<sup>6</sup> 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.

A4.4.2.1 Cotton or Soft Cloth.

A4.4.2.2 Ferric Chloride (FeCl<sub>3</sub>· $6H_2O$ )—Chemically pure grade.

A4.4.2.3 *Hydrochloric Acid (HCl) (1 N)*—Chemically pure grade.

A4.4.2.4 Sodium Sulfide (Na<sub>2</sub>S·9H<sub>2</sub>O) or Sodium Bisulfate (NaHSO<sub>3</sub>·H<sub>2</sub>O)—Chemically pure grade.

# A4.5 Test Specimen

A4.5.1 The sample consists of any convenient size piece of fused electrolytic tin plate 4 in.<sup>2</sup> or larger.

# A4.6 Procedure

A4.6.1 Method No. 1—Ferric chloride etch.

A4.6.1.1 Prepare etching solution by dissolving 100 g of FeCl<sub>3</sub>·6 H<sub>2</sub>O and 1 g of Na<sub>2</sub>S·9 H<sub>2</sub>O or NaHSO<sub>3</sub>·H<sub>2</sub>O in 1000 mL of 1 N HCl. Solution is reusable but should be replaced when etching of specimen takes longer than 30 s.

A4.6.1.2 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.

A4.6.1.3 As an alternative to A4.6.1.2 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.

A4.6.1.4 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.)

A4.6.1.5 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  $1 \times$  magnification.

A4.6.2 Method No. 2—Iron solution value disk.

A4.6.2.1 Examine the specimen after completion of the ISV test (see Annex A5) as it will already be suitably etched.

A4.6.2.2 Estimate tin crystal size same as in Method No. 1.

A4.6.3 Method No. 3—Polarized light.

A4.6.3.1 This is a rapid nondestructive method.

A4.6.3.2 Place the specimen in a beam of polarized light so the beam strikes the surface obliquely.

A4.6.3.3 Examine the reflected light beam through an analyzer. Rotate the analyzer to obtain best definition of tin crystal pattern.

A4.6.3.4 Estimate tin crystal size same as in Method No. 1.

#### A5. METHOD FOR DETERMINATION OF IRON SOLUTION VALUE ON 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 results agree with those obtained by the standard method.

#### A5.1 Scope

A5.1.1 The iron solution test,<sup>5</sup> 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 No. 50, No. 50/25, and heavier electrolytic tin plate (for K-plate, see 3.1.16.2 and J-plate, see 3.1.16.1). It is not applicable to No. 25 and lighter electrolytic tin plate.

#### **A5.2 Summary of Method**

A5.2.1 The iron solution test involves the colorimetric determination of the total amount of iron dissolved when 3.14 in.<sup>2</sup> of tin plate surface area are exposed for 2 h at  $80 \pm 1^{\circ}F$  to 50 mL of a mixture of dilute sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), and ammonium thiocyanate (NH<sub>4</sub>SCN). The amount of iron dissolved expressed as micrograms is arbitrarily called the *iron solution value (ISV)*.

# **A5.3 Apparatus**

- A5.3.1 *Cabinet, Room, or Other Means* of maintaining 80  $\pm$  1°F during the test run.
- A5.3.2 *Test Vessels*, round, tall-form, wide-mouth, 8-oz glass bottles with 63-mm diameter plastic caps.
- A5.3.3 *Gaskets* made from ½16-in. thick vinyl sheeting. Gaskets have 2-in. inside diameter (ID) and 2.42-in. outside diameter (OD).
- A5.3.4 *Burets*—Two 25-mL automatic filling rapid dispensing burets.
- A5.3.5 Equipment for Cathodically Cleaning Test Specimens—The power source should be capable of supplying 1 to 1½ A per test specimen (4-in.<sup>2</sup> disk). A stainless steel beaker or tank is recommended as the cleaning vessel as it may also serve as the anode.
  - A5.3.6 Spectrophotometer and Cuvettes.

## **A5.4 Reagents and Materials**

- A5.4.1 *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.<sup>6</sup> 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.
- A5.4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A5.4.3 For Cleaning Test Specimen:

A5.4.3.1 Acetone.

A5.4.3.2 Sodium Carbonate Solution (Na<sub>2</sub>CO<sub>3</sub>) (0.5 %).

A5.4.4 For Iron Solution Test:

A5.4.4.1 Ammonium Thiocyanate (NH<sub>4</sub>SCN) Solution (Iron-Free).

A5.4.4.2 Hydrogen Peroxide Solution (H<sub>2</sub>O<sub>2</sub>) (30 %).

A5.4.4.3 Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>) (2.18 N).

A5.4.5 For Calibration:

A5.4.5.1 Iron Wire, Analytical.

A5.4.5.2 Sulfuric Acid ( $H_2SO_4$ ) (10 N).

## A5.5 Procedure

A5.5.1 Test Solutions:

A5.5.1.1 Prepare a 3 % solution of  $H_2O_2$  by dilution of the 30 % grade.

A5.5.1.2 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.

A5.5.1.3 Prepare a stock solution of NH<sub>4</sub>SCN (40 g/L) and connect the stock bottle to the other buret.

A5.5.2 Sample Preparation:

A5.5.2.1 The specimen consists of a flat-circular piece of tin plate  $2.257 \pm 0.001$  in. in diameter. This is equivalent to 4 in.<sup>2</sup> 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.

A5.5.2.2 Cathodically clean the specimen in 0.5 % Na<sub>2</sub>CO<sub>3</sub> 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.

A5.5.2.3 Rinse the specimen successively in tap water and distilled or deionized water. Dry in acetone vapors. Do not touch the test surface.

A5.5.3 Iron Solution Test:

A5.5.3.1 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 ½16 in. of cap thread may be removed by machining on a lathe.)

A5.5.3.2 Place the vinyl gasket over the specimen, seating it so that the gasket lies flat and holds the specimen firmly in place.

A5.5.3.3 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.

A5.5.3.4 Affix the cap with specimen and gasket to the test vessel. Secure tightly. Invert the vessel immediately and let stand for 2 h at 80°F without agitation or vibration.

A5.5.3.5 Provide one extra test vessel for each run. Add 25 mL each of the two stock solutions, cover with a plastic cap, but do not invert. This mixture will act as a blank during the calculation of the iron solution value.

A5.5.3.6 After 2 h, swirl the liquid once, turn the vessel upright, and remove cap, gasket, and specimen immediately. Repeat for all test vessels in the run. Remove cap from the blank.

Note A5.1—**Caution:** A small amount of hydrogen cyanide gas may be liberated during test run. Be sure the vessels are opened in a well-ventilated room or preferably under a hood.

A5.5.3.7 Add 1 mL of 3% H<sub>2</sub>O<sub>2</sub> to each test vessel including the blank. Add the peroxide just before transferring the liquid in each test vessel to the cuvette. (See A5.8.)

A5.5.3.8 Set the spectrophotometer at 485 nm. Zero the instrument by setting the scale for  $100\,\%$  transmission on distilled or deionized water.

A5.5.3.9 Transfer a portion of the liquid to a cuvette and record the optical density or percent transmission, depending on the original calibration. If the instrument has been fitted with an *ISV* scale, read the *ISV* directly.

A5.5.3.10 Rinse the vessels successively with tap water and distilled or deionized water as soon after test as possible. Quick rinsing minimizes the buildup of a yellow sulfur deposit. Periodically the vessels should be cleaned with sulfuric acid-dichromate cleaning solution to remove the deposit.

A5.5.3.11 Soak gaskets for a few minutes in dilute  $H_2SO_4$ , rinse with distilled or deionized water and hang on a glass rod to dry. (Heating the  $H_2SO_4$  to around 150°F during the soaking of the gaskets assists in removal of any iron compounds and helps retain resiliency of the gaskets.)

# **A5.6** Calibration

A5.6.1 The spectrophotometer and cuvettes should be calibrated with standard solutions containing known amounts of iron. A typical calibration might proceed as follows:

A5.6.1.1 Prepare standard iron solution by dissolving 0.100 g of iron wire in 100 mL of 10  $N\,H_2SO_4$ . Dilute with distilled water to 1000 mL in a volumetric flask.

A5.6.1.2 Using aliquots, also prepare 10+1 and 100+1 dilutions of this solution. These three will give standard iron solutions containing 0.1, 0.01, and 0.001 mg Fe/mL, respectively.

A5.6.1.3 Mix 25 mL of the  $H_2SO_4$ - $H_2O_2$  and 25 mL of the NH<sub>4</sub>SCN stock solutions as in A5.5.3.3. Add 1 mL of the standard iron solution containing 0.1 mg Fe/mL. Repeat using the 0.01 and 0.001 mg Fe/mL standard iron solutions. The three mixtures will give iron solution values (*ISV*) of 100, 10, and 1, respectively.

A5.6.1.4 Measure the optical densities at a wavelength of 485 nm in a spectrophotometer and plot these against the *ISV*'s. The *ISV* is directly proportional to optical density. A typical calibration curve using a spectrophotometer and 19 by 150-mm round cuvettes is shown in Fig. A5.1. A full logarithmic plot is used to enhance the definition at the low end of the *ISV* scale

where most readings occur. Once the calibration is established the simplest procedure is to make and attach a scale to the spectrophotometer, which reads directly in *ISV*.

#### A5.7 Calculation

A5.7.1 If the spectrophotometer does not have an *ISV* scale, determine the *ISV* from the calibration curve for each sample including the blank.

A5.7.2 Subtract the blank *ISV* from each of the scale *ISV* readings or from the *ISV*'s obtained in A5.7.1. This is the true *ISV*.

#### **A5.8 Interferences**

A5.8.1 *Leakers*—Sometimes leaks will occur. These are generally discovered when the vessels are opened at the end of the test. If a leak has occurred, a local spot of iron-tin alloy or bare steel will show near the edge of the specimen or etching may be seen on the reverse side of the disk, or both. Sometimes the leak will not affect the *ISV*; at other times it may cause an extremely high *ISV*. Any test showing a leak or other irregularity should be discarded and a retest made.

A5.8.2 Detinning or etching of the tin plate disk by any other cause than the normal exposure to the reagents may cause erroneously high results. Such detinning or etching could be caused by, (1) inadvertent too long anodic flash or too long exposure to  $Na_2CO_3$  in sample preparation (see A5.5.2.2), (2) agitation, swirling, or vibration of test vessel during 2-h test time, (3) leakers, and (4) rise in temperature.

A5.8.3 Fading of the red ferric thiocyanate complex color may occur due to decomposition of the complex by excess peroxide. Delay between the adding of the peroxide at the end of the test and the reading of the optical density should be avoided. Also care should be exercised not to add more than the 1 mL of peroxide.

# **A5.9 Precision**

A5.9.1 The principal source of error in reproducibility of test results is variation in the tin plate itself. Variation may occur across the rolling width and along different portions of the same coil of tin plate. Generally plate with low *ISV* has much less variation than plate with high *ISV*. Plate Lots B, D, E, and F as follows show the type of variation that can occur when replicates of a given plate lot with all specimens closely adjacent to each other are run at one time. Plate Lots A and C show the type of variation that can occur when replicates of a given plate lot are run singly in tests over a long period of time.

	Iron Solution Values, mg Iron									
Plate Lot	Average	Range	Standard Deviation	Number of Samples						
Α	4.4	2-8	1.6	56						
В	9.4	8-19 <sup>A</sup>	1.9	36						
С	34	19-55	7.2	47						
D	36	25-42	5.8	8						
E	87	72-95	6.5	8						
F	97	74-120	14	8						

<sup>&</sup>lt;sup>A</sup> 35 of 36 samples in range from 8 to 12.

A5.9.2 It is recommended that at least one specimen from a lot of plate with known *ISV* be included in each test run as a

control. Preferably two controls should be used; one with low *ISV* (2-10) and one with a higher *ISV* (20-40).

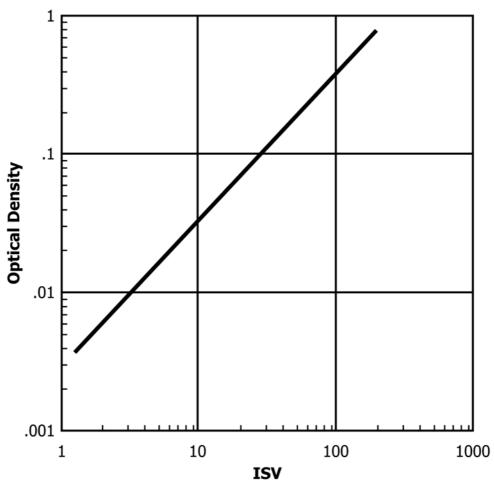


FIG. A5.1 Typical Iron Solution Value Calibration Curve

# A6. METHOD FOR ALLOY-TIN COUPLE TEST FOR ELECTROLYTIC TIN PLATE INTRODUCTION

The method described in this specification for conducting the alloy-tin couple test is one of several possible methods to obtain the same test result. It is not intended that other methods or variants of this method be precluded. Variation in apparatus, reagents, test media, and procedure from those specified may be employed for control purposes by the consumer or the supplier provided satisfactory results are obtained, which correlate with the specified method.

## A6.1 Scope

A6.1.1 The alloy-tin couple test,<sup>7</sup> also called the *ATC* 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 No. 50, No. 50/25, and heavier electrolytic tin plate (for K-plate, see 3.1.16.2). It is not applicable to No. 25 and lighter electrolytic tin plate.

## A6.2 Summary of Method

A6.2.1 The ATC test is an electrochemical procedure, which involves measuring the current flowing between a pure tin electrode and an electrode consisting of a piece of tin plate from which the free (unalloyed) tin has been removed to expose the iron-tin alloy. The measurement is made after 20-h exposure of the electrodes in a medium consisting essentially of deaerated aged grapefruit juice.

# A6.3 Apparatus

- A6.3.1 Constant-Temperature Cabinet or Room (80  $\pm$  1°F).
- A6.3.2 Test Cell (Fig. A6.1):
- A6.3.2.1 *Borosilicate Glass Test Cell*, approximately 1-mL capacity.
- A6.3.2.2 *Polymethylmethacrylate Plastic Cover* for test cell approximately ½ in. thick drilled with 5/8-in. diameter holes to accommodate cell elements.
- A6.3.2.3 Polychloroprene or Similar Synthetic Rubber O-Ring Gasket to effect seal between glass vessel and plastic cover or equivalent method to effect gas-tight seal.
- A6.3.2.4 Silicone Rubber <sup>1</sup>/<sub>4</sub> -in. Thick Grommets to act as gas-tight holders for cell elements inserted through the plastic cover.
  - A6.3.3 Magnetic Stirrer.
  - A6.3.4 Low-Resistance, High-Sensitivity Galvanometer.
- A6.3.5 *Potentiometer* to measure the tin electrode potential. Any high-impedance voltage-measuring device such as a pH meter with a 0 to 1300-mV scale is satisfactory.

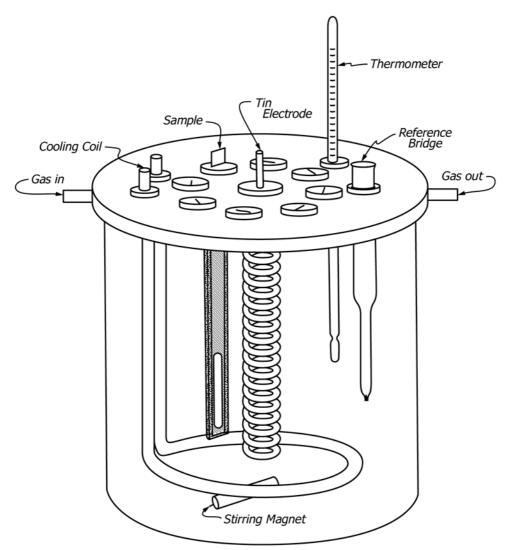


FIG. A6.1 Test Cell Used in the ATC Test

<sup>&</sup>lt;sup>7</sup> Kamm, G. G., Willey, A. R., Beese, R. E., and Krickl, J. L., "Corrosion Resistance of Electrolytic Tin Plate, Part 2, The Alloy-Tin Couple Test—A New Research Tool", *Corrosion*, Vol 17, 1961, p. 84.

A6.3.6 *Calomel Reference Electrode* (Either saturated or 0.1 *N* is satisfactory).

A6.3.7 *Power Source* capable of supplying variable d-c voltage for use in sample preparation (cathodic cleaning 10-V dc and tin stripping 0.4-V dc reducible to 0.2 V).

A6.3.8 *Various Electrical Components* such as plugs, jacks, switches, and resistors to permit construction of circuit depicted in schematic diagram (Fig. A6.2).

A6.3.9 (Optional) Special Die for applying microcrystalline wax to mask off known areas on test specimen.

# A6.4 Reagents and Materials

A6.4.1 *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.<sup>6</sup> 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.

A6.4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A6.4.3 Test Medium:

A6.4.3.1 Distilled Water or Deionized Water of equal purity.

A6.4.3.2 Ethanol, Denatured (70 % volume).

A6.4.3.3 Frozen Concentrated Grapefruit Juice.

A6.4.3.4 Nitrogen Gas (High-Purity Oxygen-Free Dry Tank Nitrogen).

A6.4.3.5 Potassium Sorbate.

A6.4.3.6 Pure Tin Wire (approximately ½-in. diameter).

A6.4.3.7 Sodium Hydroxide Solution (NaOH) (10 %).

A6.4.3.8 Stannous Chloride Solution (SnCl<sub>2</sub>·2 H<sub>2</sub>O).

A6.4.4 Sample Preparation:

A6.4.4.1 Acetone.

A6.4.4.2 *Microcrystalline Wax* (140 to 145°F melting point).

A6.4.4.3 Polymethylmethacrylate Plastic Strips ( $\frac{1}{16}$  by  $\frac{9}{16}$  by  $\frac{3}{4}$  in.).

A6.4.4.4 Sodium Carbonate Solution (Na<sub>2</sub>CO<sub>3</sub>) (0.5 %).

A6.4.4.5 Sodium Hydroxide Solution (NaOH) (5 %).

# A6.5 Test Specimen

A6.5.1 The specimen consists of a piece of tin plate cut  $\frac{1}{2}$  by  $\frac{4}{2}$  in. with the long dimension transverse to the rolling direction.

A6.5.2 Eight test specimens can be accommodated at one time in the apparatus described above. Only the number of specimens to be included in one run should be prepared at one time.

A6.5.3 Details of sample preparation are given in A6.7.

# A6.6 Preparation of Apparatus (Fig. A6.1)

A6.6.1 Drill 5%-in. diameter holes in ½-in. thick plastic cover to accommodate eight test specimens, the pure tin anode, a thermometer, and cooling coil. The reference electrode bridge can be inserted through one of the test specimen openings during potential measurement. Drill smaller diameter holes into the cover to accommodate gas inlet and outlet tubes.

A6.6.2 Cut stoppers or grommets from ½-in. thick silicone rubber to fit snugly in 5%-in. diameter openings. Cut holes or slits in stoppers and grommets to hold various cell elements.

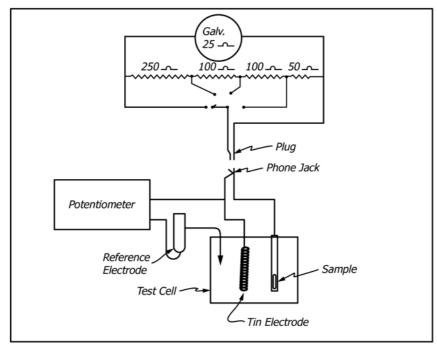


FIG. A6.2 Schematic Diagram of ATC Test Circuit

Boil all rubber parts including O-ring gasket in 10 % NaOH solution for 5 min and rinse thoroughly before use in distilled or deionized water.

A6.6.3 Thoroughly clean test cell and its components and finally rinse them in ethanol just prior to use to guard against mold and yeast growth in test medium.

A6.6.4 Fit silicone rubber parts, cooling coil, thermometer, and gas tubes into the cover. Do not insert pure tin anode or test specimens at this time.

A6.6.5 Form  $\frac{1}{8}$ -in. diameter pure tin wire into a loosely wound coil to give a total surface area of approximately 100 cm<sup>2</sup>. Cathodically clean in 0.5 % Na<sub>2</sub>CO<sub>3</sub> solution, rinse in tap water and in acetone.

#### A6.7 Procedure

A6.7.1 Test Medium:

A6.7.1.1 Place a polytetrafluoroethylene-covered magnetic stirring bar in the bottom of the test cell.

A6.7.1.2 In a separate vessel, dilute frozen concentrated grapefruit juice 3+1 with distilled or deionized water, add preservative potassium sorbate to give concentration of 0.5 g/L, deaerate by heating to boiling, and transfer to the test cell and age for not less than two days. Leave approximately ½-in. headspace. Turn on the magnetic stirrer.

A6.7.1.3 Assemble the plastic cover to the test cell with an O-ring or by other leak-proof seal. Begin the flow of nitrogen through the headspace. Bubble nitrogen through distilled or deionized water before entering the test cell in order to minimize evaporation of test medium. Maintain a slight positive pressure in the cell during actual test run by bubbling nitrogen from the gas outlet tube through 1 or 2 in. of water or ½ in. of dibutyl phthalate.

A6.7.1.4 Allow the transferred hot juice to cool for 5 min; then start cold water through the cooling coil. This minimizes settling of the pulp. Continue cooling until the test medium reaches 80°F.

A6.7.1.5 Insert the cleaned pure tin anode into the test cell. A6.7.1.6 Add SnCl<sub>2</sub>·2H<sub>2</sub>O to produce a concentration of 0.190 g/L. This yields a Sn<sup>+</sup> + concentration of 100 ppm. Continue stirring for 5 or 10 min to make sure the SnCl<sub>2</sub>·2H<sub>2</sub>O has been dissolved.

A6.7.1.7 Discontinue stirring.

A6.7.1.8 Measure the potential of the tin electrode with a high-impedance device such as a pH meter, using calomel reference electrode. The potential of the tin anode should be -615 mV against a saturated calomel electrode or -705 mV against a 0.1 N calomel electrode.

A6.7.2 Test Specimen:

A6.7.2.1 Degrease the specimen in acetone and allow to dry. A6.7.2.2 Clean the specimen cathodically in 0.5 % Na<sub>2</sub>CO<sub>3</sub> solution (carbon anode) using a current density of approximately 25 mA/cm<sup>2</sup>. A 10-V d-c power source with a polarity reversing switch and the following sequence of test specimen polarity is suggested: 2 s cathodic, 0.1 s anodic, 2 s cathodic, 0.1 s anodic, 2 s cathodic. The two short anodic flash treatments enhance the ability of the cathodic treatments to remove oxides and impurities from the surface and secure

absence of water break on the test specimen. Rinse the specimen in tap water, distilled water, and acetone and allow to dry.

A6.7.2.3 Detin the specimen electrolytically in a 5 % NaOH solution at room temperature. The specimen is the anode and a piece of stainless steel is the cathode. The area of the stainless steel cathode should be 5 to 10 times as large as the area being detinned in order to give a high anode current density and hence rapid detinning. Carry out the detinning at a constant 0.40-V dc maximum (in this method a 0.1- $\Omega$  resistor is placed in parallel with the detinning circuit to assure constant voltage). As detinning nears completion, it is possible a small area or a few isolated spots will be slow to detin. Reducing the voltage to 0.20 V speeds up the detinning of these last few spots. For convenience, detin several specimens simultaneously all connected in parallel to the power source. When this is done it is usually necessary to reduce the voltage to 0.20 V only for the last sample remaining in the detinning set up. Remove the specimen from the detinning solution with the power on to prevent reversal of the current and replating of tin as a result of the primary cell effect. Do not leave the detinned specimen in the detinning bath longer than 5 min. The electrolyte and the detinning procedure have been so chosen to remove completely all the free (unalloyed) tin and to prevent any attack whatever on the iron-tin alloy layer. Rinse specimen sequentially in tap water, distilled water, and acetone and allow

A6.7.2.4 Mask the specimen with hot microcrystalline wax to expose a given test area. This may be done by hand brushing or by mechanical means provided the test surface is not damaged or contaminated in the process. Area variations between 0.5 and 4.0 cm<sup>2</sup> do not affect the *ATC* measurement. It is strongly recommended that an area of 2.3 cm<sup>2</sup> be used in the test. A die that produces an outline of wax exposing 2.3 cm<sup>2</sup> is available (A6.3.9). After the test area has been outlined, manually wax the specimen to a thin plastic backing (A6.4.4.3) making certain all edges and surfaces other than the test area are covered.

#### A6.7.3 Current Measurement:

A6.7.3.1 Connect the test specimen to the tin anode electrically before inserting the specimen in the test cell to assure continuous galvanic protection of the alloy surface (for the same reason, refer to Fig. A6.2 and note that the phone jacks are the *shorting* type). All test specimens are coupled to the single tin anode.

A6.7.3.2 After 20 h, measure the current flowing between the tin anode and each individual specimen with a low-resistance, high-sensitivity galvanometer (A6.3.4). The test cell must be free from vibration during the time the specimens are in the cell.

A6.7.3.3 Include at least one test specimen with known *ATC* value in each run in each cell to act as a control for that run. Preferably two controls should be used: one with a known low *ATC* value and one with a known high *ATC* value.

A6.7.3.4 Use a given batch of aged juice for repeated test runs for a period of about 3 to 4 weeks. Make a fresh batch sooner if there are signs of mold growth or fermentation.



#### A6.8 Calculations

A6.8.1 Divide the current flowing between the electrodes by the area of the exposed alloy on the test specimen measured in square centimetres. Report the *ATC* in microamperes per square centimetre.

#### A6.9 Hazards

- A6.9.1 It is important to maintain oxygen-free conditions in the cell. During insertion and removal of the test specimen in the cell increase the nitrogen flow somewhat to prevent entry of air. Air causes increased *ATC* values and reduces differences between good and poor plate.
- A6.9.2 Take care to avoid vibration during the test run. Do not bump or disturb the electrodes before taking current measurements.

- A6.9.3 Different batches of juice will vary slightly in corrosivity or pH or both. This could affect the potential of the tin anode. Regardless of the original potential in a given batch of juice, the addition of 100 ppm Sn<sup>++</sup> shifts the potential approximately 50 mV in the cathodic (positive) direction.
- A6.9.4 Temperature of microcrystalline wax during masking should be sufficiently high to assure good adhesion to the test specimen but not so high as to run and distort the test area.
- A6.9.5 Reliable *ATC* data depend to a large extent on proper test specimen preparation. Once preparation has begun the test area should not be touched, scratched, or otherwise contaminated in any way.

## A7. METHODS FOR DETERMINATION OF TOTAL SURFACE OIL ON TIN MILL PRODUCTS

# A7.1 Scope

A7.1.1 Two test methods for the determination of the total extracted oil on the surface of tin mill products are described as follows:

Test Method Sections
A—Solvent Extraction (Referee A7.3 to A7.11
Method)
B—Ellipsometry A7.12 to A7.19

# A7.2 Significance and Use

A7.2.1 The amount of surface lubricating oil on the surfaces of tin mill products is critical and can be cause for users complaint. Insufficient lubricant can contribute to poor sheet mobility and poor lithography; excessive lubricant can contribute to eyeholing or dewetting of certain organic coatings.

# METHOD A—DETERMINATION OF TOTAL SURFACE OIL ON TIN MILL PRODUCTS BY SOLVENT EXTRACTION

# A7.3 Summary of Method

A7.3.1 The oil on the surface of the strips of plate is removed with boiling chloroform or equivalent. The chloroform or equivalent is evaporated to dryness and the residue is weighed.

### A7.4 Apparatus

- A7.4.1 Slotted Mandrel with handle for coiling the strips.
- A7.4.1.1 A ½-in. diameter slotted mandrel is used for high-temper materials and a 1-in. diameter slotted mandrel is used for low-temper plate.

# A7.5 Reagents and Materials

A7.5.1 *Chloroform* (CHCl<sub>3</sub>), distilled reagent grade or equivalent.

#### A7.6 Hazards

A7.6.1 Chloroform vapors present a potential health hazard. The cleaning of equipment, extraction, and evaporation of the chloroform should be done in an exhaust hood.

# A7.7 Test Specimen

A7.7.1 The samples are generally sheets of plate such as used for can making. The sample sheets should be transported between two protection sheets and the edges covered with masking or equivalent tape. The four edges of the test sheets should be trimmed to remove possible contaminant of the tape adhesive.

#### A7.8 Preparation of Apparatus

- A7.8.1 Clean the shears for cutting the plate into strips, coiling mandrel, pliers, and forceps with chloroform or equivalent.
- A7.8.2 The glassware must be rinsed with boiling chloroform or equivalent.
  - A7.8.3 Wear clean white cloth gloves when handling plate.

# A7.9 Procedure

- A7.9.1 Cut the sample of plate at least 500 in. <sup>2</sup> (preferably 1000 in. <sup>2</sup>) into 2-in. wide strips.
- A7.9.2 Determine the exact area of plate (length by width by number of strips).
- A7.9.3 Coil strips using the coiling mandrel by holding one end of the strip with pliers. Insert the other end in the slot of the mandrel. Coil the strips around the mandrel tightly using the pliers to maintain tension.
- A7.9.4 Heat two 250-mL beakers of chloroform or equivalent to boiling. Using forceps dip the coils 4 or 5 times in one beaker and then rinse similarly in the second beaker. After all the coils have been extracted, filter the chloroform or

equivalent, while hot, through filter paper into a 500-mL Erlenmeyer flask. Boil off the chloroform or equivalent to a volume of approximately 10 mL. Transfer this to a previously cleaned, dried, and weighed 10-mL beaker. While the chloroform or equivalent is boiling from the small beaker, rinse the Erlenmeyer flask two or three times with small portions of chloroform or equivalent and add each rinsing to the 10-mL beaker. When nearly all the chloroform or equivalent has evaporated from the 10-mL beaker, place the beaker in an oven at 105°C for 10 min, cool in a desiccator, and reweigh. Make a blank determination using a similar volume of chloroform or equivalent. The blank should not exceed 0.0002 g.

#### A7.10 Calculations

A7.10.1 Calculate the weight of oil per base box as follows: weight of oil per base box,  $g = (W \times 31360)/A$ 

where:

W = weight of oil, g, A = area of sample, in.<sup>2</sup> and 31 360 = area of base box, in.<sup>2</sup>

### A7.11 Precision and Bias

A7.11.1 Make all weighings to the nearest 0.0001 g.

# METHOD B—DETERMINATION OF TOTAL SURFACE OIL ON TIN MILL PRODUCTS BY ELLIPSOMETRY

#### A7.12 Scope

A7.12.1 This method covers the determination of the total oil on the surface of tin mill products by ellipsometry.

## A7.13 Summary of Method

A7.13.1 The basic ellipsometer is a highly accurate optical instrument that measures the change in the polarization state, referred to as the ellipticity, of light reflected from a surface. The change in ellipticity before and after degreasing is the measured variable and related to the oil layer thickness.

## A7.14 Apparatus

A7.14.1 Ellipsometers measure the change in polarization state of light reflected from a surface and provide information about the optical properties of thin films on that surface. Certain ellipsometers are specially designed for measurement of oils on tin mill products.<sup>8</sup> Ellipsometers not specifically designed for measuring oils on tin mill oils could be calibrated to measure tin mill oils; however, that is beyond the scope of this method.

# **A7.15 Reagents and Materials**

A7.15.1 Non-residue forming degreasing solvent such as *Trichloroethylene* or *1-Bromopropane*.

#### A7.16 Hazards

A7.16.1 Trichloroethylene and 1-Bromopopane vapors present a potential health hazard. An exhaust hood is required for operation of the ellipsometer.

#### **A7.17 Preparation of Apparatus**

A7.17.1 It is necessary to set up the instruments' various gain and sensitivity adjustments for different substrate surfaces (for example, varying brightness or tinplate versus TFS). Once set up is achieved for a sample type, the settings can be recorded and used for future testing of the same sample type. Set up does not affect calibration, but instead, adjusts measurement circuitry for best overall performance. Newer models automatically adjust these settings. The following set-up procedure for older models without the automatic adjustment has been found to provide the best repeatability, and is provided as an example.

A7.17.2 The instrument has a High Voltage Power Supply Control located above the Operator Control Panel. This control is marked 5-10. Each full setting (5-10) is  $\times$  100. For example: 5=500V with a ½ setting  $\times$  50 Example 5.5 = 550V. At this time the multiplier should be set to "0". The Panel Meter should read "0". Set the Reading switch to Base.

A7.17.3 Install a typical sample of the type to be measured with the rolling direction in the vertical position.

A7.17.4 Set high voltage control to 500V. Set the NULL control to "10" clockwise. Set the MULTIPLIER Control to "1". The panel Meter should have moved someplace above "0".

A7.17.5 Turn high voltage UP just until full scale meter deflection is observed on the Panel Meter. Note the High Voltage setting and add 150V-250V for the final voltage setting. For example: Full meter deflection 550V. Final setting is 550+250 or 800V. Even though the meter is past full scale, it is protected by internal circuitry. This is the correct meter position for the beginning or after the end of a cycle. If the final voltage setting needs to be higher than 1000V, the Multiplier Control should be reset higher, and then repeat set up.

A7.17.6 Push start button. (Leave motor switch off). Use the inch switch to jog the Amp Meter needle down until it starts coming back up. This should be near 10 microamps. Turn the NULL control counter clockwise until the needle reads between 0-5 microamps.

A7.17.7 Turn the motor switch on. The ellipsometer will finish running its cycle. Turn the Clean Switch off. Set the Reading Switch to OIL. Press the Manual Start Button. You should get a reading between 9.98 and 0.02. The ellipsometer is now set up. As long as the needle deflection is within the parameters of <.10 to .90 >, no changes are necessary. No adjustments are to be made after the unit is set up, unless the surface of the product changes.

#### A7.18 Procedure

A7.18.1 Samples for oil weight determinations are obtained by stamping disks  $2.257 \pm 0.001$  in. [57.33  $\pm 0.02$  mm] in diameter, which is equivalent to 4-in.<sup>2</sup> [25.81 cm<sup>2</sup>] per side.

<sup>&</sup>lt;sup>8</sup> The sole source of supply of the apparatus known to the committee at this time is Donart Electronics, Inc., P.O. Box 27 McDonald, PA 15057. 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, <sup>1</sup> which you may attend.

A7.18.2 Samples should be handled on the edges and free of scratches, fingerprints and other contaminants. A sample that has been dropped or touched on the surface should be discarded.

A7.18.3 Insert the sample into the magnetic sample holder with the rolling direction in the vertical position.

A7.18.4 Verify that the Null control, Multiplier, and High Voltage settings are set up correctly as described in A7.17.

A7.18.5 Set the Reading Switch on the Operator Control Panel to the Oil position.

A7.18.6 Set the Clean Auto/Off Switch to the Auto position.

A7.18.7 Depress the Manual Start button to initiate the measurement. At the completion of the measurement and cleaning cycles the oil weight can be read directly from the digital display in grams/base box.

A7.18.8 If the opposite side of the sample is to be measured rotate the sample and repeat step A7.18.7. It is advisable that one side per sample be measured to prevent spray-over to the opposite side during the cleaning cycle on smaller sample disk diameters.

A7.18.9 If significant time is to pass between procurement and testing, a sufficiently large sheet should be cut and

sandwiched between two coversheets and sealed with tape to prevent oil evaporation. The test samples can then be stamped from the sheet prior to testing.

A7.18.10 The in-factory calibration of the ellipsometer (degrees of analyzer rotation/unit of oil weight per surface area unit) is for melted (reflowed) tinplate with a bright stone finish. It is the responsibility of the instrument user to develop a correction factor for different coatings, or finishes, or both, as needed or as agreed upon by manufacturer and purchaser.

#### A7.19 Precision and Bias

A7.19.1 Four laboratories measured the repeatability using a different coil for each laboratory. The standard error was 0.005 g/bb for 30 samples with oil weights ranging from 0.1 to 0.25 grams/base box. A reproducibility study could not be conducted due to volatilization of the oil.

A7.19.2 The in-factory calibration of the ellipsometer (degrees of analyzer rotation/unit of oil weight per surface area unit) is for melted (reflowed) tinplate with a bright stone finish. It is the responsibility of the instrument user to develop a correction factor for different coatings, or finishes, or both, as needed or as agreed upon by manufacturer and purchaser.

#### A8. DETERMINATION OF CHROMIUM ON TIN PLATE BY THE DIPHENYLCARBAZIDE METHOD

#### A8.1 Scope

A8.1.1 This method<sup>9</sup> covers the determination of chromium on tin plate with the use of diphenylcarbazide.

## **A8.2** Reagents and Materials

A8.2.1 *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.<sup>6</sup> 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.

A8.2.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A8.2.3 Chromate, Standard Solution A (1 mL = 0.5 mg Cr)—Dissolve 1.410 g of  $K_2Cr_2O_7$  in water and dilute to 1.0 L.

A8.2.4 *Chromate, Standard Solution B* (1 mL =  $10.0 \mu g$  Cr)—Pipet 20 mL of the chromate Standard Solution A into a 1.0-L volumetric flask and add water to 1.0 L.

A8.2.5 Diphenylcarbazide Reagent—Add 10.0 mL of acetone, 10.0 mL of 95 % ethyl alcohol, and 20.0 mL of  $\rm H_3PO_4$ 

(85 % acid diluted with an equal volume of water) to 0.25 grams of diphenylcarbazide powder.

A8.2.6 Hydrochloric Acid (HCl) (sp gr 1.19).

A8.2.7 Potassium Permanganate, Saturated Solution (KMnO<sub>4</sub>).

A8.2.8 Sodium Hydroxide (1.0 N)—Trisodium Phosphate (5%) Solution—Dissolve 40.0 g of NaOH and 50.0 g of Na<sub>3</sub>PO<sub>4</sub> in water and dilute to 1.0 L.

A8.2.9 Sulfuric Acid (1+3)—Add 100 mL of  $H_2SO_4$  (sp gr 1.84) slowly and with stirring to 300 mL of water.

# **A8.3 Procedure**

A8.3.1 Use for analysis a sample having 8.0 in. <sup>2</sup> of surface area (one 4-in. <sup>2</sup> disk). If both sides of the sample are to be stripped, slightly bend the disk through the center so it will not lie entirely flat. If only one side of the sample is to be stripped, hold the disk tightly against a rubber stopper. The stopper should be slightly larger in diameter than the disk and grooved to allow vacuum from a tube in the center to be applied to most of the surface of the disk. Leave intact a band approximately ½ in. wide at the perimeter of the stopper.

A8.3.2 Place the sample in a 250-mL beaker, add 25 mL of NaOH·Na $_3$ PO $_4$  solution, and heat to boiling. Boil for  $1\frac{1}{2}$  min. Transfer the solution to another 250-mL beaker, washing disk and beaker once with water. Add 25 mL of  $H_2$ SO $_4$  (1+3) to the original beaker and sample, heat to boiling, and boil 1 min.

<sup>&</sup>lt;sup>9</sup> Furman, N. H., "Chromium in Acid Solution with Diphenylcarbazide," *Scott's Standard Methods of Chemical Analysis*, Vol 1, 1962, pp. 357–359.



Transfer the acid solution to the beaker containing the alkaline stripping solution, washing sample and beaker with two small portions of water. If both sides of the disk are being stripped, it is necessary to swirl the beaker continually over the flame while the  $\rm H_2SO_4$  is boiling. This is necessary to keep the surface completely wetted and strip all of the chromium from the surface of the tin plate.

A8.3.3 Heat the sample solution to boiling and add 1 to 2 drops of saturated  $KMnO_4$  solution. This amount is usually sufficient to maintain a pink color. Boil 3 to 4 min for complete oxidation of chrome. Add 5 drops of HCl (sp gr 1.19) to the sample and continue to boil until all pink color is dispelled. More acid may be used if needed. The beaker should be covered when boiling to avoid any loss that may be caused by spattering.

A8.3.4 Transfer to a 100-mL volumetric flask and cool to approximately 70°F in a water bath. Add 3.0 mL of diphenyl-carbazide reagent, make to mark with distilled water, and mix.

A8.3.5 Determine optical density, within 30 min after the addition of diphenylcarbazide reagent to the sample, at 540 nm.

A8.3.6 A reagent blank and a standard including all solutions used in treating a sample should be carried along with each set of samples.

# **A8.4** Calibration of Spectrophotometer

A8.4.1 Add to 250-mL beakers duplicate 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0-mL aliquots of the chromate Standard Solution B and carry through the entire procedure for a sample. Also run reagent blanks in duplicate.

A8.4.2 Calculate a constant, K, for the instrument as follows:

$$K = (\mu g Cr)/(O.D._1 - O.D._2)$$

where:

 $O.D._1$  = optical density for the standard, and

 $O.D._2$  = optical density for the blank.

## A8.5 Calculation of Chromium on Tin Plate

A8.5.1 Report chromium on tin plate as micrograms of chromium per square foot of surface area, as follows:

Cr, 
$$\mu g/ft^2 = [144 \ K (O.D._1 - O.D._2)]/A$$

where:

K = constant for spectrophotometer and cell used to

determine optical density,

 $O.D._1$  = optical density of sample,

O.D.<sub>2</sub> = optical density of reagent blank, and

A = area of sample used.

#### A9. METHOD FOR AERATED MEDIA POLARIZATION TEST FOR ELECTROLYTIC TIN PLATE

#### INTRODUCTION

The Aerated Media Polarization (AMP) test was originally developed at Weirton Steel Corporation by James A. Bray and J. Robert Smith (see U.S. Patent No. 3,479,256) as a quick, accurate replacement for the Alloy Tin Couple (ATC) test developed by G. Kamm at American Can Company. The AMP test results are obtained in a few minutes as compared to a minimum 20 h for ATC results. This has proven invaluable to tinplate producers who then can make adjustments during actual production.

# A9.1 Scope

A9.1.1 The AMP test is one of four special property tests used to measure certain characteristics of electrolytic tin plate, which affects internal corrosion resistance. The test is applicable to No. 50 (5.6), No. 50/25 (5.6/2.8), and heavier electrolytic tin plate, used for K-plate (for K-Plate see 3.1.16.2).

# **A9.2 Summary of Method**

A9.2.1 This test is an electrochemical procedure that involves measuring the current flowing between a pure tin electrode and an electrode consisting of a piece of tin plate from which the free (unalloyed) tin has been removed to expose the iron-tin alloy. Both electrodes are immersed in grapefruit juice concentrate (GFJ) or its equivalent.

# A9.3 Apparatus

A9.3.1 Test Cell (see Fig. A9.1).

A9.3.2 AMP Analyzer (see Fig. A9.2).

A9.3.3 *d-c Power Supply*, capable of supplying 3 to 4 A at 10 to 12 V and a means of reversing polarity.

A9.3.4 Three Laboratory Hot Plates.

A9.3.5 Crystallizing Dish, 5.9-in. diameter, 2.9-in. depth.

A9.3.6 Watch Glass, 4.5-in. diameter.

A9.3.7 Two 3.4-oz Beakers—low form.

A9.3.8 Two Watch Glasses, for the 13.5-oz beakers.

A9.3.9 *Three 13.5-oz Beakers*—tall form without pouring spout.

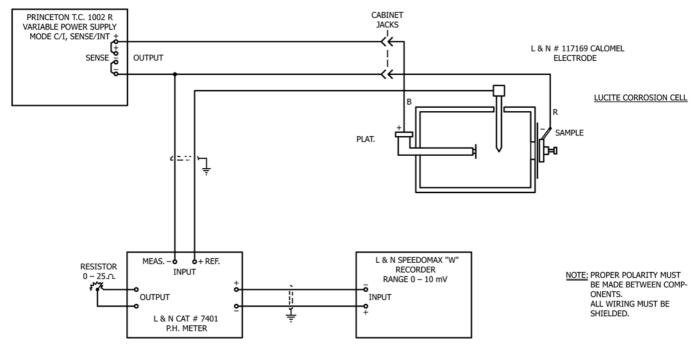


FIG. A9.1 Wiring Schematic for AMP Test

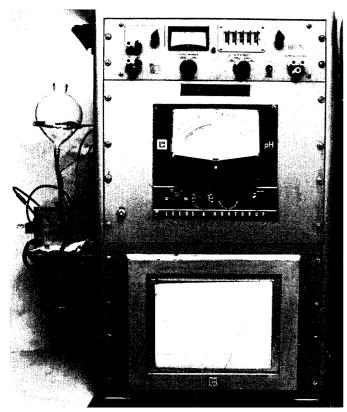


FIG. A9.2 Corrosion Analyzer (AMP)

A9.3.10 Timer or Stopwatch, capable of reading to the nearest second.

A9.3.11 13.5-oz Beaker, stainless steel.

A9.3.12 Levelling Funnel.

A9.3.13 0.875-in. Inside Diameter O-Ring—0.60 in.<sup>2</sup>

#### **A9.4 Reagents and Materials**

A9.4.1 *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.<sup>6</sup> 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.

A9.4.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A9.4.3 Anhydrous Acetone.

A9.4.4 Stripping Solution:

A9.4.4.1 5.0 % (by weight) sodium hydroxide, NaOH, in distilled water.

A9.4.4.2 1.0 % (by weight) potassium iodate  ${\rm KlO_3}$  in distilled water.

A9.4.5 0.5 M Citric Acid—3.2 oz anhydrous citric acid per 14.5 oz of distilled water.

A9.4.6 Potassium Sorbate.

A9.4.7 Frozen Sweetened Grapefruit Juice Concentrate (GFJ).

A9.4.8 Equivalent to GFJ when dissolved in 7.25 oz of distilled water:

A9.4.8.1 0.176 oz citric acid,

A9.4.8.2 0.088 oz sodium citrate,

A9.4.8.3 0.617 oz fructose,

A9.4.8.4 0.882 oz sucrose, or

A9.4.8.5 0.0088 oz potassium sorbate.

A9.4.8.6 Prepare fresh for each occasion of testing.

A9.4.9 *Cleaning Solution*—1% (by weight) sodium carbonate in 21.78 oz of distilled water.

#### A9.5 Test Media

A9.5.1 Six-ounce size cans of grapefruit juice concentrate are thawed, opened, and diluted with 16.9 oz of distilled water per can. The resulting single strength GFJ is filtered under vacuum through silica sand to remove most of the pulpy material.

A9.5.2 To retard spoilage, 0.02 oz of potassium sorbate per can is added to the GFJ. It has been found convenient to prepare the GFJ in five can batches and keep it refrigerated until needed. The GFJ must be prepared at least 24 h prior to usage to allow it to stabilize.

A9.5.3 Enough GFJ to fill the cell, 13.5 oz, is brought to room temperature, 72 to 74°F, and poured into the levelling funnel.

A9.5.4 Under normal conditions, this 13.5-oz aliquot of juice will yield about 200 results before it need be discarded. Worn-out juice is indicated by a marked decrease offset, or both, in the slope of the standard curve.

## **A9.6 Test Specimen**

A9.6.1 Samples, 1½ in. wide by any convenient length, 5 to 10 in., are cut from the tin plate area to be tested. The sample identification is scribed across the top side (or heavy-coated side) of the test piece. The following procedure is recommended for cathodic cleaning to remove the oil film and chemical treatment films without opening pores in the free tin and alloy layers.

# **A9.7 Sample Preparation**

A9.7.1 In a 1 % solution of  $Na_2CO_3$  at room temperature, using mild steel anodes, make the sample cathodic for 3 s, then anodic for 3 s, and finally cathodic for 3 s. Current densities of 20 to 30 A/ft<sup>2</sup> are satisfactory. Rinse the sample thoroughly in distilled water, dip in hot acetone, and dry in air.

Note A9.1—The final current polarity *must be cathodic* for rapid removal of the free tin in subsequent steps.

A9.7.2 Two of the hot plates should be located near a sink where distilled (or deionized) water is plentiful. The third hot plate should be next to the AMP analyzer.

A9.7.3 The crystallizing dish, with the 4.5-in. watch glass placed in it, is filled to a depth of  $1\frac{1}{2}$  in. with stripping solution and placed on one of the two hot plates. The hot plate should be adjusted to maintain the solution at  $105^{\circ}$ F.

A9.7.4 The two 3.4-oz beakers and the 13.5-oz stainless steel beaker are placed on the second hot plate. Fill one of the 3.4-oz beakers with stripping solution to a depth slightly less than  $1\frac{1}{2}$  in. Fill the second 3.4-oz beaker with a 0.5 M citric acid solution to a depth of about 2 in. Fill the 13.5-oz beaker two thirds full of acetone. Adjust this hot plate so that the acetone almost boils.

A9.7.5 The remaining two 13.5-mL beakers are placed on the hot plate located adjacent to the analyzer. One should be two thirds full of acetone. The other is two thirds filled with water for rinsing samples following testing. The samples can then be dried and stored for possible retesting.

A9.7.6 Place a sample upright in the crystallizing dish of stripping solution allowing it to lean against the side of the dish. The submerged 4.5-in. watch glass prevents the sample from falling into the solution. When the last visible trace of free tin dissolves, immediately transfer the sample to the 3.4-oz beaker containing a second stripping solution. After 30  $\pm$  5 s, remove the sample and rinse in running distilled water.

A9.7.7 Place the sample in the beaker containing citric acid. Swish around 4 to 5 times. Remove the sample and rinse very thoroughly in running distilled water. Dry the sample in the first beaker of hot acetone and transfer it to the second beaker of hot acetone. The sample is now ready for insertion into the cell.

#### **A9.8** Aerated Media Polarization Test

A9.8.1 The AMP analyzer settings should be as follows:

Mode Switch: Position 1 (range card switch in recorder set to

Position B)

Zero Adjust Pot: 200 mV (on upper scale)

Current Range: 0.5–5.0 mA
Polarizing Current: 0.800 mA

A9.8.2 Remove the sample from the acetone beaker and allow it to air dry.

A9.8.3 Place the detinned portion of the sample between the O-ring and the follower plate (heavy-coated side to be facing electrode). Tighten the follower just enough to prevent leakage around the O-ring.

Note A9.2—Overtightening will drastically reduce the life of the O-ring and may even cause it to be torn loose from the cell.

A9.8.4 Open the stopcock on the levelling funnel and allow the cell to fill with GFJ. (As the cell fills, it is a good technique to tip the cell slightly to preclude trapping air bubbles on the test area.)

A9.8.4.1 Turn the voltage switch on.

A9.8.4.2 Turn the chart switch on.

A9.8.4.3 As pen point reaches an accented line on the chart paper, turn the current switch on.

A9.8.4.4 After 90 s (3 in.) of chart travel read the end potential as chart divisions (to nearest tenth).

A9.8.4.5 Repeat A9.8.4.1 to A9.8.4.4, in reverse order, turning switches off to discontinue test.

A9.8.5 Lower the levelling funnel to drain the cell, loosen the follower plate, and remove sample. Dry the O-ring and follower plate to remove any droplets of GFJ, which may have spilled during removal of sample. (It has been found convenient to do this by folding 4 to 5 paper towels together and cutting them into 1½-in. widths. These can be inserted between the O-ring and the follower plate to effect the removal of any spillage.) The cell and analyzer are now ready for another test.

# **A9.9 Interpretation of Results**

A9.9.1 In any batch of samples to be run several standards, covering the range of ATC values from about 0.015 to 0.300  $\mu$ a/cm $^2$ , are interspersed. The potential values (chart readings at 90 s) for these are plotted versus their known ATC values on



1.2 cycle ( $E_{90}$ ) by 2 cycle (ATC) log-log paper. The best straight line is then drawn through these points to obtain the standard curve (see Fig. A9.3).

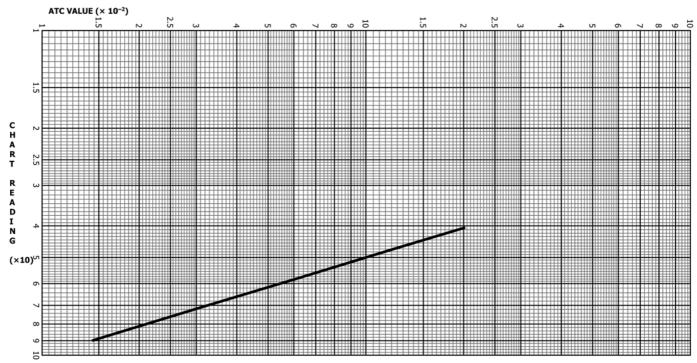


FIG. A9.3 ATC Conversion Chart from Typical Calibration Curve

#### A10. DETERMINATION OF CHROMIUM ON TIN PLATE USING ATOMIC ABSORPTION

#### A10.1 Scope

A10.1.1 The test method covers the determination of chromium on tin plate using atomic absorption.

#### A10.2 Summary of Test Method

A10.2.1 The chromium passivation level on the surface of tin plate is dissolved into, solution using concentrated hydrochloric acid. This solution is diluted to a specific volume and aspirated into an air acetylene flame. The absorbance at 357.9 nm is compared to the absorbance obtained from a series of standard chromium solutions, and the chromium present is calculated in milligrams per square foot.

# A10.3 Reagents and Materials

A10.3.1 *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.<sup>6</sup> 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.

A10.3.2 *Purity of Water*— Deionized or distilled water having a volume resistivity of greater than 1 M $\Omega$  cm at 25°C as determined by Test Method B of Test Methods D1125.

A10.3.3 *Hydrochloric Acid*—Mix 1 part HCl (specific gravity 1.19) to 1.25 parts water.

A10.3.4 Standard Solution, Chromium—Dissolve 0.2828  $\mu g$  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.

A10.3.5 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 (A8.3.4): First Flask, no solution; Second flask, 1.0 mL; Third flask, 2.0 mL; and Fourth flask, 5.0 mL. Add 50 mL of 1:1.25 HCl Solution to each flask. Dilute each flask to 100 mL using distilled water.



# A10.4 Apparatus

A10.4.1 Atomic Absorption Spectrometer

# A10.5 Procedure

A10.5.1 Blank disk with a standard size of 4 in. <sup>2</sup> (if a one-sided test is required, spray one side with high-temperature acrylic lacquer), or equivalent.

A10.5.2 Clean the disk with acetone.

A10.5.3 Place the disk in a 500 mL beaker. Bend the sample slightly so that it does not rest flat on the bottom of the beaker.

A10.5.4 Fill the beaker with 50 mL of the required stripping solution.

A10.5.5 Strip at 180-200°F for 2 min. Allow to cool before testing.

A10.5.6 Analyze the solution in atomic absorption spectrophotometer.

#### A10.6 Calculation

A10.6.1 Calculate the amount of chromium present on the surface as follows:

$$C = A \times B \times \frac{144 \text{ in.}^2/\text{ft}^2}{D}$$
 (A10.1)

where:

 $A = \text{concentration of Cr}, \mu g/mL \text{ from the chromium calibra-}$ 

tion curve,

B = dilution factor, mL $C = \text{chromium } \mu g/\text{ft}^2, \text{ and}$ 

 $D = \text{area, in.}^2 \text{ of surface dissolved.}$ 

#### A11. METHOD FOR DETERMINING DRAWING TENDENCIES OF TIN MILL PRODUCTS USING A MODUL-R DEVICE

# A11.1 Scope

A11.1.1 This method is used to assess drawing tendencies of tin mill products. These tendencies commonly are termed R-bar (normal anisotropy) and  $\Delta r$  (planar anisotropy or earing) and are indicators of a metal's performance during operations, such as can drawing. This procedure provides a method for rapidly approximating the drawing tendencies of tin mill products.

A11.1.2 The drawing tendencies of tin mill products can be determined rapidly using a device called a Modul-R. This device has been used successfully for a material thickness as low as 55 lb/bb (0.0061 in.). The test can be performed on tin plate coatings as heavy as 0.25 lb/bb without removing the tin coating. If the tin coating weight exceeds 0.25 lb/bb, then the coating should be stripped chemically.

# A11.2 Terminology

A11.2.1 Definitions of Terms Specific to This Standard: Modul-R, n—the device used to measure the resonating frequency of a steel strip sample. The resonating frequency is used to calculate drawing properties, such as R-bar and  $\Delta$  r.

*R-bar*, *n*—the unitless property of a material used to describe its ability to be drawn. It is a ratio of the average properties in the plane of the sheet to those in the thickness of the sheet. It is commonly referred to as "normal anisotropy."

 $\Delta r$ , n—the unitless property of a material used to describe the variation of properties within the plane of a sheet of material. It also signifies the degree of earing during drawing. Negative values indicate earing will occur in a direction of 45° to the

direction of rolling. Positive values indicate earing will occur in a directions along and perpendicular to the direction of rolling. A  $\Delta r$  value of 0 is ideal.  $\Delta r$  commonly is referred to as "planar anisotropy."

## A11.3 Summary of Method

A11.3.1 Three rectangular coupons are blanked from a flat sheet of material such that they align with the rolling direction of the material, perpendicular to the rolling direction, and at a 45° angle to the rolling direction. These samples then are placed into a Modul-r testing device where they are vibrated to a resonant frequency. The resultant resonant frequencies are then converted to drawing properties. Minor corrections for surface roughness and thickness effects also are taken into account. The final results are reported as an R-bar value and a  $\Delta r$  value.

# A11.4 Apparatus

A11.4.1 Modul-R testing device (illustrated in Fig. A11.1), surface roughness measurement device, and a micrometer.

# **A11.5 Sample Preparation**

A11.5.1 Three test samples are blanked from a material to be evaluated. The three samples must be secured such that one is parallel to the rolling direction, one is perpendicular to the rolling direction, and one is at an angle of 45° to the rolling direction. The samples must be sheared to dimensions of 4.12 in. long and 0.250 in. wide. The 4.12-in. dimension allows for easy calculations later in the test method in A11.6.3.1. Any burrs should be removed with light sanding using a 400-grit



FIG. A11.1 Modul-R Device

paper. TFS coatings or tin plate coatings less than or equal to 0.25 lb/bb need not be removed. Organic coatings shall be removed prior to blanking the samples.

#### A11.6 Procedure

A11.6.1 Surface Roughness and Thickness Determination— The surface roughness of the material must be measured to the nearest microinch using a surface roughness measurement device. Thickness of the material also must be measured to the nearest 0.0001 in. using a micrometer.

A11.6.2 Frequency Testing of Blanked Samples—Frequency testing is performed in accordance with the instructions provided by the vendor of the Modul-R device. For convenience they are listed as follows:

A11.6.2.1 Place the sample in the slot marked *sample* on the right side of the panel. Move the sample into the slot until it has stopped within the holder.

A11.6.2.2 Pull the sample out from the holder just enough to relieve it from the stop. In this position, the sample should have about ½ in. protruding from the surface of the front panel.

A11.6.2.3 Place the bias switch in the positive (+) position. A11.6.2.4 Place the normal/phase switch in the normal position.

A11.6.2.5 Push the test switch down and note the brightness of the amber indication light marked *oscillator*, and the reading on the counter. Within a few seconds the reading should stabilize between 23.5 and 26.5 kHz with the amber light glowing.

Note A11.1—If the amber light does not come on, comes on very dimly, or the reading is either high, low, or rapidly fluctuating, remove the sample and reposition it in the sample slot. Turn the sample around end to end or turn it over and again try to obtain a reading. If necessary, hold the test button and gently jog the sample in the sample slot. If no reading can

be obtained, return the sample to its original position in the sample slot and move the bias switch to the negative (–) position. If the light still fails to come on, reposition the sample as described above. If up to this point normal oscillation will not take place, start again with the sample in the normal position and move the normal phase switch to the phase position and repeat above steps until a reading is obtained. If no reading can be obtained after all the above steps have been performed, a micrometer should be used to check the parallel sides of the sample. Readings should not vary by more than 0.001 in. Sample should not be bent, and all rough edges should be sanded smooth. In some instances when readings cannot be obtained from 45° samples, punching an additional sample from the sheeting at approximately 5° from the 45° position will provide a proper reading.

A11.6.2.6 Note the reading on the meter as soon as this occurs. Record it as f0 (frequency reading for sample punched parallel to the rolling direction), f90 (frequency reading for sample punched perpendicular to the rolling direction), and f45 (frequency of reading for sample punched 45° to the rolling direction).

A11.6.3 Calculation of Drawing Properties—R-bar values and  $\Delta r$  values can be calculated or obtained from tables. The first step in the process requires converting the frequency values determined above to modulus or E values. The E values are then adjusted based on surface roughness and thickness effects. The corrected E values then are converted to E-bar and  $\Delta E$  values. The E-bar and  $\Delta E$  values finally are converted to R-bar and  $\Delta r$  values.

A11.6.3.1 If the sample length as described in Section A11.4 is 4.12 in, then E values are determined from frequency values by using the following equation:

$$E = f^2/20$$
 (A11.1)

where:

E = the modulus value, and



f = the frequency obtained from step A11.6.2.

Note A11.2—The E obtained from the f0 value should be noted as E0, the E from the f45 value should be noted as E45, and the E from the f90 value should be noted as E90.

Note A11.3—If the sample length (described in Section A11.5) is different from 4.12 in., then E values must be obtained from the following equation:

$$E = 0.0029465 \, 1^2 \, f^2 \tag{A11.2}$$

where:

E = the modulus value,

1 = the length of the sample in inches (measured to the nearest 0.01 in., and

f = the frequency obtained from step A11.6.2.

A11.6.3.2 The E values then are corrected for the effect of surface roughness and thickness as follows:

$$E_c = E (1 + 2S/T)$$
 (A11.3)

where:

 $E_c$  = the new corrected modulus value,

E = the original modulus value,

S = the surface roughness, in microinches divided by 1,000, 000 (measured to the nearest microinch), and

T = the thickness, in inches (measured to the nearest 0.0001 in.).

Note A11.4—The corrected E values should be distinguished by direction as indicated above, that is,  $E_c0$ ,  $E_c45$ , and  $E_c90$ .

A11.6.3.3 Calculate E-bar and  $\Delta E$  values are by the following equations:

$$E - bar = (E_c 0 + E_c 90 + 2E_c 45)/4$$
 (A11.4)

$$\Delta E = (E_c 0 + E_c 90 - 2E_c 45)/2$$
 (A11.5)

A11.6.3.4 Determine R-bar by converting from E-bar to R-bar by using the following equation:

$$R - bar = 101.44/(E - bar - 38.83)^2 - 0.564$$
 (A11.6)

A11.6.3.5 Determine  $\Delta r$  by converting from  $\Delta E$  by using the following equation:

$$\Delta r = 0.031 - 0.323 \,(\Delta E) \tag{A11.7}$$

#### A11.7 Precision

A11.7.1 *Precision*—Aside from the normal variation of the product being measured, the precision of the input variables to the above equations can effect the reported final results. The length and width of the blanked samples should not vary by more than  $\pm 0.01$  in. from the recommended blank dimensions. The thickness of the material should be measured to the nearest 0.0001 in. The surface roughness should be measured to the nearest microinch.

#### A11.8 Keywords

A11.8.1 Modul-R; R-bar; Δr

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