INTERNATIONAL STANDARD

ISO 11949

Second edition 2016-03-01

Cold-reduced tinmill products — Electrolytic tinplate

Aciers pour emballage laminés à froid — Fer blanc électrolytique





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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 9, *Tinplate and blackplate*.

This second edition cancels and replaces the first edition (ISO 11949:1995), which has been technically revised.

Introduction

Selling of packaging steels is today a worldwide business. Therefore, revision of this International Standard was expected earlier since the last edition dated from 1995. Because of the long period between revisions, harmonization became difficult. In some regions, the properties of the packaging steels are determined by the hardness test whereas in other regions, a decade ago, the hardness test was replaced by the tensile test. Since the latest available techniques should be reflected in this International Standard, the possibility of using the tensile test as the reference test for determining the mechanical properties should be considered during the next revision of this International Standard.

Cold-reduced tinmill products — Electrolytic tinplate

1 Scope

This International Standard specifies requirements for single and double cold-reduced low-carbon mild steel electrolytic tinplate in the form of sheets or coils.

Single cold-reduced tinplate is generally specified in nominal thicknesses that are multiples of 0,005 mm, from 0,150 mm up to and including 0,600 mm. Double cold-reduced tinplate is generally specified in nominal thicknesses that are multiples of 0,005 mm, from 0,100 mm up to and including 0.360 mm.

This International Standard applies to coils and sheets cut from coils in nominal minimum rolling widths of $600 \text{ mm}.^{1)}$

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 404, Steel and steel products — General technical delivery requirements

ISO 4288, Geometrical Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture

ISO 6508-1, Metallic materials — Rockwell hardness test — Part 1: Test method

ISO 6892-1:—²⁾, Metallic materials — Tensile testing — Part 1: Method of test at room temperature

ISO 10474, Steel and steel products — Inspection documents

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

blackplate

cold-reduced low-carbon mild steel, applied for manufacturing electrolytic tinplate

Note 1 to entry: See ISO 11951.[1]

3.2

electrolytic tinplate

cold-reduced low-carbon steel sheet or coil coated on both surfaces with tin that is applied in a continuous electrolytic operation

3.3

differentially coated electrolytic tinplate

cold-reduced electrolytic tinplate, one surface of which carries a heavier tin coating than the other

¹⁾ Nominal minimum rolling widths of 500 mm may be applied by agreement between the purchaser and the manufacturer.

²⁾ To be published (Revision of ISO 6892-1:2009)

3.4

single cold-reduced

description of product in which the blackplate has been reduced to the desired thickness in a cold-reduction mill and subsequently annealed and temper rolled

3.5

double cold-reduced

description of product in which the blackplate has had a second major reduction after annealing

3.6

standard grade tinplate sheet

product in sheet form which is confirmed to be suitable, under normal conditions of storage, for established lacquering and printing over the entire sheet and is

- a) free from surface imperfections which render the material unsuitable for the intended use and
- b) free from damage which render the material unsuitable for the intended use

Note 1 to entry: The standard material is compliant with the requirements as specified in this International Standard.

3.7

batch annealed

box annealed

RΔ

annealed by the process in which the cold-reduced strip is annealed in coil form, within a protective atmosphere, for a predetermined time-temperature cycle

3.8

continuously annealed

CA

annealed by the process in which cold-reduced coils are unwound and annealed in strip form within a protective atmosphere

3.9

finish

surface appearance of tinplate, determined by the surface roughness, *Ra*, of the steel base together with the conditioning of the tin coating which can be either flow-melted or unflow-melted

3.9.1

smooth finish

finish of blackplate resulting from the use of temper-mill work rolls that have been ground to a low roughness

Note 1 to entry: This finish is used for the production of bright finish tinplate.

3.9.2

bright finish

finish on flow-melted tinplate using the smooth finish blackplate

3.9.3

stone finish

finish on flow-melted tinplate characterized by a directional pattern, resulting from the use of final-mill work rolls that have been ground to a higher level of roughness than those used for the smooth finish

3.9.4

silver finish

finish resulting from the use of temper-mill work rolls with dull surface textured by shot blast, electro discharge texturing (EDT), electron beam texturing (EBT) or another suitable method, together with a flow-melted tin coating

3.9.5

matt finish

finish resulting from the use of temper-mill work rolls with dull surface textured by shot blast, electro discharge texturing (EDT), electron beam texturing (EBT) or another suitable method, together with an unflow-melted tin coating

3.10

coil

rolled flat strip product which is wound into regularly superimposed laps so as to form a coil with almost flat sides

3.11

longitudinal bow

line bow

residual curvature in the strip remaining along the direction of rolling

3.12

transverse bow

cross bow

mode of curvature in the sheet such that the distance between its edges parallel to the direction of rolling is less than the sheet width

3.13

centre fullness

centre buckle

full centre

intermittent vertical displacement or wave in the strip occurring other than at the edges

Note 1 to entry: See Figure 8.

3.14

edge wave

intermittent vertical displacement occurring at the strip edge when the strip is laid on a flat surface

3.15

feather edge

transverse thickness profile

variation in thickness, characterized by a reduction in thickness close to the edges, at right angles to the direction of rolling

3.16

edge camber

deviation of edge of coil/sheet from a straight line forming its chord

3.17

burr

metal displaced beyond the plane of the surface of the strip by shearing action

3.18

rolling width

width of the rolled strip perpendicular to the direction of rolling

3.19

pallet

base platform on which a coil is placed to facilitate ready transportation

3.20

stillage platform

base platform on which sheets are stacked to facilitate packing and ready transportation

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3.21

consignment

quantity of material of the same specification made available for dispatch at the same time

3.22

bulk package

bulk

packaging unit comprising a stillage platform, the sheets and packaging material

3.23

line inspection

final inspection of the finished product performed by instruments and/or visual examination at normal production-line speeds

3.24

anvil effect

effect that a hard anvil can produce on the numerical hardness value obtained when a hardness test is performed on very thin sheet supported on such an anvil

4 General technical delivery condition

In cases where the technical delivery condition is not specified in this International Standard, then ISO 404 shall apply.

5 Classification

Steel grades for this International Standard are generally classified as non-alloy quality steels.

6 Information to be supplied by the purchaser

6.1 Designation

For the purposes of this International Standard, tinplate is designated in terms of a steel grade classification based either on the Rockwell HR30Tm hardness values or on the tensile properties. For the hardness requirements, the steel grade designations are given in <u>Table A.1</u> for single cold-reduced tinplate and in <u>Table A.2</u> for double cold-reduced tinplate. For the tensile properties requirements, the steel grade designations are given in <u>Table B.1</u>.

Tinplate covered by this International Standard shall be designated by the following characteristics in the given sequence:

- a) a reference to this International Standard, i.e. ISO 11949;
- b) the steel grade designation in accordance with Table A.1, Table A.2 or Table B.1;
- c) the type of annealing used by the manufacturer (see $\frac{7.2}{1}$);
- d) the type of finish (see $\frac{7.3}{1}$);
- e) the coating masses and their combinations, D or S(for differentially coated), together with numbers representing the nominal coating mass on top/bottom surface (see <u>Clause 12</u>);
- f) the dimensions, in millimetres:
 - for coils, thickness × width;
 - for sheets, thickness × width × length.

By agreement, the symbol "x C" after width may be designated for coils.

By agreement, the symbol "w" may be designated after the number for the width to indicate that the number is the dimension perpendicular to the direction of rolling.

EXAMPLE

Single cold-reduced tinplate sheet, in accordance with this International Standard, steel grade T61, continuously annealed (CA), stone finish, equally coated with a coating mass of 2,8 g/ m^2 , with a thickness of 0,220 mm, a width of 800 mm and a length of 900 mm shall be designated:

Double cold-reduced tinplate coil, in accordance with this International Standard, steel grade T75, continuously annealed (CA), stone finish, differentially coated with coating masses of 8.4 g/m^2 and 5.6 g/m^2 , with marking on 5.6 g/m^2 side, with a thickness of 0.180 mm and a width of 750 mm shall be designated:

Tinplate coil, in accordance with this International Standard, steel grade TH415, continuously annealed (CA), stone finish (ST), differentially coated with a coating mass of 2,8 g/m 2 and 5,6 g/m 2 , with marking on 5,6 g/m 2 side, with a thickness of 0,200 mm, a width of 750 mm shall be designated:

Tinplate sheet, in accordance with this International Standard, steel grade TS520, batch annealed (BA), stone finish, differentially coated with coating masses of 5,6 g/m 2 and 8,4 g/m 2 , with single line marked on 5,6 g/m 2 side, with a thickness of 0,140 mm, a dimension perpendicular to the direction of rolling of 844 mm and a length of 755 mm shall be designated:

6.2 Mandatory information

The following information shall be given in the enquiry and order to assist the manufacturer in supplying the correct material:

- a) the designation as given in 6.1;
- b) the quantity, expressed on an area or mass basis, e.g. 50 tons of sheets, 100 tons of coils;
- c) marking requirements for differentially coated tinplate (see <u>Clause 12</u>);
- d) the minimum and the maximum coil weight, the minimum and the maximum coil outer diameter, the coil internal diameter, the core vertical or horizontal and the direction of winding (see 17.1);
- e) the maximum weight of bulk package;
- f) other inspection document than that specified by the manufacturer (see <u>Clause 16</u>);
- g) end use;
- h) any further special requirements.

NOTE Appropriate steel grade is suitable for shaping operations such as stamping, drawing, folding, beading and bending, and assembly work such as joint forming, soldering and welding. The end use is important when the steel grade is selected.

6.3 Options

In addition to the information in <u>6.2</u>, the purchaser may wish to provide additional information to the manufacturer to ensure that the order requirements are consistent with the end use of the product.

The purchaser shall inform the manufacturer of any modifications to his/her fabrication operations that will significantly affect the way in which the tinplate is used.

NOTE When ordering double cold-reduced tinplate, the purpose of manufacture for which the material is intended is generally stated. It is noted that double cold-reduced tinplate is relatively less ductile than single cold-reduced tinplate and has very distinct directional properties, so for some uses, for example for built-up can bodies, the direction of rolling is usually stated. When double cold-reduced tinplate is used for built-up can bodies, the direction of rolling is around the circumference of the can so as to minimize the hazard of flange cracking.

7 Manufacturing features

7.1 Manufacture

Continuously cast, fully-killed steel is applied except when otherwise specified. The examples of the steel types of tinplate are shown in $\underline{\mathsf{Annex}\,\mathsf{C}}$.

The steel type of tinplate shall be designed to secure food safety when tinplate is used for food application. The purchasers should be aware of existing national regulations which may impose limitations on some elements.

The purity of tin used to produce the coating shall be not less than 99,85 %(mass fraction).

The manufacturing method of tinplate is left to the discretion of the manufacturer and is not specified in this International Standard.

7.2 Annealing

Annealing of tinplate shall be either batch annealing (BA) or continuous annealing(CA) and shall be specified by the purchaser at the time of enquiry and order.

7.3 Finish

Tinplate is usually available in the finishes as indicated in $\underline{\text{Table 1}}$. The type of finish is designated either by the tinplate finish or the code shown in $\underline{\text{Table 1}}$.

		Bl		
Tinplate	a. T Louea		Surface roughness ^{bc}	
		Finish	Ra	ed
			μm	
Bright	ВТ	Smooth	≤0,35	Yes
Fine stone	FS	Fine stone	0,25 - 0,45	Yes
Stone	ST	Stone	0,35 - 0,60	Yes
Silver	AM	Matt	≥0.90	Yes
Matt	MM	Matt	20,90	No

Table 1 — Typical finishes for tinplate

NOTE 1 The appearance is governed by the following:

a) the surface characteristics of the blackplate principally result from controlled preparation of the work rolls used during the final stages of temper rolling;

By agreement between the purchaser and the manufacturer, another code system may be applied.

b Values of surface roughness in this table are not mandatory. The values are given for reference in order to classify the finishes.

The measurement of surface roughness is in accordance with ISO 4288.

- b) the mass of the coating applied;
- c) whether the tin layer is flow-melted or unflow-melted.

NOTE 2 Double cold-reduced tinplate is usually supplied with a stone finish and a flow-melted tin coating.

7.4 Passivation and oiling

The surface of electrolytic tinplate is normally subjected to a passivation treatment and to oiling. Passivation, produced either by a chemical or an electrochemical treatment, gives a surface with an improved resistance to oxidation and improved suitability for lacquering and printing. Unless otherwise agreed at the time of ordering, the usual passivation procedure is a cathodic treatment in an acidic chromate solution. The typical range of this passivation is generally up to 10 mg/m^2 for each side.

Under normal conditions of transport and storage, electrolytic tinplate shall be suitable for surface treatments such as established lacquering and printing operations.

Tinplate coils and sheets are supplied with an oil coating. The oil shall be one that is recognized (i.e. by the relevant national or international authority) as being suitable for food packaging. Unless otherwise agreed at the time of ordering, the kind of oil is at the discretion of the manufacturer.

NOTE 1 For the oil, dioctyl sebacate (DOS) is usually used.

NOTE 2 For measuring chromium of passivation film, the diphenylcarbazide method or the X-ray flourescence spectrometric method is usually applied. When the X-ray method is applied, it is necessary to consider the influence of chromium in base metal.

7.5 Imperfections

7.5.1 Coils

The manufacturer is expected to employ his/her normal quality control and line inspection procedures to ensure that the tinplate manufactured is in accordance with the requirements of this International Standard.

However, the production of tinplate coils in continuous-strip mill operations does not afford the opportunity for removal of all tinplate that do not comply with the requirements of this International Standard.

At the time of shearing, sheets not conforming to the standard grade shall be set aside by the purchaser or his/her agent.

The quantity of sheets complying with this International Standard shall be at least 90 % of any one coil.

List items a) and b) in 3.6 cannot be verified by specific tests. Accordingly, those items are recommended to be the subject of a special agreement between the purchaser and the manufacturer.

In processing tinplate coil, when the purchaser (or his/her agent) encounters recurring imperfections which in his/her opinion seem excessive, it is essential, where practicable, that he/she stops processing the coil and advises the manufacturer.

The purchaser is expected to have adequate handling, roller levelling and shearing equipment and inspection facilities to segregate the sheets not conforming to the standard grade, and to take reasonable care during these operations.

7.5.2 Sheets

Sheets shall not contain any imperfections as defined in <u>3.6</u>.

8 Tin coating mass

The nominal coating mass, on each surface, shall be expressed in grams per square metre. The lowest value specified in this International Standard shall be $1.0~g/m^2$ on each surface, and no upper limit is specified. Values of preferred coating masses are $1.0~g/m^2$, $1.4~g/m^2$, $2.0~g/m^2$, $2.8~g/m^2$, $4.0~g/m^2$, $5.0~g/m^2$, $5.0~g/m^2$, $8.4~g/m^2$ and $11.2~g/m^2$.

Whatever the coating mass used, the coating mass for sample average and for individual sample shall not be less than the minimum coating mass indicated in <u>Table 2</u>. The coating mass for sample average is the average of three measurements on each sheet selected. The mass per unit area for equally and differentially coated tinplate is determined on test pieces taken from samples selected in accordance with <u>Clause 13</u> and tested in accordance with <u>14.1</u>.

Range of nominal coating mass, <i>m</i> , on each surface	Minimum coating mass for sample average	Minimum coating mass for individual sample
g/m ²	g/m ²	g/m ²
1,0 ≤ <i>m</i> < 2,8	$0.80 \times m$	0,64 × m
2,8 ≤ <i>m</i> < 5,6	0,87 × m	0,70 × m
5,6 ≤ <i>m</i>	0,90 × m	0,72 × m

Table 2 — Minimum tin coating masses

NOTE The minimum coating mass for sample average and for individual sample shall be rounded to the unit of $0.1~g/m^2$.

9 Mechanical properties

9.1 General

For the purposes of this International Standard, tinplates are classified into steel grades based on either Rockwell HR30Tm hardness values or tensile properties. The purchaser shall indicate the specification either by hardness requirement or by tensile properties requirement, but not for both, when ordering the material.

When ordering the material for applications such as drawn cans, DWI cans, twist off caps, etc., it is recommended to indicate the specification according to the tensile property requirement.

Other mechanical properties might significantly influence the performance of tinplate in processing, and the subsequent intended end use might vary depending on the steel type and the method of casting, annealing and temper rolling employed.

At the time of enquiry and order it shall be agreed that properties of steel grade are to be verified either by the hardness test or by the tensile test.

9.2 Hardness requirement

The hardness values for tinplate shall be as given in <u>Table A.1</u> and <u>Table A.2</u>, when tested as described in <u>14.2</u>.

9.3 Tensile property requirement

The proof strength, $R_{\rm p0.2}$, for tinplate shall be as given in Table B.1, when tested as described in 14.3.

For routine testing, the proof strength may be determined using the springback test as described in <u>Annex D</u>. However, in cases of dispute, the method described in <u>14.3</u> shall be applied.

10 Tolerances on dimensions and shape

10.1 General

Tolerances on dimensions (i.e. thickness, width and length) and shape (i.e. edge camber, out-of-squareness and flatness) are specified in 10.2 to 10.7, together with appropriate methods of measurement.

10.2 Thickness and feather edge

10.2.1 Thickness

Nominal thickness shall be a multiple of 0,005 mm. Nominal thickness other than multiple of 0,005 mm may be specified by agreement between the purchaser and the manufacturer. Thickness out of the nominal thickness range may be specified by agreement between the purchaser and the manufacturer.

The thickness of tinplate shall not deviate from the ordered nominal thickness by more than +5, -8 % at any point except within 10 mm from the trimmed-edge.

The thickness shall be measured using a hand-operated, spring-loaded micrometer to an accuracy of 0,001 mm.

It is recommended that the micrometer should have a ball-ended shank and a curved-surface base anvil.

10.2.2 Feather edge

For both sheet and coil, the thickness when measured at a distance of 10 mm from the mill trimmed edge shall not deviate from the actual centre thickness by more than -6 %.

10.3 Width

The width of tinplate shall be measured to the nearest 0,5 mm at right angles to the direction of rolling.

For the products of this International Standard which are delivered with trimmed-edge, the measured width shall not deviate from the ordered width by more than +3, -0 mm.

10.4 Length

10.4.1 Length of coil

The difference between the actual length and the manufacturer's indicated length, measured on any single coil, shall not exceed by more than ±3 %, unless otherwise agreed.

10.4.2 Length of sheet

The length of sheet shall be measured to the nearest 0.5 mm. The measured length shall not deviate by more than +3, -0 mm from the ordered length.

10.5 Edge camber

Edge camber is the maximum deviation (in the plane of the sheet) of an edge from a straight line forming a chord to its extremities (see Figures 1 and 2).

The edge camber, *E*, expressed as a percentage of the chord length, is calculated using Formula (1):

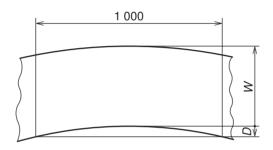
$$E = \frac{D}{L} \times 100 \tag{1}$$

where

- *D* is the deviation from a straight line, in millimetres;
- *L* is the length of chord, in millimetres.

For coils, the edge camber, measured over a distance (chord length) of 1 000 mm, shall not exceed 0.1% (i.e. $1\ mm$).

Dimensions in millimetres



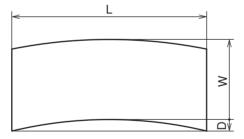
Key

W width

D deviation from a straight line

Figure 1 — Edge camber of coil

For sheets, the edge camber, measured over a chord length, shall not exceed 0,1 %.



Key

D deviation from a straight line

L length of chord

W Width

Figure 2 — Edge camber of sheet

10.6 Out-of-squareness of sheet

Out-of-squareness is the deviation of an edge from a straight line drawn at a right angle to the other edge of the sheet, touching one corner and extending to the opposite edge (see Figure 3).

The out-of-squareness, O_s , expressed as a percentage, is calculated using Formula (2):

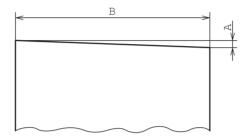
$$O_{\rm S} = \frac{A}{B} \times 100 \tag{2}$$

where

A is the deviation:

B is the length or width of the sheet measured at a right angle to an edge.

For each sheet in the sample, the out-of-squareness shall not exceed 0,20 %.



Key

A deviation

B length or width of the sheet measured at a right angle to an edge

Figure 3 — Out-of-squareness of sheet

10.7 Flatness

10.7.1 Edge wave

The height of edge wave, h_{ew} , at any point shall not exceed 2,5 mm, when tested as described in 14.4.2. No more than six waves in excess of 1,5 mm shall be present over a cut length of 1 m for coil or proportional for sheet.

10.7.2 Longitudinal and transverse bow

Bow may be either convex or concave face uppermost on the bulk package. The normal convention is to express convex bow uppermost as a positive (+) value and concave bow as a negative (-) value.

The individual values of both longitudinal and transverse bow in levelled condition shall not exceed 30 mm, when tested as described in 14.4.3. Where both convex and concave bow are present in the same coil, the sum of the maximum values of each, ignoring the sign (±), shall not exceed 30 mm. In case of unlevelled sheet from coil before cutting, the requirement of bow may be agreed between the purchaser and the manufacturer.

10.7.3 Centre fullness

Centre fullness shall be determined by either the direct method as described in $\underline{14.4.4.1}$ or the indirect method as described in $\underline{14.4.4.2}$. The selection of the method is at the discretion of the manufacturer. In case of the direct method, the height of centre fullness, h_{cf} , shall not exceed 5 mm when tested as described in $\underline{14.4.4.1}$. In case of the indirect method, the height of centre fullness, h_{if} , shall not exceed 9 mm when tested as described in $\underline{14.4.4.2}$.

NOTE Centre fullness is not clearly visible in a coil but usually becomes apparent during either printing or slitting.

11 Joint within a coil

11.1 General

The manufacturer shall ensure continuity of the coils within the limits of the lengths ordered, if necessary, by means of electrically welded joints made after cold reduction. Requirements relating to the numbers, locations and dimensions of the joints permitted within a coil are given in 11.2 to 11.4.

11.2 Number of joint

The number of joint in a coil shall not exceed three in lengths of 10 000 m.

11.3 Location of joint

The location of each joint in a coil shall be indicated clearly.

The location of each joint may be indicated, for example, by the insertion of a piece of non-rigid material and punched holes. However, alternative methods may be agreed between the purchaser and the manufacturer at the time of enquiry and order.

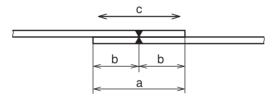
11.4 Dimension of joint

11.4.1 Thickness

The total thickness of any joint shall not exceed three times the nominal thickness of the material forming the joint.

11.4.2 Overlap

In any lap joint, the total length of overlap shall not exceed 10 mm. The free overlap shall not exceed 5 mm (see Figure 4).



Key

- a total length of overlap
- b free overlap
- c direction of rolling

Figure 4 — Joint overlap

12 Marking of differentially coated cold-reduced tinplate

12.1 General

In order to distinguish between tinplate with differential tin coatings and tinplate with equal coatings on the two surfaces, differentially coated tinplate shall be marked on one surface only, unless otherwise agreed. The surface to be marked shall be agreed by the purchaser and the manufacturer.

If no option is indicated at the time of ordering, the marking shall be in accordance with 12.2.

12.2 Marking on heavily coated surface

On heavily coated surface, unless otherwise specified, the marking shall be by dull, straight, continuous parallel lines up to 1 mm wide and spaced at 75 mm intervals. By agreement, the alternative parallel straight lines shown in $\underline{\textbf{E.1}}$ may be applied.

E.1 gives details of an alternative marking system for certain coating combinations only.

12.3 Marking on lightly coated surface

On lightly coated surface, unless otherwise specified, the marking shall be by alternate interrupted, dull, straight, parallel lines spaced at 75 mm intervals. By agreement, the marking system applied may be

- a) by geometric patterns shown in E.2, or
- b) by the single line at one edge of coil.

E.2 gives examples of geometric patterns.

12.4 Marking designation

The symbol "D" or "S" shall be put in front of number of coating mass to indicate the marked surface. The symbol "S" shall be for the single line at one edge of coil on the lightly coated surface [see $\underline{12.3}$ b)], and the symbol "D" shall be for all the other marks.

The following examples show how to indicate the marked side and its position when ordering.

- D2,8/5,6: marked on the 2,8 coated side. The marks are to be on the top of sheets or on the outside
 of the coil.
- D5,6/2,8: marked on the 5,6 coated side. The marks are to be on the top of sheets or on the outside
 of the coil.
- 2,8/D5,6: marked on the 5,6 coated side. The marks are to be on the bottom of sheets or on the inside of the coil.
- 5,6/D2,8: marked on the 2,8 coated side. The marks are to be on the bottom of sheets or on the inside of the coil.
- S2,8/5,6: the single line at one edge of coil marked on the 2,8 coated side. The marks are to be on the top of sheets or on the outside of the coil.
- 5,6/S2,8: the single line at one edge of coil marked on the 2,8 coated side. The marks are to be on the bottom of sheets or on the inside of the coil.

13 Sampling

For certifying the quality of product, the manufacturer shall take samples according to <u>Figure 5</u> and carry out test. One sheet for test specimen shall be taken for every 30 tons or less and remainder thereof of the same properties, i.e. steel grade, dimensions and coating mass.

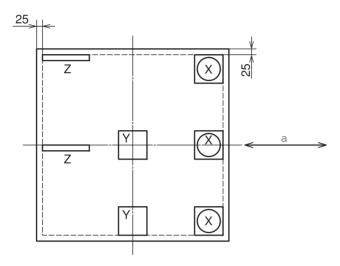
14 Test method

14.1 Tin coating mass

14.1.1 Test piece

From each sheet selected in accordance with <u>Clause 13</u>, three test pieces shall be carefully prepared. These test pieces shall be selected at edge-centre-edge locations (positions X on <u>Figure 5</u>) along a

line normal to the direction of rolling. Ensure that the edge test pieces clear the edges of the sheet by a minimum of 25 mm. A test piece shall be a disc or a square having an area of approximately 2 500 mm². However, in the case of the fluorescent X-ray spectrometric method, the irradiation area shall be 314 mm² or over.



Key

- X test pieces for the coating mass
- Y test pieces for hardness
- Z test pieces for tensile test
- a direction of rolling

Figure 5 — Location of test pieces

14.1.2 Method of determination

The tin coating mass shall be expressed in grams of tin per square metre to the nearest 0.1 g/m^2 .

For routine quality control purposes, the coating masses may be determined by any of the recognized and acceptable analytical and instrumental methods. In cases of dispute, the method described in $\underbrace{Annex\ F}$ shall be the referee method.

Whether tin coating determinations are made on individual or grouped test pieces, the tin coating mass of a consignment shall be taken as the average of all the results.

14.2 Hardness test

14.2.1 Test piece

The hardness tests shall be carried out prior to lacquering or printing.

From each of the sample sheets obtained in accordance with <u>Clause 13</u>, take two test pieces from the positions marked Y on <u>Figure 5</u>.

Before carrying out the hardness tests in accordance with $\underline{14.2.2}$, artificially age the test pieces at 200 °C for 20 min without removing the tin coating. The artificial aging may not be necessary for nonaging materials.

When necessary, the surface shall be finished with fine emery paper.

14.2.2 Test method

Determine the Rockwell HR30Tm indentation hardness either

- a) directly, in accordance with ISO 6508-1, or
- b) indirectly, on relatively thin sheets (e.g. 0,22 mm and thinner), by determining the HR15Tm hardness in accordance with ISO 6508-1 and then converting the HR15Tm values to HR30Tm values using $\underbrace{\text{Annex G}}$.

By agreement, the hardness may be determined either by HR30Tm or HR15Tm for the sample thickness between 0,20 mm and 0,22 mm.

Make three hardness measurements on each of the test pieces taken in accordance with 14.2.1. Calculate the representative hardness for the consignment as the arithmetic mean of all the hardness measurements on all the sample sheets taken from the consignment.

To measure the indentation hardness, use a Rockwell superficial hardness testing machine, employing the 30Tm or 15Tm scales specified in ISO 6508-1 with a hardened steel ball indenter, as appropriate.

Avoid testing near the edges of the test pieces because of a possible cantilever effect.

14.3 Tensile test

14.3.1 Test piece

For each sheet selected in accordance with <u>Clause 13</u>, cut two rectangular test pieces with the direction of rolling parallel to the length of the test piece, at the position marked Z on <u>Figure 5</u>. Ensure that the edge test pieces clear the edges of the sheet by a minimum of 25 mm. Before carrying out the tensile test described in <u>14.3.2</u>, artificially age the test pieces at 200 °C for 20 min without removing the tin coating. The artificial aging may not be necessary for non-aging materials.

14.3.2 Test method

Determine the 0.2% proof strength as described in ISO 6892-1 using the conditions specified in ISO 6892-1:—, Annex B for thin products.

Carry out one test on each of the test pieces selected in accordance with $\underline{14.3.1}$, i.e. two tests per sheet selected.

Calculate the representative proof strength for the consignment as the arithmetic mean of all the results on all the sample sheets taken from the consignment.

14.4 Flatness test

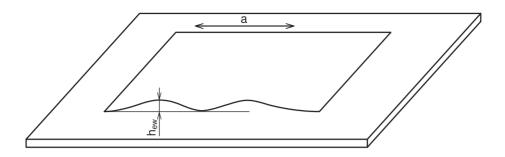
14.4.1 General

The method of measuring flatness is at the discretion of the manufacturer. However in cases of dispute, the following method shall be applied as a referee method.

14.4.2 Edge wave

Each sample shall be laid on a flat horizontal surface which is larger than the sample itself. The height of edge wave, $h_{\rm ew}$, shall be given as the feeler gauge diameter that just fits under the wave at the edge of the sample.

Heights of edge wave, h_{ew} , shall be determined by using feeler gauges of standard diameters in increments of 0,25 mm (Figure 6).



Key

hew edge wave

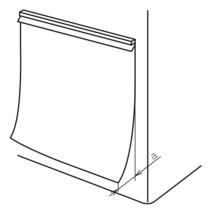
a direction of rolling

Figure 6 — Edge wave

14.4.3 Longitudinal or transverse bow

The maximum value of longitudinal or transverse bow shall be determined by hanging the sample from one horizontal edge against a rigid vertical surface, noting whether the upper or lower surface is against the vertical surface, so that the bow causes the bottom edge of the sample to stand away from that surface. When selecting the sample it is necessary to identify the outer and inner face of the coil.

The sample shall be evenly supported along the top to a depth not exceeding 25 mm from the edge. The maximum distance, the bottom edge stands away from the vertical (value *a* on Figure 7), is measured with a steel ruler to the nearest 1 mm and recorded with the appropriate plus or minus sign indicating convex or concave bow, respectively.



Key

a maximum distance between the bottom edge and the vertical

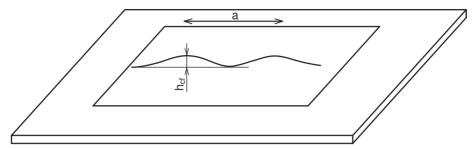
Figure 7 — Longitudinal or transverse bow

14.4.4 Centre fullness

14.4.4.1 Direct method

The sample sheet shall be laid on a flat and horizontal surface which is larger than the sample. A rigid, flat and straight bar, which is supported by two rigid blocks with the same and proper height, shall be set over the sample to be approximately right above the wave of the centre fullness and parallel to the rolling direction (see Figure 9).

Both distances at the points of the top and the bottom of the wave from the lower edge of the bar shall be measured, respectively. The height of centre fullness (value h_{cf} on Figure 8) shall be determined as the difference between these two values (values h_1 and h_2 on Figure 9).

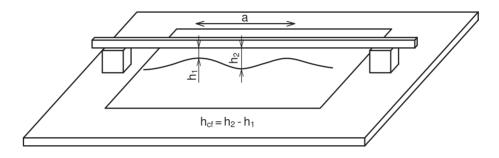


Key

*h*_{cf} centre fullness

a direction of rolling

Figure 8 — Definition of centre fullness



Key

a direction of rolling

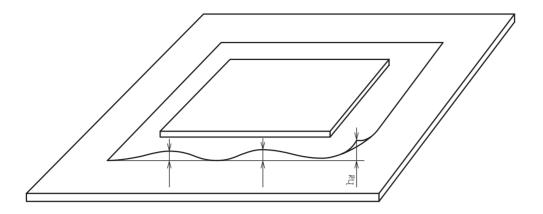
Figure 9 — Method of measuring centre fullness (direct method)

14.4.4.2 Indirect method

The sample sheet shall be laid on a flat and horizontal surface which is larger than the sample. A rigid and flat board shall be placed on the centre of the sample.

The board shall be moved around the surface of the sample centre until a position giving the highest edge lift can be identified. Pressure shall then be applied to the board so as to flatten the sample in the centre and raise the edge to a maximum height. During the test, the board shall not overlap the edges to be measured.

Edge lift shall be determined by using a 9 mm diameter feeler gauge and the product is deemed out of specification when the feeler gauge fits under the edge of the sheet at the point of maximum lift (value h_{if} on Figure 10).



Key

 $h_{\rm if}$ maximum lift of the edge

Figure 10 — Method of measuring centre fullness (indirect method)

15 Retests

If any of the results obtained are unsatisfactory for coating mass and mechanical properties, the manufacturer may either withdraw the test unit or perform retest. In case of retest, the measurements for that particular property shall be repeated twice on new samples, on each occasion using the sampling specified in <u>Clause 13</u>. If the results or both repeated tests meet the stated requirements the consignment represented shall be deemed to comply with this International Standard, but if the results of either of the retests fail to meet the stated requirements, the consignment represented shall be deemed not to comply with this International Standard.

16 Inspection document

Tinplate complying with this International Standard shall be ordered and delivered with one of the inspection documents specified in ISO 10474. The type of document shall be agreed upon at the time of enquiry and order. Unless otherwise specified, the type of document shall be at the discretion of the manufacturer.

17 Dispatch and packaging

17.1 Coils

Unless otherwise requested at the time of ordering, coils shall be dispatched with their cores in a vertical position (the other option would be with the cores horizontal). The internal diameters of the coils shall be either (420^{+10}_{-15}) mm or (508^{+10}_{-15}) mm.

Tinplate strip is usually supplied in consignments of coils with outside diameters of at least 1 200 mm, but a limited number of coils with smaller outside diameters may be included in the consignment.

The manufacturer shall state the direction of winding in the coils to ensure that the correct surface is maintained throughout manufacture. Where coils are supplied with cores vertical (the normal method of delivery) the purchaser shall specify the required direction of winding (see Figure 11).

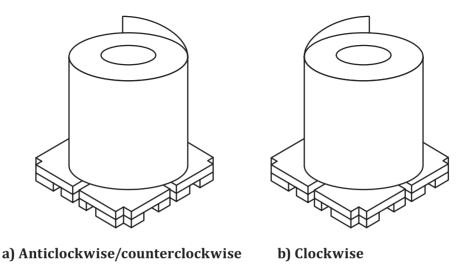


Figure 11 — Direction of coil winding

17.2 Sheets

The sheets shall be supplied in bulk packages in which the numbers of sheets are multiples of 100.

The direction of the runners of the stillage platform shall be consistent within a consignment.

The sheets are customarily packed on a stillage platform forming a bulk package weighing approximately between 1 000 kg and 2 000 kg. A limited number of bulk packages of mass less than 1 000 kg may be included in a consignment.

If the purchaser has any preference for the direction of the runners of the stillage platform, his/her requirements should be agreed with the manufacturer and stated on the order. If the purchaser does not indicate his/her wish, the direction is at the discretion of the manufacturer.

17.3 Labelling

The products shall be marked by a label with the following items:

- a) the manufacturer's trademark or symbol;
- b) the steel grade;
- c) the dimensions;
- d) the identification number related to an appropriate inspection certificate.

Annex A

(normative)

Hardness requirements for tinplate

A.1 Hardness requirement for single cold-reduced tinplate

The hardness values for single cold-reduced tinplate shall be as given in $\underline{\text{Table A.1}}$, when tested as described in $\underline{\text{14.2}}$.

Table A.1 — Hardness values (HR30Tm) for single cold-reduced tinplate

Steel g	rade	Hardness		
Jicei g.	lauc	HR30Tm		
	Alternative designation	Thickness, t		
Designation		mm		
		<i>t</i> ≤ 0,21	$0,21 < t \le 0,28$	0,28 < t
T49	T-1	50 ± 4	49 ± 4	48 ± 4
T53	T-2	54 ± 4	53 ± 4	52 ± 4
T55	T-2.5	56 ± 4	55 ± 4	54 ± 4
T57	T-3	58 ± 4	57 ± 4	56 ± 4
T59	T-3.5	60 ± 4	59 ± 4	58 ± 4
T61	T-4	62 ± 4	61 ± 4	60 ± 4
T65	T-5	66 ± 4	65 ± 4	64 ± 4

A.2 Hardness requirement for double cold-reduced tinplate

The hardness values for double cold-reduced tinplate shall be as given in <u>Table A.2</u>, when tested as described in <u>14.2</u>.

Table A.2 — Hardness values (HR30Tm) for double cold-reduced tinplate

Steel g	Hardness	
Designation	Alternative designation	HR30Tm
T71	DR-7.5	71 ± 4
T72	DR-8	72 ± 4
T73	DR-8.5	73 ± 4
T75	DR-9	75 ± 4
T76	DR-9M	76 ± 4

Annex B

(normative)

Tensile property requirements for tinplate

The proof strength, $R_{p0,2}$, for tinplate shall be as given in <u>Table B.1</u>, when tested as described in <u>14.3</u>.

For routine testing, the proof strength may be determined using the springback test as described in <u>Annex D</u>. However, in cases of dispute, the method described in <u>14.3</u> shall be applied.

Table B.1 — Tensile properties for tinplate

Chaol and do	Ammodina	$R_{p0,2}^{a}$	Deviation ^b
Steel grade	Annealing	МРа	MPa
TS200	BA	200	±50
TS230	BA	230	±50
TS245	BA	245	±50
TS260	BA	260	±50
TS275	BA	275	±50
TS290	BA	290	±50
TS340	BA	340	±50
TS480	BA	480	±50
TS520	BA	520	±50
TS550	BA	550	±50
TS580	BA	580	±50
TS620	BA	620	±50
TH230	CA	230	±50
TH245	CA	245	±50
TH260	CA	260	±50
TH275	CA	275	±50
TH300	CA	300	±50
TH330	CA	330	±50
TH350	CA	350	±50
TH385	CA	385	±50
TH400	CA	400	±50
TH415	CA	415	±50
TH435	CA	435	±50
TH450	CA	450	±50
TH480	CA	480	±50
TH520	CA	520	±50
TH550	CA	550	±50
TH580	CA	580	±50

Table B.1 (continued)

Steel grade	Annealing	$R_{p0,2}^{a}$	Deviation ^b
Steer grade		MPa	МРа
TH620	CA	620	±50
TH650	CA	650	±50

NOTE 1 Steel grades TS480 may be delivered in either single or double reduced form. Steel grades TS520/TH520, TS550/TH550, TS580/TH580, TS620/TH620 and TH650 are usually delivered in double reduced form only. All other grades are delivered in single reduced form.

NOTE 2 The deviations shown refer to measurements of individual samples.

 $^{^{\}rm a}$ $R_{\rm eL}$ or $R_{\rm eH}$ may be applied for steels that show a yield point elongation by agreement between the purchaser and the manufacturer.

 $^{^{\}rm b}$ In case that the deviation is out of ± 50 MPa, tinplate may be supplied by agreement between the purchaser and the manufacturer.

Annex C (informative)

Steel types

The chemical composition of blackplate determined by cast analysis should be less than the following maximum values (% mass fraction): C: 0,13, Si: 0,03, Mn: 0,60, P: 0,020 and S: 0,030.

The following are examples of steel types.

- a) steel type MR: base steel, low in residual elements that has corrosion resistance, widely used in general applications.
- b) steel type L: base steel, extremely low in residual elements that has excellent corrosion resistance to certain types of food can.
- c) steel type D: base steel, low in residual elements that has corrosion resistance, involving deep drawing or other types of severe forming that tend to give rise to Lueder's lines.

The choice of a suitable physical or chemical analytical method for the analysis shall be at the discretion of the manufacturer. In cases of dispute, the method for analysis used shall be agreed taking into account the relevant existing International Standards.

NOTE The list of available International Standards on chemical analysis is given in ISO/TR 9769.[2]

Annex D

(informative)

Springback test for routine determination of proof strength for tinplate

D.1 General

The test described in this annex is not the reference method. In cases of dispute, the method described in 14.3 (i.e. ISO 6892-1) shall be applied.

D.2 Principle

This test provides a simple and rapid means of estimating the proof strength of double cold-reduced tinplate from measurement of thickness and angle of springback of a rectangular strip test piece, after bending through 180° around a cylindrical mandrel and then releasing.

D.3 Test piece

The test pieces used are identical to those for tensile test described in 14.3.1

D.4 Test method

Make one test on each of the test pieces obtained in accordance with 14.3.1 (i.e. two tests per sheet selected).

In making the test, strictly observe the operational instructions provided with the springback tester. The principal steps in the test are the following:

- a) measure the thickness of the tinplate test piece, to the nearest 0,001 mm;
- b) insert the test piece into the tester and fix it firmly in the testing position by gently tightening the clamping screw using light finger pressure;
- c) bend the test piece through an angle of 180° around the mandrel by a gentle swing of the forming arm;
- d) return the forming arm to its "start" position and read and record the springback angle by sighting directly over the test piece;
- remove the test piece from the tester and, using the recorded thickness of the test piece and the springback angle, determine the appropriate springback index value from a suitable conversion formula (e.g. Bower) agreed between the purchaser and the manufacturer.

Calibrate each new springback tester using the standard tensile test (see 14.3) or another "reference" springback tester. In addition, since malfunctions arising, for example, from excessive wear or inadvertent abuse of the test equipment, may not be readily apparent, it is recommended that the springback test readings be regularly compared with readings from the standard tensile test or a "reference" springback tester. It is also recommended that such direct cross-checks be further supplemented by the frequent use of reference samples of known proof stress.

Annex E

(informative)

Alternative marking system for differentially coated tinplate

E.1 Alternative marking system on heavily coated surface

The marking system consists of parallel straight lines up to 1 mm wide, the distance between the lines indicating the coating masses.

The following spacings should be used:

Designation	Line spacing
D5,6/2,8	12,5 mm
D8,4/2,8	25 mm
D8,4/5,6	25 mm alternating with 12,5 mm
D11,2/8,4	37,5 mm alternating with 25 mm
D11,2/2,8	37,5 mm
D11,2/5,6	37,5 mm alternating with 12,5 mm

An illustration of the marking system is given in Figure E.1.

This alternative marking system may be used for certain coating combinations only.

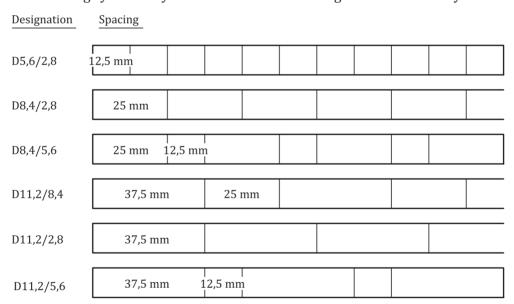


Figure E.1 — Alternative marking system on heavily coated surface

E.2 Marking system by geometric patterns on lightly coated surface

The following marking system on lightly coated surface may be applied by agreement (see Figure E.2).

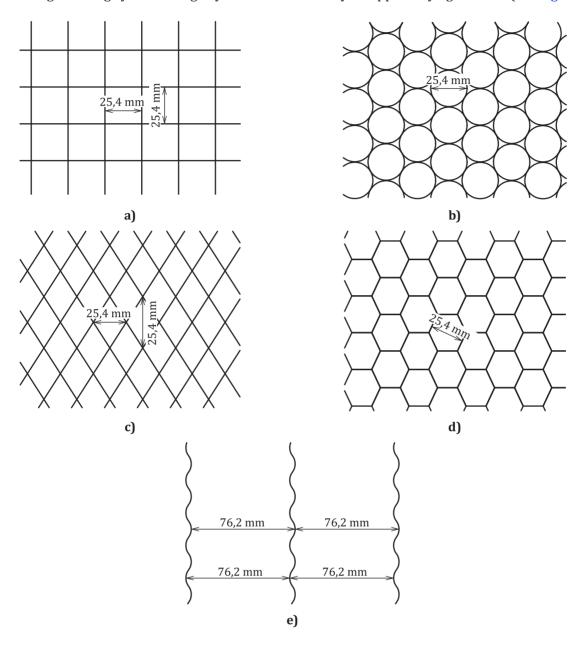


Figure E.2 — Marking system by geometric patterns on lightly coated surface

Annex F

(normative)

Referee method for determining tin coating mass

F.1 General

For determination of tin coating mass, the referee method shall be one of the following methods shown in $\underline{F.2}$ and $\underline{F.3}$. In cases of dispute, the method should be agreed between the purchaser and the manufacturer.

E.2 Electrochemical method

F.2.1 Principle

The tin and tin/iron alloy layers are stripped electrochemically in hydrochloric acid at constant current. Faraday's Law is used to calculate the mass of tin removed from the time taken to strip each layer. The effective range of the method is from 0.5 g/m^2 up to 50 g/m^2 and the reproducibility is 0.1 g/m^2 .

F.2.2 Reagent

Hydrochloric acid: 5 %.

Add 135 ml of concentrated hydrochloric acid (1,18 g/ml) to 500 ml of distilled water, mix well, and make up to 1 l.

F.2.3 Apparatus

- **F.2.3.1** Cell and electrodes, sample holder of the type shown in Figure F.1, which exposes at least 1 cm², a platinum gauze or platinized titanium cathode and a suitable reference electrode (e.g. standard calomel electrode). While the method is accurate down to a 1 cm² exposed area, a minimum of 2,5 cm² typically is adopted for control.
- **F.2.3.2 Power supply**, capable of delivering a constant current in the range 3 mA to 100 mA.
- **F.2.3.3 Voltmeter**, with a full-scale of 0 V to \pm 2 V, and from which an output can be taken to a y-t recorder. One terminal of the voltmeter is connected to the sample, and the other to the reference electrode. A potentiostat and computerized data acquisition and plotting system may be used to perform the functions of both the power supply and voltmeter/y-t recorder.

F.2.4 Procedure

Wash the sample with acetone and allow to dry in air. Mount in the sample holder and add sufficient 5 % hydrochloric acid to cover the cathode. From the known exposed area of the sample, calculate the current required to maintain a current density of 4 mA/cm², and set the power supply to this current. Switch on the power supply and observe the voltmeter and voltage/time curve on the y-t recorder. Two large jumps in voltage should be observed, the first indicates the end of tin dissolution, and the second the end of alloy dissolution.

F.2.5 Calibration of the measuring system

Take a strip from the middle of the produced material ensuring it is cut with the rolling direction. Cut 36 adjacent samples in the form of disks with an area of 2 500mm² from the strip; number each one. Measure the even numbered samples using the electrochemical method below. Use the odd numbered samples for the wet chemical titration method.

F.2.6 Calculation

From the voltage/time curve, determine t_1 and t_2 , the times of the steepest points of the two jumps in voltage, as in Figure F.2.

The times at which the two steep rises in potential are quantified as follows.

The end points are determined by the intersection of the tangential lines to the centres of the potential climbing curves with the horizontal extrapolated plateaux of the potential curve. From these points perpendicular lines are drawn to the basic, as in Figure F.2. At this line, the seconds are read t_1 (free tin) and t_2 (tin in alloy) are measured.

The coating masses of free tin, and tin in the alloy are calculated from Faradays Law; coating mass, m_C , (g/m^2) as given by Formula (F.1):

$$m_{\rm C} = \frac{m_{\rm a} lt}{nF} \tag{F.1}$$

where

 m_a is the atomic mass of tin, 118,69 g/mol;

- *l* is the current density in A/m^2 (4 mA/cm² = 40 A/m²);
- t is the time t_1 or t_2 in seconds, determined as above;
- *n* is the number of the electrons involved in the electrochemical reactions;

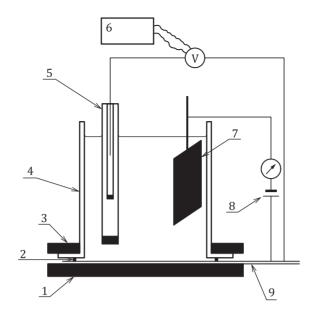
free tin Sn
$$\rightarrow$$
 Sn²⁺ + 2e $n = 2$ alloy 1/2FeSn₂ \rightarrow Sn²⁺ + 1/2Fe²⁺ + 3e $n = 3$

F is Faraday's constant 9,648 46×10^4 C/mol.

These equations reduce to the following:

- free tin $(g/m^2) = 0.024 6 t_1$
- tin in alloy $(g/m^2) = 0.016 4(t_2 t_1)$

The total coating mass is the sum of these two amounts.

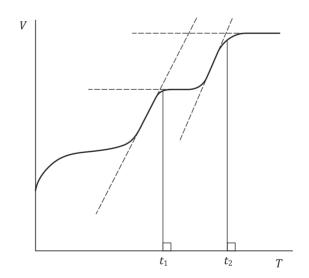


Key

- 1 cell base plate
- 2 O-ring
- 3 cell clamping plate
- 4 flange cell
- 5 reference electrode

- 6 y-t recorder
- 7 cathode
- 8 DC constant current power supply
- 9 tinplate sample

Figure F.1 — Apparatus and circuit for tin coating mass determination



Key

- V voltage, in millivolts
- T time, in seconds

 $\label{eq:FigureF.2} Figure F.2 — Typical response from tin coating mass measurement showing end point determination$

F.3 Volume metric method

F.3.1 Principle

The tin coating is dissolved in hydrochloric acid and the tin in an aliquot is reduced to the bivalent state with metallic aluminium. The tin in the reduced state is determined by titration with potassium iodate standard solution.

The effective range of the method is from 0.5 g/m^2 up to 50 g/m^2 and the reproducibility is $\pm 0.1 \text{ g/m}^2$.

F.3.2 Reagents and materials

During the analysis, use only reagents of recognized analytical grade and only distilled water.

Freshly prepare and, where necessary, filter all solutions.

Prepare reagents <u>F.3.2.3</u>, <u>F.3.2.4</u> and <u>F.3.2.5</u> with freshly boiled distilled water to ensure that the solutions are as free from dissolved oxygen as is practicable.

F.3.2.1 Hydrochloric acid, $\rho = 1,16$ g/ml, diluted 3 + 1.

Dilute 750 ml of hydrochloric acid (ρ = 1,16 g/ml) to 1 000 ml with water.

F.3.2.2 Iron(III) chloride, 100 g/l solution.

Dissolve 100 g of hydrated iron(lll) chloride in water containing 100 ml of hydrochloric acid ($\rho = 1,16$ g/ml) and dilute to 1 000 ml with water.

F.3.2.3 Potassium iodate standard solution, c(1/6 KlO3) = 0.05 mol/l. For use only with electrolytic tinplate, equally coated.

Dissolve 1,783 5 g of potassium iodate, previously dried to constant mass at 180 °C, and 19 g of potassium iodide in water containing 0,5 g of sodium hydroxide and dilute to 1 000 ml with water.

1 ml of this solution is equivalent to 0,002 967 g of tin.

F.3.2.4 Potassium iodate standard solution, c(1/6 KlO3) = 0.025 mol/l. For use only with electrolytic tinplate, differentially coated.

Dissolve 0,901 8 g of potassium iodate, previously dried to constant mass at $180\,^{\circ}$ C, and 1 g of potassium iodide in water containing 0,5 g of sodium hydroxide and dilute to 1 000 ml with water.

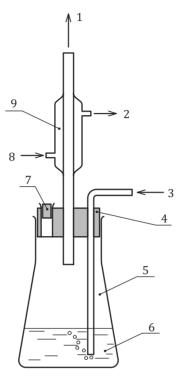
1 ml of this solution is equivalent to 0,001 484 g of tin.

- **F.3.2.5 Starch solution**, prepare a suspension of 1 g of soluble starch in 10 ml of water and add to 100 ml of boiling water. Boil for 2 min to 3 min and cool.
- **F.3.2.6 Diethyl ether**, $\rho = 0.72$ g/ml technical grade.
- **F.3.2.7 Platinum wire**, approximately 750 mm long and 0,6 mm in diameter, formed into a flat spiral of two turns and approximately 125 mm in diameter (see Figure F.4).
- **F.3.2.8** Aluminium metal foil, of 99,99 % (mass fraction) purity, tin-free, of 0,25 mm thickness.
- **F.3.2.9 Carbon dioxide**, oxygen-free.
- **F.3.2.10 Cellulose lacquer**, a suitable lacquer that dries in air.

F.3.2.11 Acetone, analytical reagent (AR) grade.

F.3.3 Apparatus

A suitable assembly for carrying out the reduction of tin consists of a 500 ml wide-neck conical flask marked at a volume of 200 ml. The flask is fitted with a rubber bung containing a bent gas inlet tube, a small Liebig-type condenser and a rubber-sealed tube for burette entry at the titration stage (see Figure F.3).



Key

- 1 gas outlet
- 2 water out
- 3 CO₂ entry
- 4 rubber bung
- 5 500 ml wide-neck flask

- 6 test solution
- 7 rubber seal and tube for burette entry
- 8 water in
- 9 small Liebig condenser

Figure F.3 — Apparatus for the reduction of tin

F.3.4 Procedure

F.3.4.1 Electrolytic tinplate — Equally coated

F.3.4.1.1 Tin coating mass equal to or greater than 2.5 g/m^2

Degrease with diethyl ether ($\underline{F.3.2.6}$) the test pieces (in the form of discs) taken from sheets as described in $\underline{14.1.1}$. Place the spiral of platinum wire ($\underline{F.3.2.7}$) centrally in a shallow dish (see Figure F.4). Place six of the discs in a circle on the platinum wire and carefully pour 150 ml of hydrochloric acid ($\underline{F.3.2.1}$) into the dish.

As soon as the tin coating is completely dissolved from both faces, leaving the steel surfaces exposed (see NOTE 1), transfer the acid quantitatively to a 1 000 ml one-mark volumetric flask. Wash twice with 25 ml of water, transferring the washings to the flask. Repeat the whole procedure with the remaining

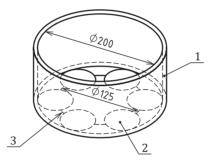
six discs, combining the acid and washings in the same volumetric flask, and finally dilute to the mark with water.

Transfer a 100 ml aliquot of the solution to the 500 ml wide-neck conical flask (F.3.3), add 75 ml of hydrochloric acid (F.3.2.1) and 10 ml of iron(lll) chloride solution (F.3.2.2) and dilute to the 200 ml mark with water. Add 2 g of aluminium metal foil (F.3.2.8). Insert the rubber bung fitted with a small Liebig condenser, a carbon dioxide entry tube and a rubber-sealed burette entry tube (see Figure F.3). Connect the apparatus to the appropriate supply points and pass carbon dioxide gas (F.3.2.9) through for 5 min to displace the air within the flask. Heat carefully until boiling starts, avoiding vigorous evolution of hydrogen. Continue boiling for 5 min to 10 min after dissolution of the aluminium metal. Cool quickly to less than 20 °C, maintaining an adequate supply of carbon dioxide.

Remove the burette entry tube seal, add 5 ml of starch solution ($\underline{F.3.2.5}$) and titrate with potassium iodate standard solution ($\underline{F.3.2.4}$) to a permanent blue colour.

NOTE 1 The time required for complete dissolution depends on the coating mass. It can vary from about 3 min for an 2,8/2,8 coating to about 10 min for an 11,2/11,2 coating.

NOTE 2 Care is necessary when adding the aluminium foil, to avoid a violent reaction; it is intended that the foil be cut into small sections beforehand.



Key

- 1 shallow dish
- 2 tinplate specimen
- 3 platinum wire

Figure F.4 — Arrangement of specimens for dissolution of tin

F.3.4.1.2 Tin coating mass less than 2.5 g/m^2

Degrease with the diethyl ether ($\underline{F.3.2.6}$) the test pieces (in the form of discs) taken from sheets as described in $\underline{14.1.1}$. Place the spiral of platinum wire ($\underline{F.3.2.7}$) centrally in a shallow dish (see Figure F.4). Place six of the discs in a circle on the platinum wire and carefully pour 150 ml of hydrochloric acid ($\underline{F.3.2.1}$) into the dish.

As soon as the tin coating is completely dissolved from both faces, leaving the steel surfaces exposed (see <u>F.3.4.1.1</u>, NOTE 1), transfer all the acid to a 1 000 ml one-mark volumetric flask. Wash twice with 25 ml of water, transferring the washings to the flask. Repeat this procedure with the remaining six discs, pouring the acid and washings into the same flask, and finally dilute to the mark with water.

Transfer 200 ml of the solution to the 500 ml wide-neck flask ($\underline{F.3.3}$), add 30 ml of hydrochloric acid ($\underline{F.3.2.1}$) and 10 ml of iron(lll) chloride solution ($\underline{F.3.2.2}$). Continue with the reduction and titration as described in $\underline{F.3.4.1.1}$, but using the potassium iodate standard solution ($\underline{F.3.2.4}$) as titrant.

F.3.4.2 Electrolytic tinplate - differentially coated

Degrease with diethyl ether (F.3.2.6) the test pieces (in the form of discs) from sheets as described in 14.1.1 and coat the faces carrying the heavier tin coating with a cellulose lacquer (F.3.2.10). Allow to

dry for 15 min, apply a second coat of lacquer and allow to dry for 1 h. Place the spiral of platinum wire (<u>F.3.2.7</u>) centrally in a shallow dish (see <u>Figure F.4</u>). Place six of the discs in a circle with the unlacquered faces in contact with the platinum wire. Carefully pour 150 ml of hydrochloric acid (<u>F.3.2.1</u>) into the dish.

As soon as the tin coating is completely dissolved from the unlacquered faces, leaving the steel surfaces exposed (see F.3.4.1.1, NOTE 1), transfer the acid quantitatively to a 1 000 ml one-mark volumetric flask. Wash twice with 25 ml of water, transferring the washings to the flask. Repeat the whole procedure with the remaining six discs, combining the acid and the washings in the same volumetric flask, and finally dilute to the mark with water. Dry the discs and keep them for determination of the coating on the lacquered faces.

Transfer a 100 ml aliquot of the solution to the 500 ml wide-neck conical flask ($\underline{F.3.3}$), add 75 ml of hydrochloric acid ($\underline{F.3.2.1}$) and 10 ml of iron(lll) chloride solution ($\underline{F.3.2.2}$) and dilute to the 200 ml mark with water. Continue with the reduction and titration as in $\underline{F.3.4.1.1}$, but using the potassium iodate standard solution ($\underline{F.3.2.4}$) as titrant.

Remove the lacquer from the test pieces used above by swabbing with cotton wool soaked in acetone (F.3.2.11). Place six of the discs with the unstripped surface uppermost in a circle on the platinum wire and continue as above.

F.3.5 Expression of results

Calculate the average coating mass, *m*, in grams per square metre, using Formula (F.2):

$$m = \frac{V \times c \times 5,935 \times 10^5}{A} \tag{F.2}$$

where

- *V* is the volume, in millimetres, of the potassium iodate solution;
- c is the concentration, in moles per litre, of the potassium iodate solution;
- *A* is the total test piece area, in square millimetres.

Annex G

(normative)

Rockwell HR15Tm values and their HR30Tm equivalents

Table G.1 — Rockwell HR15Tm values and their HR30Tm equivalents

HR15Tm value	Equivalent HR30Tm value	HR15Tm value	Equivalent HR30Tm value
93,0	82,0	83,0	62,5
92,5	81,5	82,5	61,5
92,0	80,5	82,0	60,5
91,5	79,0	81,5	59,5
91,0	78,0	81,0	58,5
90,5	77,5	80,5	57,0
90,0	76,0	80,0	56,0
89,5	75,5	79,5	55,0
89,0	74,5	79,0	54,0
88,5	74,0	78,5	53,0
88,0	73,0	78,0	51,5
87,5	72,0	77,5	51,0
87,0	71,0	77,0	49,5
86,5	70,0	76,5	49,0
86,0	69,0	76,0	47,5
85,5	68,0	75,5	47,0
85,0	67,0	75,0	45,5
84,5	66,0	74,5	44,5
84,0	65,0	74,0	43,5
83,5	63,5	73,5	42,5

Bibliography

- $[1] \hspace{0.5cm} \textbf{ISO 11951, } \textit{Cold-reduced tinmill products} \textit{Blackplate}$
- [2] ISO/TR 9769, Steel and iron Review of available methods of analysis

