
International Standard



1111/2

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**Single cold-reduced tinplate and single cold-reduced
blackplate —
Part 2: Electrolytic tinplate coil and blackplate coil for
subsequent cutting into sheet form**

Fer-blanc et fer noir laminés à froid par simple réduction — Partie 2: Bobines de fer-blanc électrolytique et de fer noir, destinées au découpage ultérieur en feuilles

Second edition — 1983-12-15

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

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.

International Standard ISO 1111/2 was developed by Technical Committee ISO/TC 17, *Steel*, and was circulated to the member bodies in October 1982.

It has been approved by the member bodies of the following countries:

Australia	Hungary	Norway
Austria	India	Poland
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No member body expressed disapproval of the document.

This second edition cancels and replaces the first edition (i.e. ISO 1111/2-1976).

Annex A

Recommended marking system to indicate coating mass combination for differentially coated tinplate

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

The spacings shown in table 6 should be used.

Table 6 – Spacings used

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

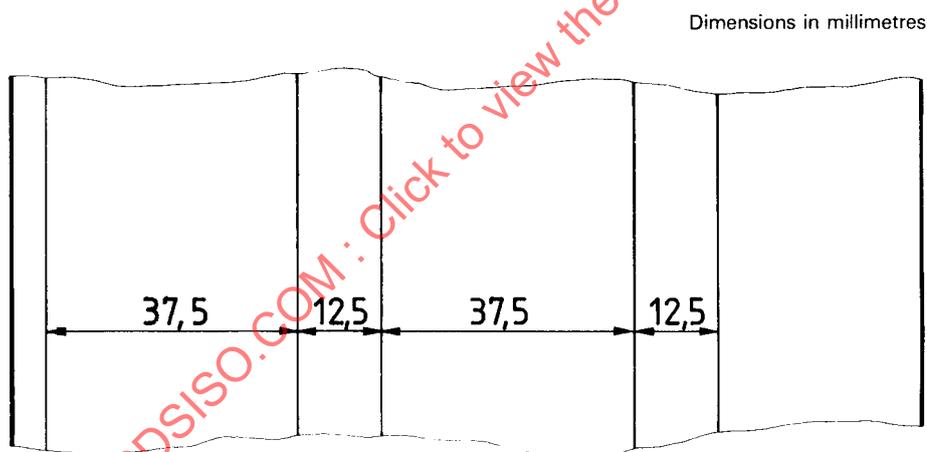


Figure 4 – Marking system for electrolytic tinplate differentially coated (Example of marking for D11,2/5,6)

NOTE – Differentially coated tinplate with nominal coatings not contained in table 2 should be marked with parallel lines employing 75 mm spacings.

Annex B

Rockwell HR 30T and HR 15T test

The indentation hardness shall be measured on a Rockwell superficial hardness testing machine employing 30T or 15T scales (see ISO/R 1024), as appropriate. The machine shall be provided with an anvil having a diamond centre spot and in the case of tinplate tests shall always be carried out on de-tinned specimens. Avoid testing near the edges of the specimen because of a possible cantilever effect.

To carry out the test, place the specimen on the anvil and bring it into contact with the ball indenter by turning the hand wheel

until the indicator on the dial shows that the minor load is applied. Then turn the adjustable rim of the dial until the pointer reads zero and apply the major load by operating the handle. The rate of loading is controlled by a dash-pot incorporated in the machine. As soon as the loading is complete, remove the major load by pulling the handle forward and read the Rockwell hardness number directly on the appropriate scale.

If on relatively thin material the HR 15T scale is used (see 9.4), convert the values to HR 30T values using table 7.

Table 7 — Hardness conversion values

HR 15T value	Equivalent HR 30T value
90,0	76,0
89,5	75,5
89,0	74,5
88,5	74,0
88,0	73,0
87,5	72,0
87,0	71,0
86,5	70,0
86,0	69,0
85,5	68,0
85,0	67,0
84,5	66,0
84,0	65,0
83,5	63,5
83,0	62,5
82,5	61,5
82,0	60,5
81,5	59,5
81,0	58,5
80,5	57,0
80,0	56,0
79,5	55,0
79,0	54,0
78,5	53,0
78,0	51,5
77,5	51,0
77,0	49,5
76,5	49,0
76,0	47,5
75,5	47,0
75,0	45,5
74,5	45,0
74,0	43,5
73,5	43,0
73,0	41,5

Annex C

Volumetric method for determining tin coating mass (iodometric method)

C.1 Principle

Dissolution in hydrochloric acid of the tin coating; reduction of the tin in an aliquot portion to the bivalent state with metallic aluminium. Determination of the tin in the reduced state by titration with standard potassium iodate solution.

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

C.2 Reagents

All reagents shall be of recognized analytical grade or higher purity and distilled water shall be used throughout. Solutions shall be freshly prepared and, where necessary, filtered.

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

C.2.1 Hydrochloric acid, 75 % (V/V) solution.

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

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

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.

C.2.3 Potassium iodate, 0,05 mol/l standard 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 iodide in water containing 0,5 g sodium hydroxide and dilute to 1 000 ml with water.

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

C.2.4 Potassium iodate, 0,025 mol/l standard 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.

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

C.2.5 Potassium iodate, 0,10 mol/l standard solution (Solution 3).

For use with hot-dipped tinplate

Dissolve 3,567 0 g of potassium iodate (previously dried to constant mass at 180 °C) and 37,5 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 standard solution is equivalent to 0,005 935 g of Sn.

C.2.6 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.

C.2.7 Ethyl ether, technical grade (ρ 0,72 g/ml).

C.2.8 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 6).

C.2.9 Aluminium metal, 99,99 % purity (tin-free), as foil, 0,25 mm thickness.

C.2.10 Carbon dioxide (oxygen-free).

C.2.11 Air-drying cellulose lacquer.

C.3 Apparatus

A suitable assembly for carrying out the reduction of tin consists of a 50 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 5).

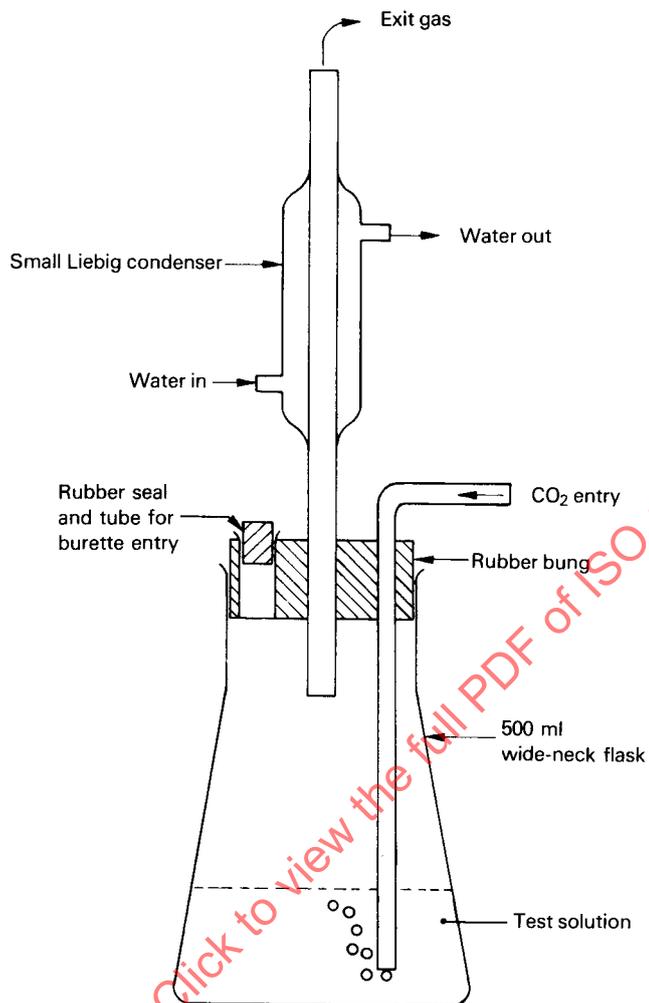


Figure 5 – Assembly for the reduction of tin

Dimensions in millimetres

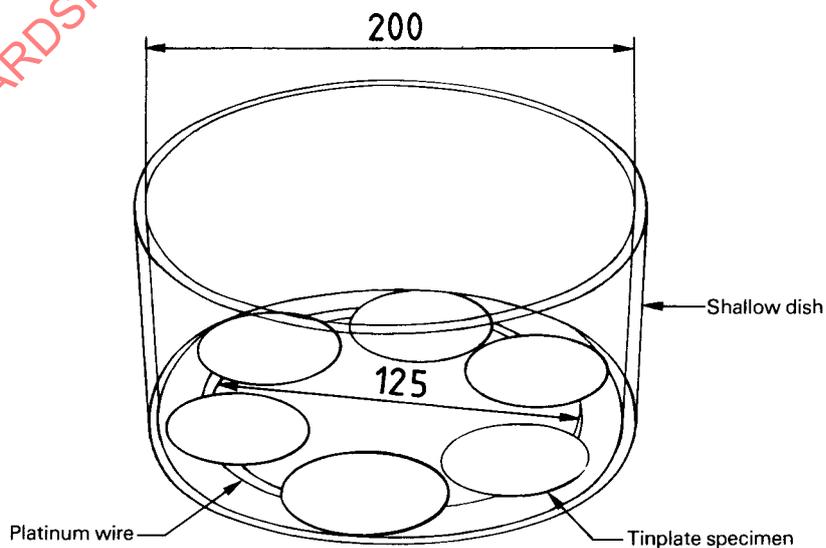


Figure 6 – Arrangement of specimens for dissolution of tin

C.4 Procedure

C.4.1 Electrolytic tinfoil, equally coated

Degrease with the ether (C.2.7) the specimens from sheets taken in accordance with 8.1. Place the spiral of platinum wire (C.2.8) centrally in a shallow dish (see figure 6). Place six of the discs in a circle on the platinum wire and carefully pour 150 ml of the hydrochloric acid solution (C.2.1) into the dish. As soon as the coating is completely dissolved from both surfaces, leaving the steel surfaces exposed (see note), 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 successive lots of six discs, combining the acid and washings in the same volumetric flask, finally diluting to the mark with distilled water.

Transfer a 100 ml aliquot portion of the solution to the 500 ml wide-neck conical flask, add 75 ml of the hydrochloric acid solution (C.2.1), 10 ml of the iron(III) chloride solution (C.2.2) and dilute to the mark with water. Add 2 g of the aluminium metal foil (C.2.9). Insert the rubber bung fitted with a small Liebig condenser, a carbon dioxide entry tube and a rubber-sealed burette entry tube (see figure 6). Connect the apparatus to the appropriate supply points and pass the carbon dioxide gas (C.2.10) for 5 min to displace the air within the flask. Heat carefully to boiling, avoiding vigorous evolution of hydrogen. Continue boiling for 5 min to 10 min after solution 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 the starch solution (C.2.6) and titrate with the potassium iodate Solution 1 (C.2.3) to a permanent blue colour.

C.4.2 Electrolytic tinfoil, differentially coated

Degrease with the ether (C.2.7) the specimens from sheets taken in accordance with 8.1 and coat the surfaces carrying the greater tin mass with the cellulose lacquer (C.2.11). 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 (C.2.8) centrally in a shallow dish (see figure 6). Place six of the discs in a circle with the unlacquered surfaces in contact with the platinum wire. Carefully pour 150 ml of the hydrochloric acid solution (C.2.1) into the dish. As soon as the coating is completely dissolved from the unlacquered surfaces, leaving the steel surfaces exposed (see note), 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 successive lots of six discs, combining the acid and washings in the same volumetric flask, finally diluting to the mark with water. Dry the discs and retain for subsequent 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 the hydrochloric acid solution (C.2.1), 10 ml of the iron(III) chloride solution (C.2.2)

and dilute to the mark with water. Continue the reduction and titration as in C.4.1 but using the potassium iodate Solution 2 (C.2.4) as titrant.

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

C.4.3 Hot-dipped tinfoil

Degrease with the ether (C.2.7) the specimens from sheets taken in accordance with 8.1. Place the spiral of platinum wire (C.2.8) centrally in a shallow dish (see figure 6). Place six of the discs in a circle on the platinum wire and carefully pour 150 ml of the hydrochloric acid solution (C.2.1) into the dish. As soon as the coating is completely dissolved from both surfaces, leaving the steel surface exposed (see note), 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 successive lots of six discs, 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 a 500 ml wide-neck conical flask, add 55 ml of hydrochloric acid (ρ 1,16 g/ml), 10 ml of the iron(III) chloride solution (C.2.2) and dilute to the mark with water. Continue the reduction and titration as in C.4.1 but using the potassium iodate Solution 3 (C.2.5) as titrant.

NOTES

- 1 The time required for complete dissolution depends on the coating mass. It may vary from about 3 min for E2,8/2,8 coating to about 15 min for H17/17 coating.
- 2 Care is necessary when adding the aluminium foil, to avoid violent reaction; it is recommended that the foil be cut into small sections beforehand.

C.5 Expression of results

The average tin coating mass expressed in grams per square metre as in tables 1, 2 and 4, is given by the formula

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

where

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

c is the concentration, in moles per litre, of the potassium iodate solution;

A is the total specimen area, in square millimetres (see 8.1).