INTERNATIONAL STANDARD 1111/II

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Cold reduced tinplate and cold reduced blackplate — Part II : Coil for subsequent cutting into sheet form

Fer blanc et fer noir laminés à froid —
Partie II : Bobines destinées au découpage ultérieur en feuilles

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the Member Bodies of the following countries:

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6 CONDITIONS OF MANUFACTURE AND UTILIZATION

6.1 The methods of manufacture of tinplate and blackplate are the province of the manufacturer.

6.2 The tin used for the coating shall have a purity of not less than 99,75 %.

6.3 For special applications, limits on the chemical composition of the steel can be agreed between the manufacturer and the purchaser provided that they are consistent with this International Standard (see clause 19).

6.4 The methods of using tinplate and blackplate are the province of the purchaser.

6.5 At the time they are made available by the manufacturer and under normal conditions of storage and transport, tinplate and blackplate are suitable for surface treatments such as lacquering and printing. Appropriate grades and tempers are also suitable for shaping operations such as stamping, drawing, folding and bending, and assembly work such as joint forming, soldering (tinplate only) and welding.

The purchaser's order requirements shall be consistent with the end use of the product.

NOTES

1 When ordering tinplate and blackplate it is recommended that the purpose for which the tinplate or blackplate is intended should be stated.

2 It is recommended that the purchaser should be informed of any alterations in methods of manufacture which will significantly affect the properties of the purchased product. Similarly, the purchaser should inform the manufacturer of modifications in his fabrication methods which will significantly affect the way in which the purchased product is used.

7 CONDITIONS GOVERNING THE SUPPLY AND USE OF COILS

7.1 Tinplate and blackplate supplied in coil form represent the normal production of processing lines for these products. Although subjected to normal inspection, the production of coils does not afford the opportunity for removal of all material not conforming to dimensions, or of all defects associated with the steel base, or of all surface defects.

A coil of tinplate or blackplate, as shipped, may therefore contain unusable portions which the manufacturer has been unable to cut out. These arise from imperfections or blemishes, the type and occurrence of which varies considerably from coil to coil or lot to lot. Some of these conditions are not detectable until the coil is sheared.

7.2 A coil of tinplate or blackplate as shipped may contain a number of joints (see clause 10).

7.3 It is the responsibility of the manufacturer to line inspect the material during manufacture and to ensure that the material conforms to the requirements of clause 8.

7.4 It is the responsibility of the purchaser to have adequate handling, roller levelling and shearing equipment and to take reasonable care during these operations.

It is also the responsibility of the purchaser to inspect the material (see clause 8).

8 MATERIAL GRADING

8.1 The material termed "standard coil grade" tinplate is defined as being from the normal production of tinplating lines employing the usual methods of inspection.

Responsibility for inspecting on receipt and for classifying and/or assorting during and after cutting the coil to sheet form, rests with the purchaser or his agent.

NOTE – Any portion of the consignment found to be damaged on receipt at the purchaser's works shall be the subject of immediate notification of exception to the delivering carrier and separate negotiation between the manufacturer, the purchaser, and the carrier as appropriate.

8.2 Each coil in a consignment is deemed to contain a major proportion of "standard coil grade" tinplate and a small proportion of tinplate not of "standard coil grade". The purchaser shall be prepared to justify his opinion regarding tinplate he judges not to be of "standard coil grade".

8.3 It is from the "standard coil grade" tinplate in the consignment that the test samples indicated in 14.2 are taken.

8.4 Blackplate shall be inspected by the manufacturer but material having some imperfections may be included. Blackplate is liable to rust but, at the time it is made available by the manufacturer, shall be suitable for normal lacquering and printing.

8.5 Since it is difficult objectively to define all types of defects and their magnitude in tinplate and blackplate in coil form, standards of acceptance relating to shape (see 9.6), surface defects, and holes shall be the subject of an agreement between the manufacturer and the purchaser.

8.6 The area of all sheets outside the thickness tolerances (see 9.3.4.1) shall be the subject of an agreement between the manufacturer and the purchaser.

8.7 If, when processing a coil, the purchaser encounters recurring defects which, in his opinion, seem excessive, he shall, if practicable, stop processing the coil and advise the manufacturer. The manufacturer then has the option of requesting test procedure in accordance with this International Standard.

9 COIL GEOMETRY

9.1 Length of coil

9.1.1 In cases where it is required to verify the length of material supplied in a coil, this shall be done by multiplying the average sheared length by the number of sheets obtained and adding the cumulative length of any other portions of the coil as received. The average sheared length shall be determined by measuring at least ten sheets, taken at random, to an accuracy of 0,2 mm. The measurement of length by other means, provided that they are acceptable to both manufacturer and the purchaser, are not precluded.

9.1.2 The manufacturer should aim to despatch the indicated length. The actual length of a single coil shall not vary from the indicated length by more than ± 3,5 %.

9.2 Width

9.2.1 The measurements shall be made on the sample selected in accordance with clause 14 with the sheets laid on a flat surface. The measurement of width to the nearest 0,5 mm (0.02 in) shall be made across the centre of the sheet, at right angles to the rolling direction.

9.2.2 The width of each sheet in the sample measured in accordance with 9.2.1 shall be not less than the ordered dimension. Normally no sheet will exceed the ordered width dimension by more than 3 mm.

TABLE 1 — **Nominal inch thicknesses**

0.0060
0.0066
0.0071
0 0077
0.0082
0.0088
0.0093
0 0099
0.0104
0.0110
0 0113
0.0118
0 0123
0 0130
0 0141
0 0149
0.0171
0.0193

9.3 Thickness

9.3.1 *Metric thicknesses*

Tinplate and blackplate ordered in metric thicknesses are normally available in any nominal thickness which is a multiple of 0,01 mm from 0,15 mm up to and including 0,49 mm.

9.3.2 *Inch thicknesses*

Tinplate and blackplate ordered in inch thicknesses are normally available in the nominal inch thicknesses listed in table 1.

NOTES

1 Other thicknesses in inch dimensions may be supplied by agreement between the manufacturer and the purchaser.

2 Annex C gives the relationship between designations in inches and designations in traditional nomenclature.

3 The inch thicknesses 0.017 1 and 0.019 3 are described as tinned steel sheets in the continent of North America.

9.3.3 The manufacturer shall aim to supply the thickness ordered.

9.3.4 *Thickness tolerances*

Thickness determinations shall be made on the sample sheets selected in accordance with clause 14.

9.3.4.1 When shearing a coil, any sheet deviating by more than ± X % of the nominal thickness shall be eliminated. The value of X shall be 10 or 8,5 by agreement between the manufacturer and the purchaser. However, in special circumstances other tolerances may be agreed.

9.3.4.2 The thickness of each sheet selected in accordance with clause 14 shall be measured as described in 9.3.5. The average thickness of a consignment shall be represented by the arithmetic mean of all the specimen sheets tested.

9.3.4.3 The value of the arithmetic mean shall not deviate from the nominal thickness :

by more than ± 2,5 % for a consignment comprising more than 15 000 m, i.e. twenty units (see clause 14);

by more than ± 4 % for a consignment comprising 15 000 m, i.e. twenty units, or less.

9.3.4.4 TOLERANCES ON INDIVIDUAL SHEETS

No sheet among those selected in accordance with clause 14 and measured as described in 9.3.5 shall deviate from the nominal thickness by more than the tolerance agreed between the manufacturer and the purchaser.

9.3.4.5 TOLERANCES ACROSS STRIP WIDTH

The thickness of either of the two individual specimens determined in accordance with 9.3.6 shall not deviate from the actual average thickness of the whole sheet determined in accordance with 9.3.5 by more than ± 4 %.

9.3.5 Except when determining the variation across the strip width (see 9.3.6), thickness shall be determined by weighing a whole sheet, measuring the area and applying one of the formulae given in 9.3.7. The mass of the sheet shall be determined to a precision of 2 g (0.005 lb) and the thickness shall be stated to the nearest 0,001 mm (0.000 01 in). The dimensions of the sheet shall be measured to a precision of 0,5 mm (0.02 in).

9.3.6 For determining the variation of thickness across the strip width, the thickness of each of the specimens Y (see clause 14 and figure 2) shall be determined by weighing the specimen, measuring the area and applying one of the formulae given in 9.3.7.

The mass of the specimens shall be determined to a precision of 0,01 g or better and the thickness shall be stated to the nearest 0,001 mm (0.000 01 in). The dimensions of the specimens shall be measured to a precision of 0,1 mm (0.005 in).

9.3.7 Thickness shall be calculated by applying one of the following formulae :

$$\text{thickness (mm)} = \frac{\text{mass (g)}}{\text{actual area (cm}^2\text{)} \times 0{,}785}$$

$$\text{thickness (in)} = \frac{\text{mass (lb)}}{\text{actual area (in}^2\text{)} \times 0{,}283\,3}$$

9.4 Camber

9.4.1 Camber is the deviation of an edge from a straight line forming a chord to it.

Camber is expressed as

$$\frac{\text{deviation}}{\text{length of chord}} \times 100\ \%$$

9.4.2 The camber, measured over a distance of 6 m (20 ft), shall not exceed 0,1 %.

9.5 Lateral weave (short pitch camber)

9.5.1 Lateral weave is the deviation of a mill-trimmed edge from a straight line lying in the same plane and forming a chord to it over a relatively short distance.

9.5.2 The lateral weave measured over a distance of 1 m (40 in) shall not exceed 1,5 mm (1/16 in) when measured prior to shearing. If the strip is required for scroll shearing, the acceptable lateral weave shall be the subject of an agreement between the manufacturer and the purchaser.

9.6 Other geometrical features

9.6.1 Other geometrical features may be present in material supplied in coil, such as :

Edge wave – an intermittent vertical displacement occurring at the strip edge when the strip is laid on a flat surface.

Centre buckle (full centre) – an intermittent vertical displacement or wave in a strip occurring other than at the edges.

Longitudinal bow (line bow) – residual curvature remaining along the direction of rolling after the strip has been uncoiled.

Transverse bow (cross bow) – a mode of curvature of the strip such that the distance between its edges is less than the strip width.

9.6.2 It is not possible at present to define methods of measuring, or to specify limits for these geometrical features, certain of which are subjective to the equipment employed by the purchaser.

The manufacturer shall endeavour to keep the occurrence and magnitude of edge wave, centre buckle and transverse bow to a minimum. He shall also endeavour to minimize variation of the longitudinal bow.

10 JOINTS

The manufacturer shall ensure continuity of the coil within the limit of the specified length, if necessary by means of electric welded joints made after cold reduction.

10.1 Number of joints

The number of joints in a coil shall not exceed four in a length of 6 000 m or *pro rata*.

10.2 The location of each joint in a coil shall be indicated clearly by the insertion of a piece of non-rigid material; however, other methods may be used by agreement between the manufacturer and the purchaser.

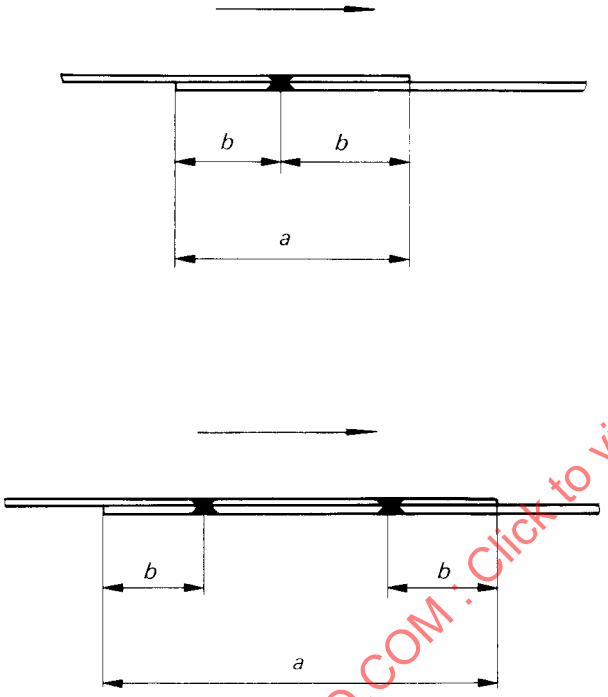
10.3 Dimensions of joints

10.3.1 *Thickness*

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

10.3.2 *Overlap*

In any lap joint, the total length of overlap shall not exceed 200 mm (8 in). The free overlap shall not exceed 25 mm (1 in) (see figure 1).



a = total length of overlap

b = free overlap

FIGURE 1 – **Alternative joints**

11 TIN COATING MASSES

11.1 Tin coating masses shall be expressed in grams per square metre.

The minimum average coating mass values as shown in tables 2 and 3 represent the minimum permissible values for the arithmetic mean of a sample selected in accordance with clause 15.

11.2 Electrolytic tinplate – equally coated

11.2.1 Four coating masses are specified as shown in table 2.

TABLE 2 – **Coating masses for electrolytic tinplate – equally coated**

Code (see note)	**Nominal coating mass** (see note)	**Minimum average coating mass** (see 11.1 and 11.2.3)
	g/m²	g/m²
E2,8/2,8	5,6 (2,8/2,8)	4,9
E5,6/5,6	11,2 (5,6/5,6)	10,5
E8,4/8,4	16,8 (8,4/8,4)	15,7
E11,2/11,2	22,4 (11,2/11,2)	20,2

NOTE – The code figures are derived from the nominal coating mass on each surface of the tinplate. The nominal coating mass values refer to the total mass of coating on both surfaces; thus the nominal coating mass on each surface is half the stated value; for example E2,8/2,8 has 2,8 g/m² on each surface and a total of 5,6 g/m² on both surfaces.

11.2.2 Other coating masses may be supplied by agreement between the manufacturer and the purchaser.

11.2.3 The average value of the coating masses of the sample selected to represent a consignment in accordance with clause 14 and tested in accordance with clause 15 shall not be lower than the appropriate minimum average coating mass specified in table 2.

NOTE – Isolated specimens have no representative value in relation to the consignment under consideration; therefore, on the individual specimens of the sample, the check may show tin coatings as for example, 80 % of the nominal coating mass.

11.3 Electrolytic tinplate – differentially coated

11.3.1 Four coating masses are specified as shown in table 3.

TABLE 3 – **Coating masses for electrolytic tinplate – differentially coated**

Code (see note)	**Nominal coating mass** (see note)	**Minimum average coating mass** (see 11.1 and 11.3.3)
	g/m²	g/m²
D8,4/2,8	8,4/2,8	7,85/2,25
D11,2/2,8	11,2/2,8	10,1/2,25
D11,2/5,6	11,2/5,6	10,1/4,75
D15,1/5,6	15,1/5,6	13,4/4,75

NOTE – The code figures are derived from the nominal coating mass on each surface of the tinplate. The nominal coating mass values refer to the nominal mass of coating on each surface; for example D8,4/2,8 has 8,4 g/m² on one surface and 2,8 g/m² on the other.

11.3.2 Other coating masses may be supplied by agreement between the manufacturer and the purchaser.

11.3.3 The average value of the coating masses of the sample selected to represent a consignment in accordance with clause 14 and tested in accordance with clause 15 shall not be lower than the appropriate minimum average coating mass specified in table 3.

NOTE – On the individual specimens of the sample the check may show tin coating masses as low as, for example, 80 % of the nominal coating mass, but it is emphasized that isolated specimens have no representative value in relation to the consignment under consideration.

11.4 Marking of differentially coated tinplate

In order to distinguish material having differential coatings, the strip should be marked on one face, which, by arrangement, may be either the light or heavy coated face. In all cases, both the face to be marked and the outer face of the coil should be clearly designated on the contract by the purchaser. Generally the marking system should be in the form of dull straight parallel lines about 1 mm (0.04 in) wide, in which case the code indicated in annex D should be used. Other marking methods may be employed by arrangement between the manufacturer and the purchaser.

12 TEMPER CLASSIFICATIONS

12.1 The term "temper", when applied to tinplate and blackplate, summarizes a combination of inter-related mechanical properties, and no single mechanical test can measure all the various factors which contribute to the fabrication characteristics of the material. However, the Rockwell 30 T hardness test (HR 30 T) is the best single test available and serves as a guide to the properties of the material. This test forms the basis for a system of temper classification as shown in tables 4 and 5, which give the hardness values at which the manufacturer should aim.

12.2 The Rockwell hardness numbers shown in tables 4 and 5 are for tests performed with a diamond anvil on tinplate after de-tinning. For a given temper classification, the corresponding HR 30 T values for blackplate may be as much as four units lower, depending on the age of the plate and the conditions of storage.

12.3 The hardness of tinplate and blackplate shall be determined on samples selected in accordance with clause 14 and tested in accordance with clause 17. When evaluating the hardness of tinplate and blackplate, average values and not individual values shall be considered.

12.4 The Rockwell hardness test is sensitive to the "anvil" effect and hence is affected by the thickness of the test specimen. The values in tables 4 and 5 are typical for nominal thicknesses in the range of 0,25 to 0,31 mm (0.009 9 to 0.011 8 in). Material of the same metallurgical quality in 0,23 mm (0.008 8 in) thickness, for instance, would be one HR 30 T unit higher and material of 0,43 mm (0.017 1 in) thickness, one unit lower. Thinner plate, for example 0,17 mm (0.006 6 in), may be two units higher.

12.5 The purchaser should specify the temper required by reference to the appropriate temper classification as set out in table 4 or 5.

TABLE 4 – **Rockwell HR 30 T hardness values normally associated with the temper classifications of batch annealed tinplate**

Temper classification	Rockwell HR 30 T hardness aim	
	mean	maximum deviation of sample average
T 50	52 max.	
T 52	52	± 4
T 57	57	+ 4 − 3
T 61	61	± 4
T 65	65	+ 3 4
T 70	70	+ 3 4

TABLE 5 – **Rockwell HR 30 T hardness values normally associated with the temper classifications of continuously annealed tinplate**

Temper classification	Rockwell HR 30 T hardness aim	
	mean	maximum deviation of sample average
CA 61	61	± 4
CA 65	65	+ 5 4
CA 70	70	+ 3 4

12.6 Other tempers of batch annealed or continuously annealed tinplate may be supplied by agreement between the manufacturer and the purchaser.

12.7 The mechanical properties of continuously annealed tinplate and blackplate and batch annealed material of the same HR 30 T hardness number are not identical.

13 SAMPLING AND TESTING

13.1 Sampling for quality testing requires cutting the coil and in practice this is normally carried out by the purchaser during his usual shearing operation.

13.2 If the purchaser wishes to make tests to ascertain compliance with the requirements of this International Standard, he should permit the manufacturer to be present during sampling and testing, and be able to justify the identity of the material to be tested and its correspondence with the coils from which the samples have been taken.

13.3 To ascertain compliance with the quality as laid down in 9.3 and clauses 11 and 12, samples should be taken only from the "standard coil grade" tinplate (see clause 8).

13.4 Sampling of blackplate shall, with the exception of hardness testing, be the subject of an agreement between the manufacturer and the purchaser (see 14.4.1 b).

14 SELECTION OF SAMPLES

14.1 If tests are made to ascertain compliance with the requirements of this International Standard, the following procedure should be adopted.

14.2 Initial sampling procedure

For the purpose of sampling tinplate, each consignment of coils shall be considered as one lot. After cutting into rectangular or scrolled sheets, the sheets deemed not to be of "standard coil grade" tinplate shall be excluded. The remaining "standard coil grade" tinplate shall be sampled using a unit[1] of strip 750 m (2 461 ft) in length as the basis, in accordance with 14.3 and 14.4, for the verification of properties and dimensions respectively.

Initial sampling procedures for blackplate shall be the subject of an agreement between manufacturer and purchaser.

14.3 Number of units

For consignments comprising up to and including twenty units, four units shall be selected at random.

For consignments of more than twenty units, units shall be selected at the rate of four units for each twenty units or part thereof.

14.4 Number of sample sheets

14.4.1 *For verification of properties*

a) Electrolytic tinplate

From each of the units selected in accordance with 14.3, one sheet shall be taken at random for checking the tin coating and hardness.

b) Blackplate

From each of the units selected in accordance with 14.3, one sheet shall be taken at random for checking the hardness.

14.4.2 *For verification of dimensions*

From each of the units selected in accordance with 14.3, five sheets shall be taken at random for checking the thickness and width.

14.5 Location of test specimens

The test specimens shall be taken from the positions indicated in figure 2.

15 DETERMINATION OF TIN COATING MASS

15.1 Specimens

For tin coating mass determination, from each sheet selected in accordance with clause 14, three specimens, each of an area not less than 25 cm^2 (4 in^2) and preferably in the form of disks, shall be accurately punched, in the positions indicated X in figure 2. The edge specimens shall clear the edges of the sheet by a minimum of 25 mm (1 in).

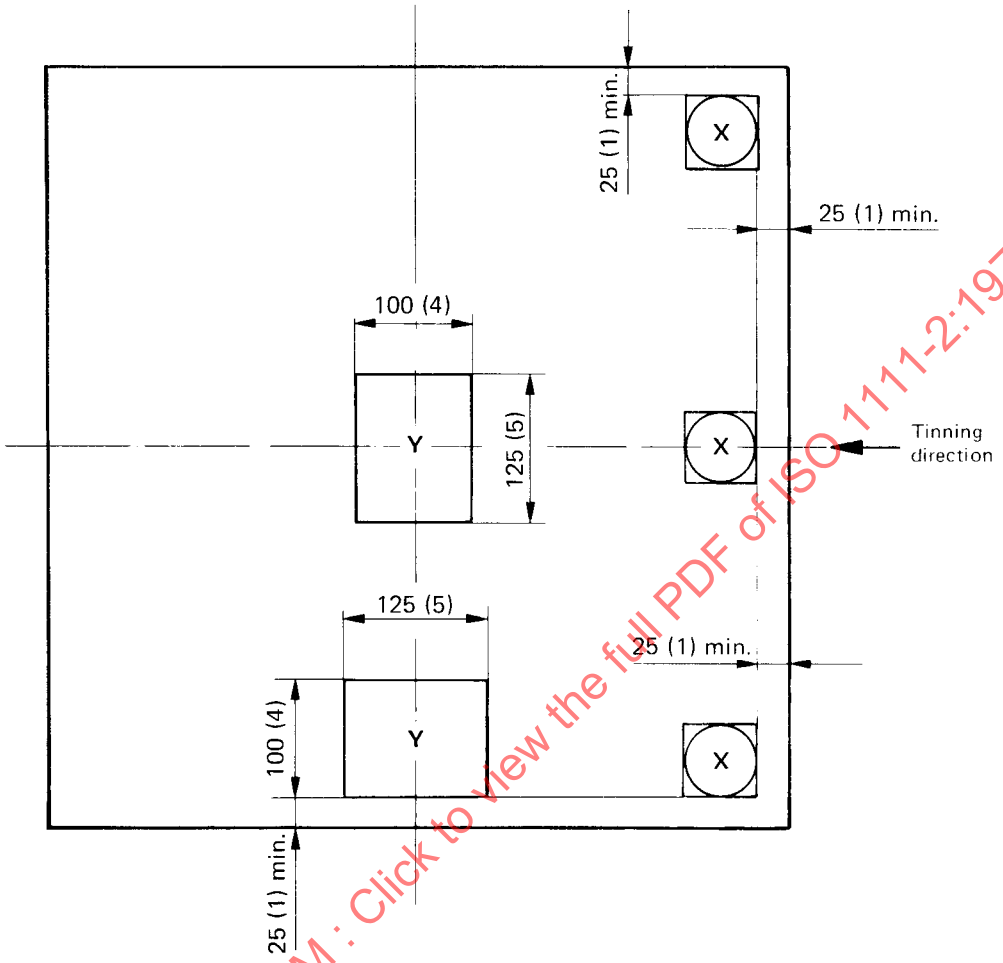
15.2 Determining tin coating mass

The tin coating mass may be determined by any of the recognized and accepted analytical methods.

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

1) This unit of strip approximates to one bulk package of tinplate in sheet form.

Dimensions in millimetres
(Inch values in parentheses)



X — specimens for tin coating mass tests

Y — specimens for hardness tests and determination of local thickness within a sheet.

FIGURE 2 — **Position of test specimens**

16 DETERMINATION OF HARDNESS

16.1 Specimens

For hardness determination, from each sheet selected in accordance with clause 14, two rectangular specimens 100 mm × 125 mm (4 in × 5 in) shall be cut from the positions indicated Y in figure 2.

16.2 Hardness test

Three measurements shall be made on each of the specimens selected in accordance with clause 14.

The HR 30 T test shall be carried out by the method described in ISO/R 1024 under the conditions described in 4.4 a) of that document and using a diamond anvil. The test shall be made on the test pieces after chemically or electrochemically de-tinning. The average value shall be the arithmetic mean of all the values so obtained.

16.3 On light gauge material (for example on 0,22 mm (0.008 8 in) and thinner) the hardness test may be made using the HR 15 T test, in which case the values obtained shall be converted using the table given in annex B.

17 RETESTS

17.1 Tin coating mass

If any determination fails to meet the specified requirements, two further samples shall be taken from other units to be selected as specified in clause 14 and specimens taken as described in clause 16.

If both retests are satisfactory, the consignment shall be deemed to meet the requirements of this International Standard, but if either of the additional tests fails, the consignment shall be deemed not to meet the requirements of this International Standard.

The retest determination shall be made using the iodine titration reference method specified in annex A.

17.2 Hardness tests

In the event of any hardness test value, determined by the procedure in clause 16, failing to meet the appropriate values specified in table 4 or 5, a retest shall be made on two further samples selected from other units according to the procedure outlined in clause 14. If both retests are satisfactory, the consignment shall be deemed to meet the requirements of this International Standard, but if either of the additional tests fails, the consignment shall be deemed not to meet the requirements of this International Standard.

17.3 Dimensional tests

If the result of any dimensional check is unsatisfactory, a further check shall be made on two further samples selected from other units. If both retests are satisfactory, the consignment shall be deemed to meet the requirements of this International Standard, but if either of the additional tests fails, the consignment shall be deemed not to meet the requirements of this International Standard.

18 IDENTIFICATION AND PACKAGING

18.1 Packaging

This is a matter for negotiation between the manufacturer and the purchaser, but cold reduced tinplate and blackplate in coil form is normally packed on a platform forming a package weighing between 4 000 and 12 000 kg (see ISO 3798).

18.2 Method of shipping

The preferred method of shipping, for example core vertical or horizontal, shall be stated.

19 SPECIAL ARRANGEMENTS

19.1 Special arrangements between the manufacturer and purchaser may contain additional provisions provided that they are not inconsistent with this International Standard.

19.2 Examples of such arrangements are :

19.2.1 Heavy oil coatings : it should be noted that such coatings may affect lacquerability.

19.2.2 Chemical composition of the steel : limitation of certain elements which may affect performance.

19.2.3 Utilization of cupping tests.

19.2.4 Marking of differentially coated tinplate, for example broken lines, diamond patterns, etc.

19.2.5 Tinplate surface chemical treatment, if other than cathodic sodium dichromate is required.

ANNEX A

VOLUMETRIC METHOD FOR DETERMINING TIN COATING MASS (IODINE METHOD)

A.1 PRINCIPLE

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

The effective range of the method is from 2,5 up to 50 g/m² and the reproducibility is ± 0,1 g/m².

A.2 REAGENTS

All reagents shall be of the highest purity obtainable, and distilled water shall be used throughout. Solutions shall be freshly prepared and where necessary filtered.

Reagents A.2.3, A.2.4 and A.2.5 shall be prepared with freshly boiled distilled water, to ensure that the solutions are as free from dissolved oxygen as precticable.

A.2.1 Hydrochloric acid, 75 % (*V*/*V*).

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

A.2.2 Iron(III) chloride solution, 100 g/l.

Dissolve 100 g of hydrated iron(III) chloride in water containing 100 ml of hydrochloric acid (ρ 1,16 g/ml) and dilute to 1 000 ml with water.

A.2.3 Potassium iodate, 0,05 N standard volumetric solution (solution 1).

For use 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 iodate in water containing 0,5 g of sodium hydroxide, and dilute to 1 000 ml with water.

A.2.4 Potassium iodate, 0,025 N standard volumetric solution (solution 2).

For use with electrolytic tinplate, differentially coated.

Dissolve 0,891 8 g of potassium iodate (previously dried to constant mass at 180 °C) and 10 g of potassium iodide in water containing 0,5 g of sodium hydroxide, and dilute to 1 000 ml with water.

A.2.5 Starch solution.

Make a suspension of 1 g of soluble starch in 10 ml of water and add to 100 ml of boiling water. Boil for 2 or 3 min and cool.

A.2.6 Ethyl ether, technical grade (ρ 0,72 g/ml).

A.2.7 Platinum wire.

A length of approximately 750 mm of 0,6 mm diameter platinum wire shall be formed into a flat spiral of two turns and approximately 125 mm diameter (see figure 4).

A.2.8 Aluminium metal, 99,99 % purity (tin free), as foil, 0,25 mm thickness.

A.2.9 Carbon dioxide (oxygen free).

A.2.10 Lacquer. A suitable air-drying cellulose lacquer.

A.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 3).

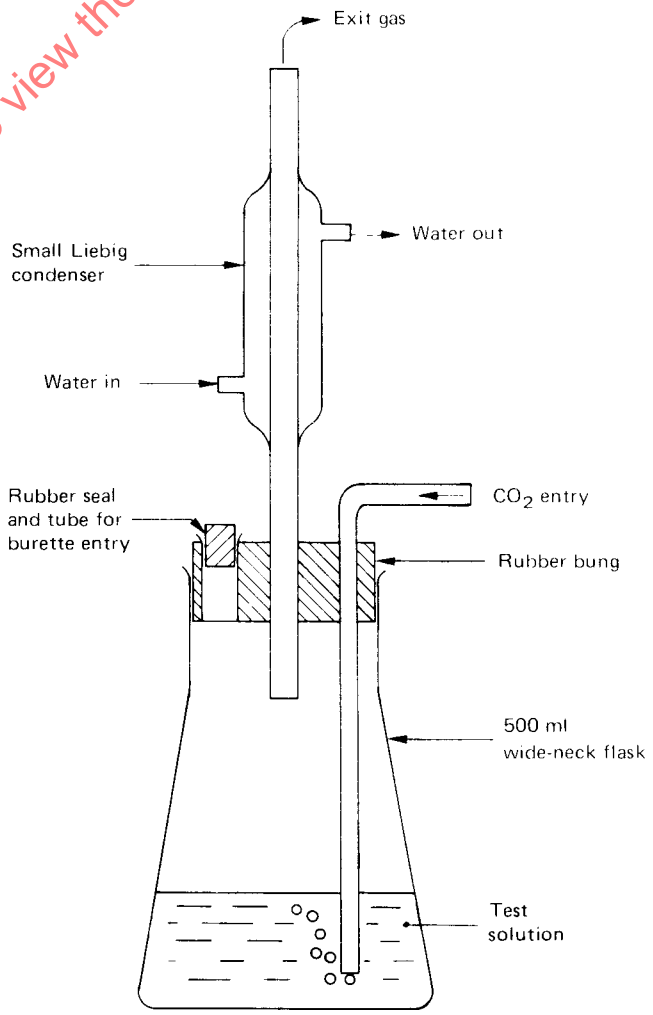


FIGURE 3 – **Assembly for the reduction of tin**

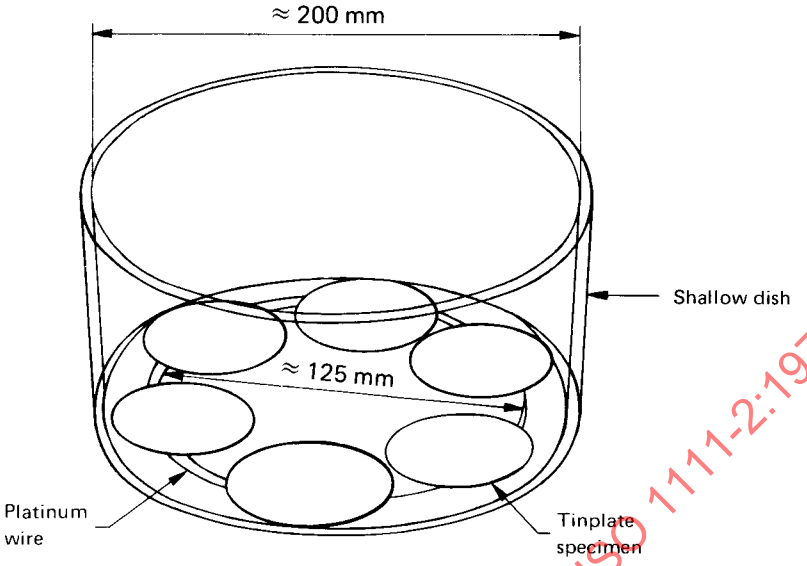


FIGURE 4 – **Arrangement of specimens for dissolution of tin**

A.4 PROCEDURE

A.4.1 Electrolytic tinplate – equally coated

Degrease with ether (A.2.6) the specimens from four sheets taken in accordance with 15.1. Place the spiral of platinum wire (A.2.7) centrally in a shallow dish (see figure 4). Place six of the disks in a circle on the platinum wire and carefully pour 150 ml of hydrochloric acid (A.2.1) into the dish. As soon as the coating is completely dissolved from both faces, leaving the steel surfaces exposed (see note below), transfer the acid quantitatively to a 1 000 ml volumetric flask. Wash twice with 25 ml of water, transferring the washings to the flask. Repeat the whole procedure with the remaining six disks, combining the acid and washings in the same volumetric flask, finally diluting to the mark with water.

Transfer a 100 ml aliquot portion of the solution to the 500 ml wide-neck conical flask, add 75 ml of hydrochloric acid (ρ 1,16 g/ml) and 10 ml of iron(III) chloride solution (A.2.2) and dilute to the 200 ml mark with water. Add 2 g of aluminium metal foil (A.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 3). Connect the apparatus to the appropriate supply points, and pass carbon dioxide gas for 5 min to displace the air within the flask. Heat carefully to boiling, avoiding vigorous evolution of hydrogen. Continue boiling for 5 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 (A.2.5) and titrate with standard volumetric potassium iodate solution 1 (A.2.3) to a permanent blue colour.

NOTE – The time required for complete dissolution depends on the coating mass. It may vary from about 3 min for an E2,8/2,8 coating to about 10 min for an E11,2/11,2 coating.

A.4.2 Electrolytic tinplate – differentially coated

Degrease with ether (A.2.6) the specimens from four sheets taken in accordance with 15.1 and coat the faces carrying the heavier tin coating with a suitable cellulose lacquer. Allow to dry for 15 min, apply a seoond coat of lacquer and allow to dry for 1 h. Place the spiral of platinum wire (A.2.7) centrally in a shallow dish (see figure 4). Place six of the disks in a circle with the unlacquered faces in contact with the platinum wire. Carefully pour 150 ml of hydrochloric acid (A.2.1) into the dish. As soon as the coating is completely dissolved from the unlacquered faces, leaving the steel surfaces exposed (see note to A.4.1), transfer the acid quantitatively to a 1 000 ml volumetric flask, Wash twice with 25 ml of water, transferring the washings to the flask. Repeat this whole procedure with the remaining six disks, combining the acid and washings in the same volumetric flask, finally diluting to the mark with water. Dry the disks and keep them for determination of the coating on the lacquered faces.

Transfer a 100 ml aliquot portion of the solution to the 500 ml wide-neck conical flask, add 75 ml of hydrochloric acid (ρ 1,16 g/ml) and 10 ml of iron(III) chloride solution (A.2.2) and dilute to the 200 ml mark with water. Continue the reduction and titration as in A.4.1 but using the standard volumetric potassium iodate solution 2 (A.2.4) as titrant.

Remove the lacquer from the specimens used above by swabbing with cotton wool soaked in acetone. Place six of the disks with the unstripped surface uppermost in a circle on the platinum wire and continue as above.

A.5 EXPRESSION OF RESULTS

The average tin coating mass value, in grams per square metre, as expressed in tables 2 and 3, is given by the formula

$$\frac{V \times T \times 5{,}935 \times 10^6}{A}$$

where

V is the volume of potassium iodate solution, in millilitres;

T is the normality of the potassium iodate solution;

A is the total specimen area, in square millimetres (see 15.1).