**Designation: A623M − 16**

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

This standard is issued under the fixed designation A623M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

## INTRODUCTION

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

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

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

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

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

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

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

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

## 1. Scope

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

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

1.3 The following safety hazards caveat covers Annex A1 through Annex A8 of this specification: *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

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

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

E18 Test Methods for Rockwell Hardness of Metallic Materials

E112 Test Methods for Determining Average Grain Size

2.2 *Military Standards:*[3]

MIL-STD-129 Marking for Shipment and Storage

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

2.3 *Federal Standard:*[3]

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

## 3. Terminology

3.1 *Definitions:*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Special Properties Aims

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

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

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

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

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

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

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

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

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

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

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

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

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

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

3.1.24 *SITA, n*—100 square metres.

Formula for cut lengths:

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

Formula for coils:

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

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

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

[A] When steel produced by the silicon killed method is ordered, the silicon maximum may be increased to 0.080 %.

[B] When strand cast steel produced by the aluminum killed method is ordered or furnished, the silicon maximum may be increased to 0.030 % when approved by the purchaser.

[C] Types L and MR may be supplied as non-killed or killed, which would respectively be produced without and with aluminum additions. Minimum aluminum level for Type D is usually 0.02 %.

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

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

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

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

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

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

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

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

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

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

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

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

[A] These ranges are based on the use of the diamond spot anvil and a 1.588 mm hardened steel ball indenter.

[B] The hardness ranges are requirements unless otherwise agreed upon between producer and user.

Test conditions:

1. For referee purposes, samples of blackplate, unreflowed ETP, and ECCS shall be aged prior to testing by holding at 400°F for 10 min.
2. For referee purposes, the hardness test area on material produced with SBF or equivalent rolls shall be sanded smooth on both surfaces.
3. To avoid incorrect results due to the cantilever effect, samples shall have an area no larger than 4 in.$^2$ and the point of testing shall be no more than ½ in. off the center of the samples.

## 4. Base Metal

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

## 5. Chemical Composition

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

## 6. Cast or Heat Analysis

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

## 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

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

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

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

[A] These ranges are based on the use of the diamond spot anvil and a 1.588 mm hardened steel ball indenter.

[B] The hardness ranges are requirements unless otherwise agreed upon between producer and user.

Test conditions:

1. For referee purposes, samples of blackplate, unreflowed ETP, and ECCS shall be aged prior to testing by holding at 400°F for 10 min.
2. For referee purposes, the hardness test area on material produced with SBF or equivalent rolls shall be sanded smooth on both surfaces.
3. To avoid incorrect results due to the cantilever effect, samples shall have an area no larger than 4 in.$^{2}$ and the point of testing shall be no more than ½ in. off the center of the samples.

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

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

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

**TABLE 4 Mechanical Designations Double-Reduced Tin Mill Products**

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

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

[A] These values are based on the use of the diamond spot anvil and a 1.588 mm steel ball indenter. Testing will be in accordance with Test Methods and Definitions A370. Rockwell values are too varied to permit establishment of ranges. For details see *AISI Contributions to the Metallurgy of Steel*, "Survey of Mechanical Properties of Double Reduced Tin Plate," January 1966.

[B] Double-reduced products requiring a minimum % elongation or ductility will be designated as HEDR (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.

## 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

**TABLE 5 Thickness Tolerances**

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

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

order specification, the following procedure shall be used: Random and representative measurements using a hand micrometer must be made throughout the coil length. Measurements may be made at any location across the coil width except 10 mm from the mill-trimmed edge. The hand micrometers are assumed to be accurate to ±0.003 mm. No measurements are to be made within 1.0 m of a weld.

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

9.1.3 *Transverse Thickness Profile* is the change in sheet thickness from strip center to edge at right angles to the rolling direction. Thickness measured near the edge is normally less than the center thickness. The gauge measured 6 mm in from the mill trimmed edge shall be no more than either 13 % below the ordered thickness or 10 % less than the center thickness of the individual sheet being measured. Common components of transverse thickness profile are crown and feather edge.

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

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

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

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

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

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

**TABLE 6 Ordered Thickness and Thickness Tolerances**

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

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

**TABLE 7 Coil Length Variation**

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

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

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

9.2 *Dimensional Characteristics, Cut Sizes:*

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

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

9.2.3 *Transverse Thickness Profile* is the change in sheet thickness from strip center to edge at right angles to the rolling direction. Thickness measured near the edge is normally less than the center thickness. The gauge measured 6 mm in from the mill trimmed edge shall be no more than either 13 % below the ordered thickness or 10 % less than the center thickness of the individual sheet being measured. Common components of transverse thickness profile are crown and feather edge.

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

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

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

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

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

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

## 10. Special Requirements

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

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

## 11. Sheet Count—Cut Sizes

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

## 12. Retest Procedure

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

## 13. Conditions of Manufacture

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

## 14. Inspection

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

## 15. Rejection

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

## 16. Packaging

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

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

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

## 17. Marking

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

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

# ANNEXES

### (Mandatory Information)

## A1. ROCKWELL HARDNESS TESTING OF TIN MILL PRODUCTS

### A1.1 Scope

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

### A1.2 Anvil

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

### A1.3 Specimens

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

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

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

### A1.4 Reports

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

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

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

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

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

### INTRODUCTION

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

## A2.1 Scope

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

## A2.2 Summary of Method

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

## A2.3 Apparatus

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

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

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

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

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

## A2.4 Reagents and Materials

A2.4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

A2.4.3 *For Rate of Pickling Test:*

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

A2.4.4 *For Sample Preparation:*

A2.4.4.1 *Acetone.*

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

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

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

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

A2.4.5 *For Water Bath:*

A2.4.5.1 *Paraffin Oil.*

## A2.5 Test Specimen Preparation

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

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

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

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

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

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

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

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

A2.5.1.8 Trim specimen to 8 by 65 mm.

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

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

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

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

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

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

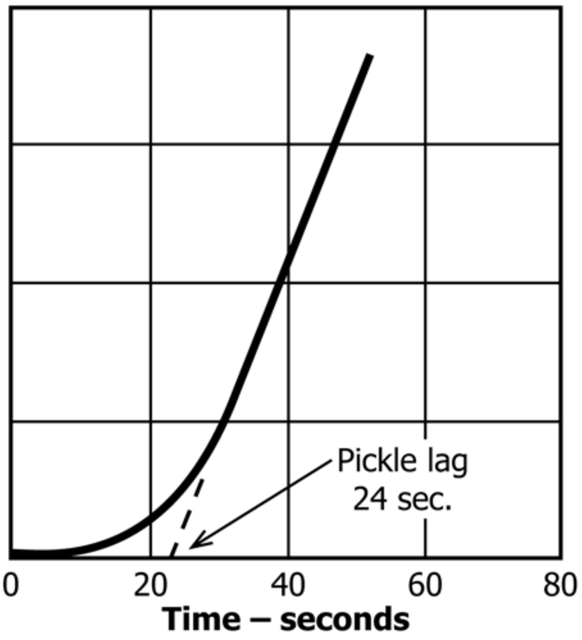

FIG. A2.1 Pickle Lag

## A2.6 Procedure

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

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

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

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

A2.6.5 Remove the specimen with a magnetized rod.

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

A2.6.7 Change acid after every ten specimens.

## A2.7 Calculation

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

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

## A2.8 Interferences

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

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

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

## A2.9 Assembly and Preparation of Apparatus

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

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

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

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

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

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

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

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

### INTRODUCTION

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

### A3.1 Scope

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

### A3.2 Summary of Method

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

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

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

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

A3.4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

A3.4.3 *Cotton or Soft Cloth.*

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

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

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

### A3.5 Test Specimen

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

### A3.6 Procedure

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

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

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

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

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

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

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

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

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

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

A3.6.3.1 This is a rapid nondestructive method.

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

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

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

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

### INTRODUCTION

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

### A4.1 Scope

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

### A4.2 Summary of Method

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

### A4.3 Apparatus[5,6]

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

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

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

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

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

A4.3.6 *Spectrophotometer and Cuvettes.*

### A4.4 Reagents and Materials

A4.4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

A4.4.3 *For Cleaning Test Specimen:*

A4.4.3.1 *Acetone.*

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

A4.4.4 *For Iron Solution Test:*

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

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

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

A4.4.5 *For Calibration:*

A4.4.5.1 *Iron Wire, Analytical.*

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

### A4.5 Procedure

A4.5.1 *Test Solutions:*

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

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

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

A4.5.2 *Sample Preparation:*

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

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

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

A4.5.3 *Iron Solution Test:*

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

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

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

A4.5.3.4 Affix the cap with specimen and gasket to the test vessel. Secure tightly. Invert the vessel immediately and let stand for 2 h at 27°C without agitation or vibration.

A4.5.3.5 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.

A4.5.3.6 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. (**Warning—**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.)

A4.5.3.7 Add 1 mL of 3 % $H_2O_2$ to each test vessel including the blank. Add the peroxide just before transferring the liquid in each test vessel to the cuvette. (See A4.8)

A4.5.3.8 Set the spectrophotometer at 485 nm. Zero the instrument by setting the scale for 100 % transmission on distilled or deionized water.

A4.5.3.9 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.

A4.5.3.10 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.

A4.5.3.11 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 66°C during the soaking of the gaskets assists in removal of any iron compounds and helps retain resiliency of the gaskets.)

## A4.6 Calibration

A4.6.1 The spectrophotometer and cuvettes should be calibrated with standard solutions containing known amounts of iron. A typical calibration might proceed as follows:

A4.6.1.1 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.

A4.6.1.2 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.

A4.6.1.3 Mix 25 mL of the $H_2SO_4$-$H_2O_2$ and 25 mL of the $NH_4SCN$ stock solutions as in A4.5.3.3. 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.

A4.6.1.4 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 Coleman Model 6A Junior spectrophotometer[9,6] and 19 by 150-mm round cuvettes is shown in Fig. A4.1. 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*.

## A4.7 Calculation

A4.7.1 If the spectrophotometer does not have an *ISV* scale, determine the *ISV* from the calibration curve for each sample including the blank.

A4.7.2 Subtract the blank *ISV* from each of the scale *ISV* readings or from the *ISV's* obtained in A4.7.1. This is the true *ISV*.

## A4.8 Interferences

A4.8.1 *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.

A4.8.2 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 A4.5.2.1), (*2*) agitation, swirling, or vibration of test vessel during 2-h test time, (*3*) leakers, and (*4*) rise in temperature.

A4.8.3 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.

## A4.9 Precision

A4.9.1 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 |
| A | 4.4 | 2–8 | 1.6 | 56 |
| B | 9.4 | 8–19$^A$ | 1.9 | 36 |
| C | 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 |

---

[9] Such an instrument or its equivalent has been found satisfactory.

[A] 35 of 36 samples in range from 8 to 12.

A4.9.2 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).

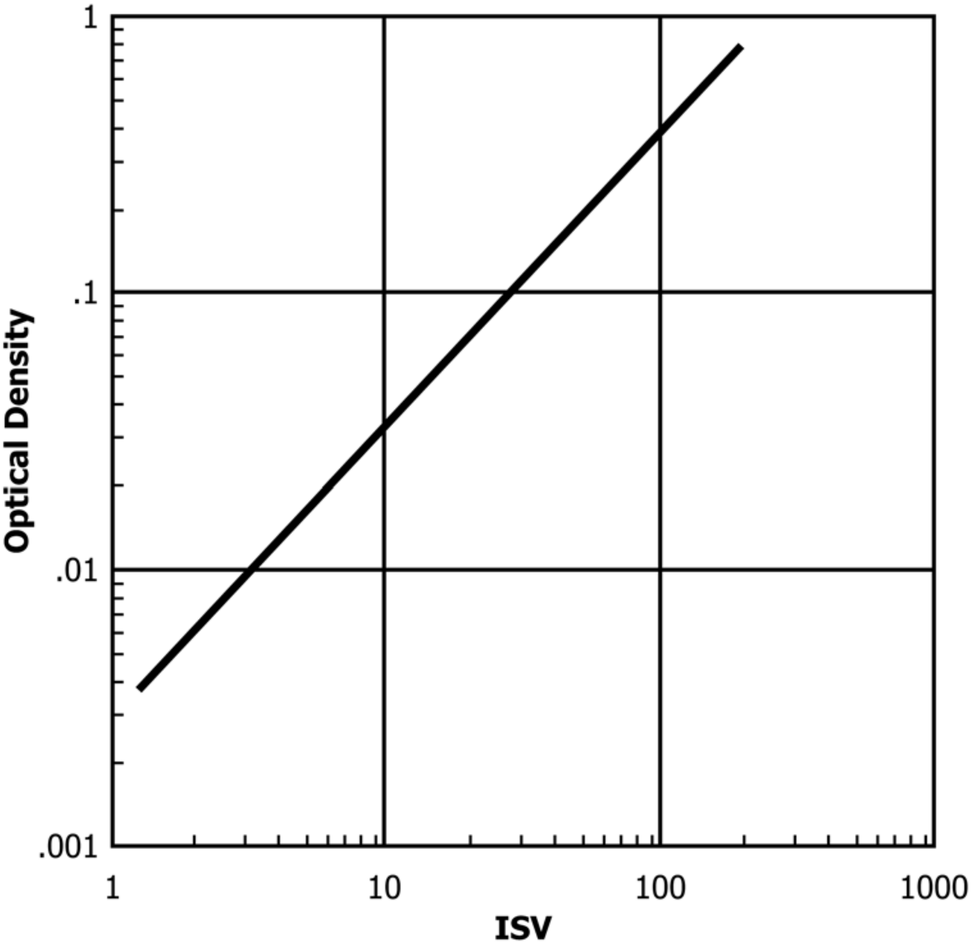

FIG. A4.1 Typical Iron Solution Value Calibration Curve

## A5. 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.

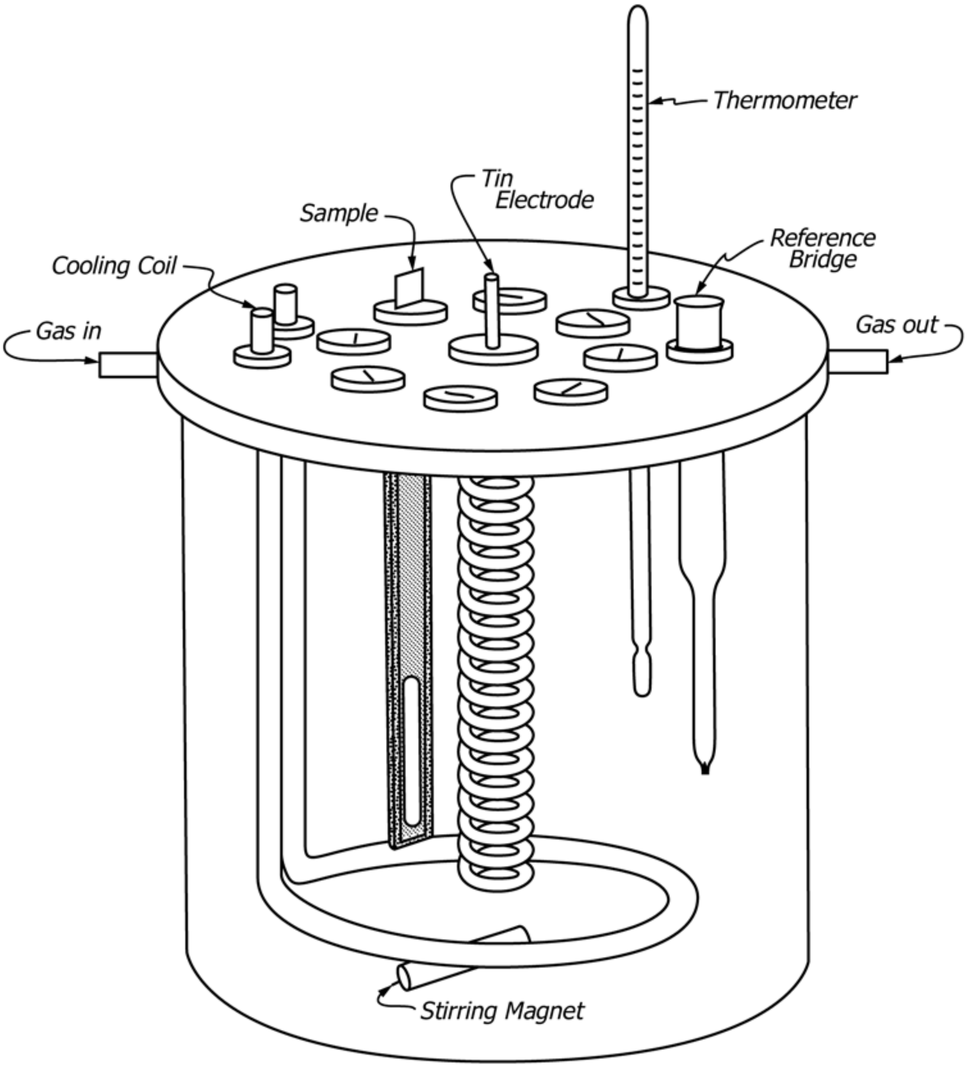

FIG. A5.1 Test Cell Used in the *ATC* Test

## A5.1 Scope

A5.1.1 The alloy-tin couple test,[10] 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 nominal tin coating weights 5.6/2.8 g/m$^2$ and heavier electrolytic tin plate (for K-plate, see 3.1.13.2). It is not applicable to 2.8/2.8 g/m$^2$ and lighter electrolytic tin plate.

## A5.2 Summary of Method

A5.2.1 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.

## A5.3 Apparatus

A5.3.1 *Constant-Temperature Cabinet or Room* (27 ± 0.5°C).

A5.3.2 *Test Cell* (Fig. A5.1):

A5.3.2.1 *Borosilicate Glass Test Cell,* approximately 1.5 L capacity.

A5.3.2.2 *Poly(Methyl Methacrylate) Plastic Cover* for test cell approximately 12.7 mm thick drilled with 15.9-mm diameter holes to accommodate cell elements.

A5.3.2.3 *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.

A5.3.2.4 *Silicone Rubber 6.35-mm Thick Grommets* to act as gas-tight holders for cell elements inserted through the plastic cover.

A5.3.3 *Magnetic Stirrer.*

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[10] 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.

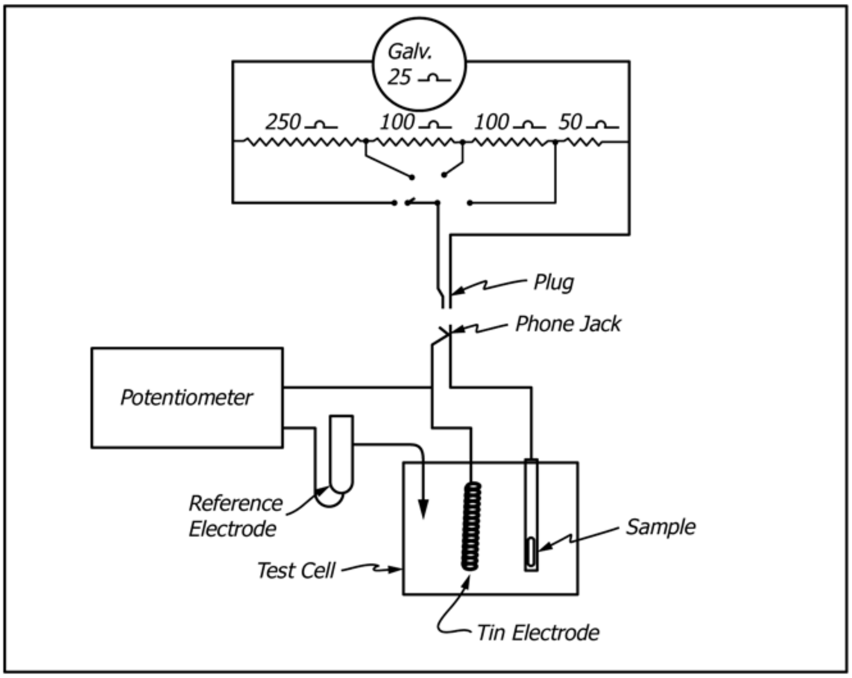

**FIG. A5.2 Schematic Diagram of *ATC* Test Circuit**

A5.3.4 *Low-Resistance, High-Sensitivity Galvanometer.*[11,6]

A5.3.5 *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.

A5.3.6 *Calomel Reference Electrode* (Either saturated or 0.1 *N* is satisfactory).

A5.3.7 *Power Source* capable of supplying variable dc voltage for use in sample preparation (cathodic cleaning 10-V dc and tin stripping 0.4-V dc reducible to 0.2 V).

A5.3.8 *Various Electrical Components* such as plugs, jacks, switches, and resistors to permit construction of circuit depicted in schematic diagram (Fig. A5.2).

A5.3.9 *(Optional) Special Die* for applying microcrystalline wax to mask off known areas on test specimen.[5,6]

### 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.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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 *Test Medium:*

A5.4.3.1 *Distilled Water or Deionized Water* of equal purity.

A5.4.3.2 *Ethanol, Denatured* (70 % volume).

A5.4.3.3 *Frozen Concentrated Grapefruit Juice.*

A5.4.3.4 *Nitrogen Gas (High-Purity Oxygen-Free Dry Tank Nitrogen).*

A5.4.3.5 *Potassium Sorbate.*

A5.4.3.6 *Pure Tin Wire* (approximately 3.18-mm diameter).

A5.4.3.7 *Sodium Hydroxide Solution* (NaOH) (10 %).

A5.4.3.8 *Stannous Chloride Solution* ($SnCl_2 \cdot 2\ H_2O$).

A5.4.4 *Sample Preparation:*

A5.4.4.1 *Acetone.*

A5.4.4.2 *Microcrystalline Wax* (140 to 145°F melting point).

A5.4.4.3 *Poly(Methyl Methacrylate) Plastic Strips* 1.59 by 14.3 by 82.6 mm.

A5.4.4.4 *Sodium Carbonate Solution* ($Na_2CO_3$) (0.5 %).

A5.4.4.5 *Sodium Hydroxide Solution* (NaOH) (5 %).

### A5.5 Test Specimen

A5.5.1 The specimen consists of a piece of tin plate cut 12.7 by 114.3 mm with the long dimension transverse to the rolling direction.

A5.5.2 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.

A5.5.3 Details of sample preparation are given in A5.7.

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[11] A Leeds and Northrup Model 2430-C, 25-Ω galvanometer with 0.003-µA/mm sensitivity or equivalent has been found satisfactory.

## A5.6 Preparation of Apparatus (Fig. A5.2)

A5.6.1 Drill 15.9-mm diameter holes in 12.7-mm 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.

A5.6.2 Cut stoppers or grommets from 6.35-mm thick silicone rubber to fit snugly in 15.9-mm diameter openings. Cut holes or slits in stoppers and grommets to hold various cell elements. 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.

A5.6.3 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.

A5.6.4 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.

A5.6.5 Form 3.18-mm diameter pure tin wire into a loosely wound coil to give a total surface area of approximately 100 $cm^2$. Cathodically clean in 0.5 % $Na_2CO_3$ solution, rinse in tap water and in acetone.

## A5.7 Procedure

A5.7.1 *Test Medium:*

A5.7.1.1 Place a polytetrafluoroethylene-covered magnetic stirring bar in the bottom of the test cell.

A5.7.1.2 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 6.35-mm headspace. Turn on the magnetic stirrer.

A5.7.1.3 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 25.4 or 50.8 mm of water or 12.7 mm of dibutyl phthalate.

A5.7.1.4 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 27°C.

A5.7.1.5 Insert the cleaned pure tin anode into the test cell.

A5.7.1.6 Add $SnCl_2{\cdot}2H_2O$ to produce a concentration of 0.190 g/L. This yields a $Sn^{++}$ concentration of 100 ppm. Continue stirring for 5 or 10 min to make sure the $SnCl_2 \cdot 2H_2O$ has been dissolved.

A5.7.1.7 Discontinue stirring.

A5.7.1.8 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.

A5.7.2 *Test Specimen:*

A5.7.2.1 Degrease the specimen in acetone and allow to dry.

A5.7.2.2 Clean the specimen cathodically in 0.5 % $Na_2CO_3$ solution (carbon anode) using a current density of approximately 25 $mA/cm^2$. A 10-V dc 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.

A5.7.2.3 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-Ω 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 to dry.

A5.7.2.4 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 $cm^2$ do not affect the *ATC* measurement. It is strongly recommended that an area of 2.3 $cm^2$ be used in the test. A die that produces an outline of wax exposing 2.3 $cm^2$ is available (A5.3.9). After the test area has been outlined, manually wax the specimen to a thin plastic backing (A5.4.4.3) making certain all edges and surfaces other than the test area are covered.

A5.7.3 *Current Measurement:*

A5.7.3.1 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. A5.2 and note that the phone jacks are the *shorting* type). All test specimens are coupled to the single tin anode.

A5.7.3.2 After 20 h, measure the current flowing between the tin anode and each individual specimen with a low-resistance, high-sensitivity galvanometer (A5.3.4). The test cell must be free from vibration during the time the specimens are in the cell.

A5.7.3.3 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.

A5.7.3.4 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.

### A5.8 Calculation

A5.8.1 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.

### A5.9 Hazards

A5.9.1 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.

A5.9.2 Take care to avoid vibration during the test run. Do not bump or disturb the electrodes before taking current measurements.

A5.9.3 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 $Sn^{++}$ shifts the potential approximately 50 mV in the cathodic (positive) direction.

A5.9.4 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.

A5.9.5 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.

## A6. METHODS FOR DETERMINATION OF TOTAL SURFACE OIL ON TIN MILL PRODUCTS

### A6.1 Scope

A6.1.1 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 Method) | A6.3 to A6.11 |
| B—Ellipsometry | A6.12 to A6.19 |

### A6.2 Significance and Use

A6.2.1 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

### A6.3 Summary of Method

A6.3.1 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.

### A6.4 Apparatus

A6.4.1 *Slotted Mandrel* with handle for coiling the strips.

A6.4.1.1 A 12.7-mm diameter slotted mandrel is used for high-temper materials and a 25.4-mm diameter slotted mandrel is used for low-temper plate.

### A6.5 Reagents and Materials

A6.5.1 *Chloroform* ($CHCl_3$), distilled reagent grade or equivalent.

### A6.6 Hazards

A6.6.1 Chloroform vapors present a potential health hazard. The cleaning of equipment, extraction, and evaporation of the chloroform should be done in an exhaust hood.

### A6.7 Test Specimen

A6.7.1 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.

### A6.8 Preparation of Apparatus

A6.8.1 Clean the shears for cutting the plate into strips, coiling mandrel, pliers, and forceps with chloroform or equivalent.

A6.8.2 The glassware must be rinsed with boiling chloroform or equivalent.

A6.8.3 Wear clean white cloth gloves when handling plate.

### A6.9 Procedure

A6.9.1 Cut the sample of plate at least 3225 $cm^2$ (preferably 6450 $cm^2$) into 50.8-mm wide strips.

A6.9.2 Determine the exact area of plate (length by width by number of strips).

A6.9.3 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.

A6.9.4 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.

### A6.10 Calculation

A6.10.1 Calculate the weight of oil per square metre as follows:

$$\text{mg/m}^2 = \frac{W \times 10\,000}{A}$$

*where:*

$W$ = weight of extracted oil, mg, and

$A$ = area of sample, cm$^2$.

To convert to g/SITA: divide mg/m$^2$ by 10.

### A6.11 Precision and Bias

A6.11.1 Make all weighings to the nearest 0.0001 g.

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

### A6.12 Scope

A6.12.1 This method covers the determination of the total oil on the surface of tin mill products by ellipsometry.

### A6.13 Summary of Method

A6.13.1 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.

### A6.14 Apparatus

A6.14.1 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.[12,6] 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.

### A6.15 Reagents and Materials

A6.15.1 Non-residue forming degreasing solvent such as *Trichloroethylene* or *1-Bromopropane*.

### A6.16 Hazards

A6.16.1 *Trichloroethylene* and *1-Bromopopane* vapors present a potential health hazard. An exhaust hood is required for operation of the ellipsometer.

### A6.17 Preparation of Apparatus

A6.17.1 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.

A6.17.2 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 × 100. For example: 5 = 500V with a ½ setting × 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.

A6.17.3 Install a typical sample of the type to be measured with the rolling direction in the vertical position.

A6.17.4 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".

A6.17.5 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.

A6.17.6 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.

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[12] 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.

A6.17.7 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.

### A6.18 Procedure

A6.18.1 Samples for oil weight determinations are obtained by stamping disks 57.33 ± 0.02 mm in diameter, which is equivalent 25.81 $cm^2$ per side.

A6.18.2 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.

A6.18.3 Insert the sample into the magnetic sample holder with the rolling direction in the vertical position.

A6.18.4 Verify that the Null control, Multiplier, and High Voltage settings are set up correctly as described in A6.17.

A6.18.5 Set the Reading Switch on the Operator Control Panel to the Oil position.

A6.18.6 Set the Clean Auto/Off Switch to the Auto position.

A6.18.7 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.

A6.18.8 If the opposite side of the sample is to be measured rotate the sample and repeat step in A6.18.7. It is advisable that one side per sample is measured to prevent spray-over to the opposite side during the cleaning cycle.

A6.18.9 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.

A6.18.10 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.

### A6.19 Precision and Bias

A6.19.1 Four laboratories measured the repeatability using a different coil for each laboratory. The standard error was 0.25 $mg/m^2$ for 30 samples with oil weights ranging from 5.0 to 12.5 $mg/m^2$. A reproducibility study could not be conducted due to volatilization of the oil.

A6.19.2 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.

## A7. DETERMINATION OF CHROMIUM ON TIN PLATE BY THE DIPHENYLCARBAZIDE METHOD

### A7.1 Scope

A7.1.1 This method[13] covers the determination of chromium on tin plate with the use of diphenylcarbazide.

### A7.2 Reagents

A7.2.1 *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.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A7.2.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A7.2.3 *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.

A7.2.4 *Chromate, Standard Solution B* (1 mL = 10.0 µg Cr)—Pipet 20 mL of the chromate Standard Solution A into a 1.0-L volumetric flask and add water to 1.0 L.

A7.2.5 *Diphenylcarbazide Reagent*—Add 10.0 mL of acetone, 10.0 mL of 95 % ethyl alcohol, and 20.0 mL of $H_3PO_4$ (85 % acid diluted with an equal volume of water) to 0.25 grams of diphenylcarbazide powder.

A7.2.6 *Hydrochloric Acid* (HCl) (sp gr 1.19).

A7.2.7 *Potassium Permanganate Saturated Solution* ($KMnO_4$).

A7.2.8 *Sodium Hydroxide* (1.0 *N*)*—Trisodium Phosphate* (5 %) *Solution*—Dissolve 40.0 g of NaOH and 50.0 g of $Na_3PO_4$ in water and dilute to 1.0 L.

A7.2.9 *Sulfuric Acid* (1+3)—Add 100 mL of $H_2SO_4$ (sp gr 1.84) slowly and with stirring to 300 mL of water.

### A7.3 Procedure

A7.3.1 Use for analysis a sample having 52 $cm^2$ of surface area (one 26-$cm^2$ disk). If both sides of the sample are to be stripped, slightly bend the disk through the center so it will not

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[13] Furman, N. H., "Chromium in Acid Solution with Diphenylcarbazide," *Scott's Standard Methods of Chemical Analysis,* Vol 1, 1962, pp. 357–359.

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 3.2 mm wide at the perimeter of the stopper.

A7.3.2 Place the sample in a 250-mL beaker, add 25 mL of $NaOH \cdot Na_3PO_4$ solution, and heat to boiling. Boil for 1½ min. Transfer the solution to another 250-mL beaker, washing disk and beaker once with water. Add 25 mL of $H_2SO_4$ (1+3) to the original beaker and sample, heat to boiling, and boil 1 min. 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 $H_2SO_4$ is boiling. This is necessary to keep the surface completely wetted and strip all of the chromium from the surface of the tin plate.

A7.3.3 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.

A7.3.4 Transfer to a 100-mL volumetric flask and cool to approximately 70°F [21°C] in a water bath. Add 3.0 mL of diphenylcarbazide reagent, make to mark with distilled water, and mix.

A7.3.5 Determine optical density, within 30 min after the addition of diphenylcarbazide reagent to the sample, at 540 nm.[14]

A7.3.6 A reagent blank and a standard including all solutions used in treating a sample should be carried along with each set of samples.

### A7.4 Calibration of Spectrophotometer

A7.4.1 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.

A7.4.2 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.

### A7.5 Calculation of Chromium on Tin Plate

A7.5.1 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,
$O.D._2$ = optical density of reagent blank, and
$A$ = area of sample used.

## A8. 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.

### A8.1 Scope

A8.1.1 The AMP test is one of four special property tests used to measure certain characteristics of electrolytic tin plate that affect 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.

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[14] The Coleman Spectrophotometer with 1.5-cm cell is a suitable instrument.

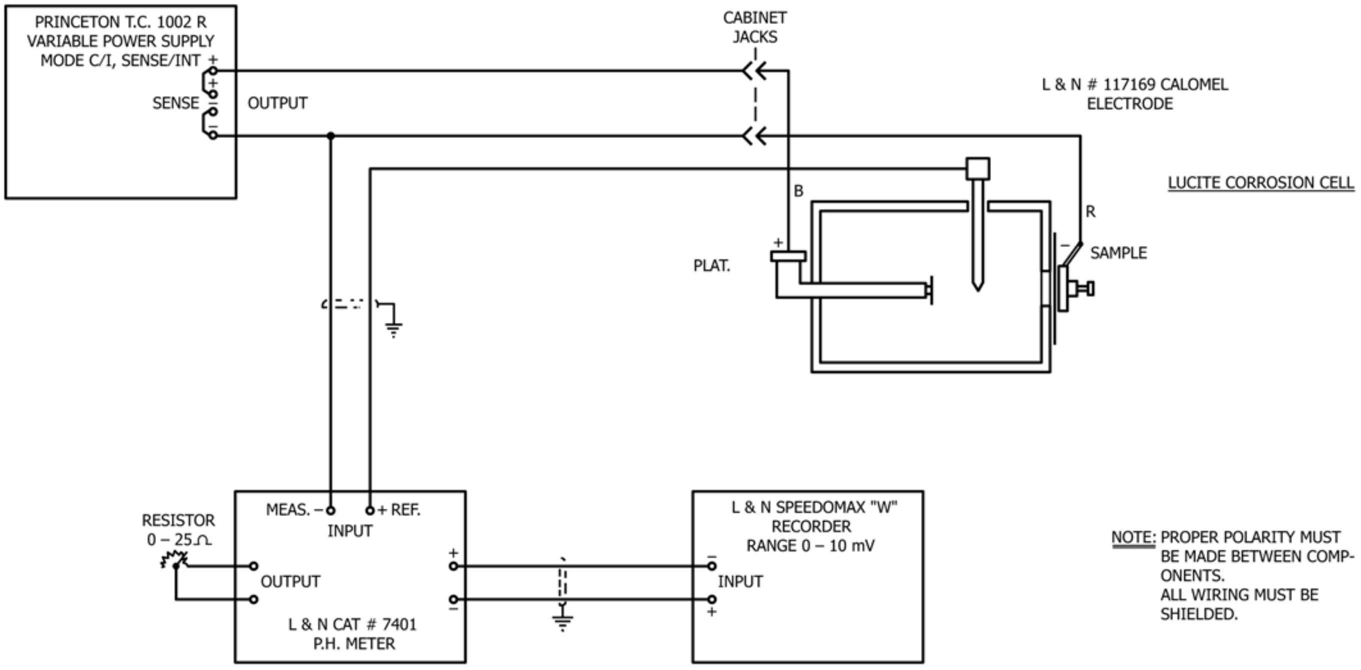

FIG. A8.1 Wiring Schematic for AMP Test

## A8.2 Summary of Method

A8.2.1 This method 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.

## A8.3 Apparatus

A8.3.1 *Test Cell* (see Fig. A8.1).

A8.3.2 *AMP Analyzer* (see Fig. A8.2).

A8.3.3 *dc Power Supply,* capable of supplying 3 to 4 A at 10 to 12 V and means of reversing polarity.

A8.3.4 *Three Laboratory Hot Plates*—Thermolyne-type P 100, Model HP-A1915B, or equivalent.

A8.3.5 *Crystallizing Dish,* 150-mm diameter, 25-mm depth.

A8.3.6 *Watch Glass,* 115-m diameter.

A8.3.7 *Two 250-mL Beakers*—Griffin low form.

A8.3.8 *Two Watch Glasses,* for the 400-mL beakers.

A8.3.9 *Three 400-mL Beakers*—Berzelius (tall form without pouring spout).

A8.3.10 *Timer or Stopwatch,* capable of reading to the nearest second.

A8.3.11 *400-mL Beaker,* stainless steel.

A8.3.12 *Levelling Funnel.*

A8.3.13 *22.2-mm Inside Diameter O-Ring*—387 mm$^2$.

## A8.4 Reagents and Materials

A8.4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

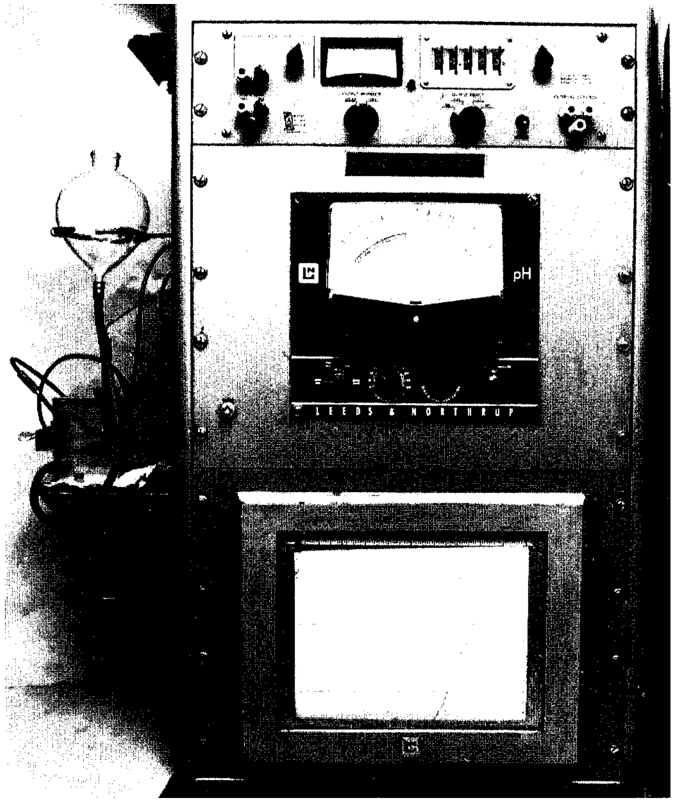

FIG. A8.2 Corrosion Analyzer (AMP)

A8.4.2 *Purity of Water—* Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

A8.4.3 *Anhydrous Acetone.*

A8.4.4 *Stripping Solution.*

A8.4.4.1 *5.0 % (by weight) sodium hydroxide, (NaOH),* in distilled water.

A8.4.4.2 *1.0 % (by weight) potassium iodate ($KlO_3$),* in distilled water.

A8.4.5 *0.5 M Citric Acid*—9.1 g anhydrous citric acid per 1 L distilled water.

A8.4.6 *Potassium Sorbate.*

A8.4.7 *Frozen Sweetened Grapefruit Juice Concentrate (GFJ).*

A8.4.8 *Equivalent to GFJ,* when dissolved in 500 mL of distilled water:

A8.4.8.1 5 g citric acid,
A8.4.8.2 2.5 g sodium citrate,
A8.4.8.3 17.5 g fructose,
A8.4.8.4 25 g sucrose, or
A8.4.8.5 0.25 g potassium sorbate.
A8.4.8.6 Prepare fresh for each occasion of testing.

A8.4.9 *Cleaning Solution*—1 % (by weight) sodium carbonate in 1500 mL of distilled water.

### A8.5 Test Media

A8.5.1 The 575-mL size cans of grapefruit juice concentrate are thawed, opened, and diluted with 510 mL of distilled water per can. The resulting single strength GFJ is filtered under vacuum through silica sand to remove most of the pulpy material.

A8.5.2 To retard spoilage, 0.5 g 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.

A8.5.3 Enough GFJ to fill the cell, 400 mL, is brought to room temperature, 22 to 23°C, and poured into the levelling funnel.

A8.5.4 Under normal conditions, this 400-mL aliquot of juice will yield about 200 results before it need be discarded. Worn-out juice is indicated by a marked decrease or offset, or both, in the slope of the standard curve.

### A8.6 Test Specimen

A8.6.1 Samples, 3.81 cm wide by any convenient length, 12.7 to 25.4 cm 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.

### A8.7 Sample Preparation

A8.7.1 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 222 to 223 A/$m^2$ are satisfactory. Rinse the sample thoroughly in distilled water dip in hot acetone and dry in air.

NOTE A8.1—The final current polarity *must be cathodic* for rapid removal of the free tin in subsequent steps.

A8.7.2 Two of the hot plates should be located near a sink where distilled water is plentiful. The third hot plate should be next to the AMP analyzer.

A8.7.3 The crystallizing dish, with the 115-mm watch glass placed in it, is filled to a depth of 3.81 cm with stripping solution and placed on one of the two hot plates. The hot plate should be adjusted to maintain the solution at 40°C.

A8.7.4 The two 250-mL beakers and the 400-mL stainless steel beaker are placed on the second hot plate. Fill one of the 250-mL beakers with stripping solution to a depth slightly less than 3.81 cm. Fill the second 250-mL beaker with a 0.5 *M* citric acid solution to a depth of about 5.08 cm. Fill the 400-mL beaker two thirds full of acetone. Adjust this hot plate so that the acetone almost boils.

A8.7.5 The remaining two 400-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.

A8.7.6 Place a sample upright in the crystallizing dish of stripping solution allowing it to lean against the side of the dish. The submerged 115-mm 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 250-mL beaker containing a second stripping solution. After 30 ± 5 s, remove the sample and rinse in running distilled water.

A8.7.7 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. It is now ready for insertion into the cell.

### A8.8 Aerated Media Polarization Test

A8.8.1 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 |

A8.8.2 Remove the sample from the acetone beaker and allow it to air dry.

A8.8.3 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 A8.2—Overtightening will drastically reduce the life of the O-ring and may even cause it to be torn loose from the cell.

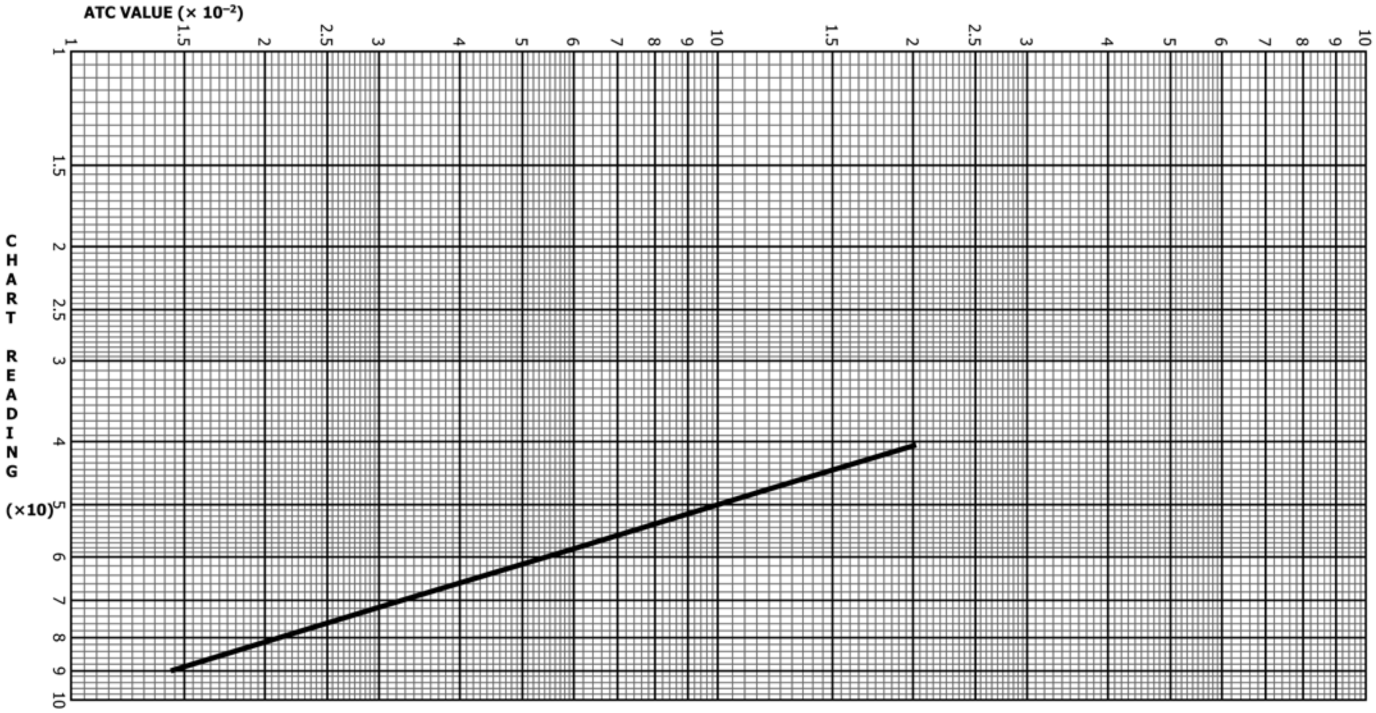

FIG. A8.3 ATC Conversion Chart from Typical Calibration Curve

A8.8.4 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.)

A8.8.4.1 Turn voltage switch on.

A8.8.4.2 Turn chart switch on.

A8.8.4.3 As pen point reaches an accented line on the chart paper, turn current switch on.

A8.8.4.4 After 90 s (7.62 cm of chart travel) read the end potential as chart divisions (to nearest tenth).

A8.8.4.5 Repeat A8.8.4.1 through A8.8.4.4, in reverse order, turning switches off to discontinue test.

A8.8.5 Lower the levelling funnel to drain the cell, loosen the follower plate, and remove the sample. Dry the O-ring and follower plate to remove any droplets of GFJ that 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 3.81-cm 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.

## A8.9 Interpretation of Results

A8.9.1 In any batch of samples to be run several standards, covering the range of ATC values from about 0.015 to 0.300 μa/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 (Fig. A8.3).

A8.9.2 The AMP value (equivalent ATC) for the unknown can then be obtained from this standard curve.

A8.9.2.1 *Temperature*—Close temperature control of the GFJ is as critical in this test as it is in the ATC test. Therefore, it is important that the analyzer and cell be installed in a location where temperature can be closely monitored.

(*1*) It is strongly recommended that the temperature variations of the GFJ be no more than ±1°F during testing. It is possible to thermostat only the cell, but this would defeat the ease and speed of inserting and removing samples.

A8.9.2.2 *Standard*—Standards for calibration should be developed in the following manner:

*(1)* Secure 40 to 50 consecutive sheets from a differentially coated coil of tin plate. (This prevents the top and bottom surfaces from getting mixed when the 3.81-cm wide samples are cut.)

*(2)* Shear the edges from these sheets so that only about 25.4 cm or 27.9 cm of the center of the original sheet width remains.

*(3)* Select the first, middle, and last sheets of the number taken and cut 3.81-cm wide samples from each across the rolling direction, perpendicular to the (25.4 to 27.94 cm) dimension.

*(4)* Note, however, that all of the preceding precautions should be observed in obtaining tin plate for this treatment.

A8.9.3 These standards may be obtained by testing 40 to 50 samples of differentially coated tinplate and performing the ATC test in accordance with ASTM.

A8.9.4 Six standards are probably the minimum requirement. The values of these should be well spaced along the standard curve:

1—0.015 μa/cm$^2$ (approximately)
2—0.030
3—0.060
4—0.100
5—0.180
6—0.300

A8.9.5 If difficulty is experienced in obtaining 1 or 2, it has been found that subjecting the tin plate to a 10-min heating cycle at 420°F will result in a five- to ten-fold reduction in ATC value.

## A9. METHOD FOR DETERMINING DRAWING TENDENCIES OF TIN MILL PRODUCTS USING A MODUL-R DEVICE

### A9.1 Scope

A9.1.1 This method is used to assess drawing tendencies of tin mill products. These tendencies commonly are termed R-bar (normal anisotropy) and Δr (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.

A9.1.2 The drawing tendencies of tin mill products can be rapidly determined using a device called a Modul-R. This device has been used successfully for a material thickness as low as 0.155 mm. The test can be performed on tin plate coatings as heavy as 2.8 g/m$^2$ without removing the tin coating. If the tin coating weight exceeds 2.8 g/m$^2$, the coating should be chemically stripped.

### A9.2 Terminology

A9.2.1 *Definitions of Terms Specific to This Standard:*

A9.2.1.1 *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 Δ r.

A9.2.1.2 *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. Commonly, it is referred to as "normal anisotropy."

A9.2.1.3 *Δr, 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 Δr value of 0 is ideal. Δr commonly is referred to as "planar anisotropy."

### A9.3 Summary of Method

A9.3.1 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 are then placed into a Modul-R testing device where they are vibrated to a resonant frequency. The resultant resonant frequencies then are 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 Δr value.

### A9.4 Apparatus

A9.4.1 Modul-R testing device (see Fig. A9.1), surface roughness measurement device, and a micrometer are to be used.

### A9.5 Sample Preparation

A9.5.1 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 104.63 mm long and 6.35 mm wide. The 104.63-mm dimension allows for easy calculations later in the test method under A9.6.1. Any burrs should be removed with light sanding using a 400-grit paper. TFS coatings or tin plate coatings less than or equal to 2.8 g/m$^2$ need not be removed. Organic coatings shall be removed prior to blanking the samples.

### A9.6 Procedure

A9.6.1 *Surface Roughness and Thickness Determination*—The surface roughness of the material must be measured to the nearest 0.025 micron using a surface roughness measurement device. Thickness of the material also must be measured to the nearest 0.0025 mm using a micrometer.

A9.6.2 *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 here as follows:

A9.6.2.1 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.

A9.6.2.2 Pull the sample out from the holder just enough to relieve it from the stop. In this position, the sample should have about 6 mm protruding from the surface of the front panel.

A9.6.2.3 Place the bias switch in the positive (+) position.

A9.6.2.4 Place the normal/phase switch in the normal position.

A9.6.2.5 Push the test switch down and note the brightness of the amber indication light marked *oscillator* , and the

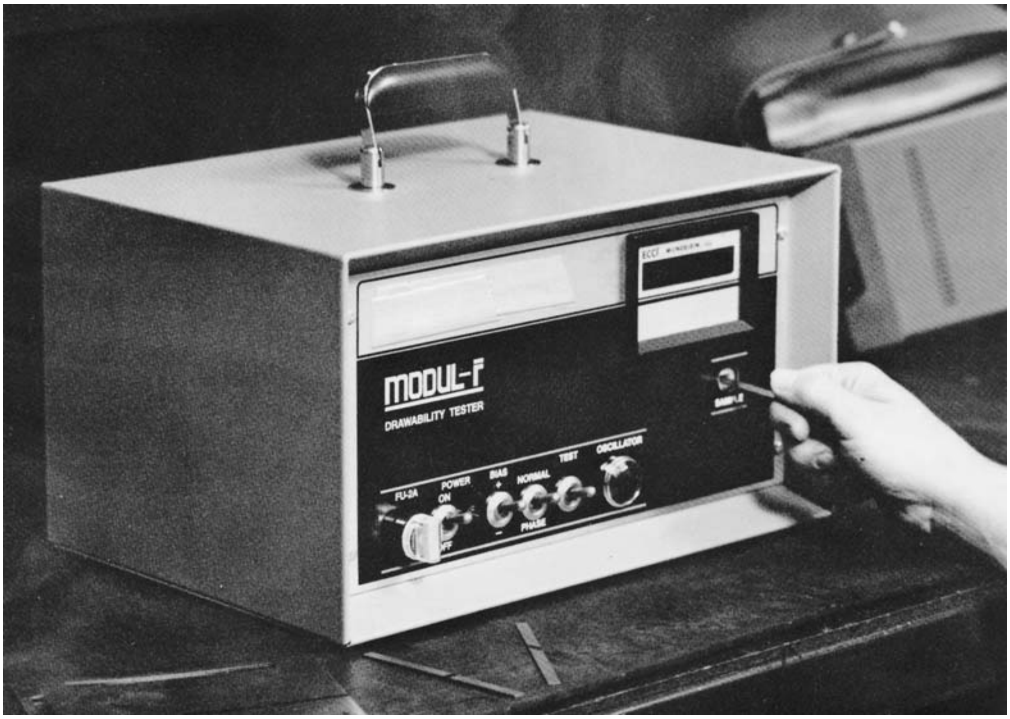

**FIG. A9.1 Modul-R Device**

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 A9.1—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 0.025 mm. 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.

A9.6.2.6 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).

A9.6.3 *Calculation of Drawing Properties*—R-bar values and Δr 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 then are adjusted based on surface roughness and thickness effects. The corrected E values then are converted to E-bar and ΔE values. The E-bar and ΔE values finally are converted to R-bar and Δr values.

A9.6.3.1 If the sample length (described in A9.5) is 104.63 mm, then E values are determined from frequency values by using the following equation:

$$E = f^2/20 \qquad (A9.1)$$

where:

E = the modulus value, and
f = the frequency obtained from A9.6.2.

Note A9.2—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 A9.3—If the sample length (described in A9.5) is different from 104.63 mm, the E values must be obtained from the following equation:

$$E = 0.0000045671\, l^2 f^2 \qquad (A9.2)$$

where:

E = the modulus value,
l = the length of the sample in mm (measured to the nearest 0.25 mm), and
f = the frequency obtained from A9.6.2.

A9.6.3.2 The E values are then corrected for the effect of surface roughness and thickness as follows:

$$E_c = E\left(1+2S/T\right) \qquad (A9.3)$$

where:

$E_c$ = the new corrected modulus value,
E = the original modulus value,

S = the surface roughness in microns divided by 1,000(measured to the 0.025 microns), and

T = the thickness in millimetres (measured to the nearest 0.0025 mm).

NOTE A9.4—The corrected E values should be distinguished by direction as indicated above, that is, $E_c0$, $E_c45$, and $E_c90$.

A9.6.3.3 Calculate E-bar and ΔE values are by the following equations:

$$E - bar = (E_c0 + E_c90 + 2E_c45)/4 \tag{A9.4}$$

$$\Delta E = (E_c0 + E_c90 - 2E_c45)/2$$

A9.6.3.4 Determine R-bar by converting from E-bar to R-bar by using the following equation:

$$R - bar = 101.44/(E - bar - 38.83)^2 - 0.564 \tag{A9.5}$$

A9.6.3.5 Determine Δr by converting from ΔE by using the following equation:

$$\Delta r = 0.031 - 0.323\,(\Delta E) \tag{A9.6}$$

## A9.7 Precision

A9.7.1 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 ±0.25 mm from the recommended blank dimensions. The thickness of the material should be measured to the nearest 0.0025 mm. The surface roughness should be measured to the nearest 0.025 micron.

## A9.8 Keywords

A9.8.1 Modul-R; R-bar; Δr

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