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INTERNATIONAL STANDARD

ISO 4999

Second edition
1991-04-15

Continuous hot-dip terne (lead alloy) coated cold-reduced carbon steel sheet of commercial and drawing qualities

Tôles en acier au carbone laminées à froid, revêtues d'un alliage au plomb en continu par immersion à chaud, de qualité commerciale et pour emboutissage

Reference number
ISO 4999:1991(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4999 was prepared by Technical Committee ISO/TC 17, *Steel*.

This second edition cancels and replaces the first edition (ISO 4999:1978), table 5 of which has been technically revised and annex A deleted.

Annex A of this International Standard is for information only.

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Continuous hot-dip terne (lead alloy) coated cold-reduced carbon steel sheet of commercial and drawing qualities

1 Scope

1.1 This International Standard applies to cold-reduced carbon steel sheet of commercial and drawing qualities coated by a continuous hot-dip terne (lead alloy) coating process. It includes that group of products commonly known as terne plate or terne sheets (or in the U.S.A. as long ternes). Terne sheets are used where ease of solderability, a degree of corrosion resistance, or amenability to stamping, pressing or deep-drawing will be advantageous. The mass of coating may be specified in accordance with table 2. It is expressed as the total coating on both surfaces in grams per square metre. The coating mass specified should be compatible with the desired service life, thickness of the base metal and the forming requirements involved. A designation system (see clause 4) includes the coating designation, coating condition and quality.

1.2 Terne sheet is normally produced in thicknesses from 0,30 mm to 2,0 mm, and in widths of 600 mm to 1 400 mm in coils and cut lengths. Terne sheet less than 600 mm wide may be slit from wide sheet and will be considered as sheet. Slit sheet is not available from all producers.

1.3 Commercial quality terne sheet (TO 01) is intended for general fabricating purposes where sheet is used in the flat, or for bending or moderate forming.

1.4 Drawing quality terne sheet (TO 02, TO 03 and TO 04) is intended for drawing or severe forming. It is furnished to all the requirements of this International Standard, or, with agreement when ordered, to fabricate an identified part, in which case the mechanical properties in table 3 do not apply. Drawing qualities are identified as follows:

TO 02 Drawing quality

TO 03 Deep drawing quality

TO 04 Deep drawing quality special killed

1.5 Terne sheet is suitable for welding, soldering or brazing if appropriate methods and procedures are selected with special attention to the heavier coatings. When sheet is subjected to joining techniques involving heat, suitable precautions must be taken to avoid toxic effects.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 6892:1984, *Metallic materials — Tensile testing.*

ISO 7438:1985, *Metallic materials — Bend test.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 continuous hot-dip terne (lead alloy) coated cold-reduced steel sheet: A product obtained by hot-dip coating cold-reduced sheet coils on a continuous lead alloy coating line to produce either lead alloy coated coils or lead alloy coated cut lengths.

3.2 terne (lead alloy): In the context of this International Standard, any lead-based alloy in commercial use for the hot-dip coating of steel sheet. Tin is the most common alloying element, but antimony is also commercially used, as are combinations of both elements. If a specific alloy composition is required, it shall be by agreement between the manufacturer and purchaser.

3.3 skin pass: A light cold rolling of terne sheet. The purposes of skin passing are one or more of the following:

a) To minimize temporarily the occurrence of conditions known as stretcher strain (Lüder's lines) or fluting during fabrication of finished parts.

b) To control shape.

c) To produce a higher degree of surface smoothness and to improve appearance or suitability for decorative painting. This process may adversely affect the ductility of the base metal.

4 Designation system — Terne coating and qualities

The produced hot-dip terne coating is designated T0 (the "0" is inserted to fill a computer space and has no significance in the designation) as shown in table 1. The coating mass designation follows the T0 and three spaces are allocated for coating mass designation. If only two spaces are required, such as for designation "75", then the "75" is preceded by a "0" to fill computer space and is shown as "075". If the product is skin passed, designation "S" is used to indicate the coating condition. If the product has not been skin passed, the designation "N" for normal coating (as produced) is shown. The numbers 01, 02, 03 and 04 are common to other standards indicating the qualities of commercial, drawing, deep drawing, and deep drawing special killed. An example of a complete designation, including coating, coating mass, coating condition and quality, is T0120N01. This is composed by combining the following:

T0 = Terne coating
120 = Coating designation (see table 2)
N = Normal coating
01 = Commercial quality

5 Conditions of manufacture

5.1 Steelmaking

The processes used in making the steel and in manufacturing terne sheet are left to the discretion of the producer. When requested, the purchaser should be informed of the steelmaking process being used.

5.2 Chemical composition

The chemical composition of the steel (cast analysis) shall not exceed the values given in table 1.

Table 1 — Chemical composition (cast analysis), %

Quality		C	Mn	P	S
Designation	Name	max.	max.	max.	max.
T0 01	Commercial	0,15	0,60	0,05	0,05
T0 02	Drawing	0,12	0,50	0,04	0,04
T0 03	Deep drawing	0,10	0,45	0,03	0,03
T0 04	Deep drawing special killed	0,08	0,45	0,03	0,03

5.3 Chemical analysis

5.3.1 Cast analysis

A cast analysis of each cast of steel shall be made by the manufacturer to determine the percentage of carbon, manganese, phosphorus and sulphur. On request, this analysis shall be reported to the purchaser or his representative.

5.3.2 Verification analysis

A verification analysis may be made by the purchaser to verify the specified analysis of the semi-finished or finished steel and shall take into consideration any normal heterogeneity. Non-killed steels (such as rimmed or capped) are not technologically suited to verification analysis. For killed steels, the sampling method and deviation limits shall be agreed upon between manufacturer and purchaser at the time of ordering.

5.4 Terne (lead alloy) coating mass

The mass of coating shall conform to the requirements presented in table 2 for the specific coating designation. The mass of coating is the total amount on both surfaces of the sheet, expressed in grams per square metre (g/m^2) of sheet. Methods of checking that the material complies with this International Standard are given in 7.2, 8.2, and annex A. Procedures other than those covered in annex A should be permitted upon agreement between manufacturer and purchaser.

5.5 Application

It is desirable that terne sheet be identified for fabrication by name of the part or by the intended application. Terne sheet of drawing qualities (T0 02, T0 03, T0 04) may be produced to make an identified part within a properly established breakage allowance, which shall be previously agreed upon between producer and purchaser. In this case, the part name, the details of fabrication and special requirements (i.e. exposed or unexposed, freedom from stretcher strains or fluting, coating performance requirements) shall be specified and the mechanical properties of table 3 do not apply.

5.6 Mechanical properties

Except when ordered to an identified part as explained in 5.5, at the time that the steel is made available for shipment, the mechanical properties shall be as stated in table 3 when they are determined on test pieces obtained according to the requirements of 8.1 (mechanical property tests).

NOTE 1 Prolonged storage of the sheet after skin passing can cause a change in mechanical properties (increase in hardness and decrease in elongation), leading to a decrease in drawability. To minimize this effect, quality T0 04 should be specified.

5.7 Strain ageing

Terne sheet (except T0 04) tends to strain age. Grade T0 04 should be specified where strain ageing is not acceptable and where roller levelling is not possible.

5.8 Oiling

Terne sheet is produced either oiled or not oiled, and is usually not degreased.

6 Dimensional tolerances

Applicable tolerance limits for terne sheet are shown in table 5 to table 13 inclusive.

7 Sampling

7.1 Mechanical property tests

7.1.1 Tensile test

When ordered to mechanical properties, one representative sample for the tensile property test required in table 3 shall be taken from each lot of sheet for shipment. A lot consists of 50 tonnes or less of sheet of the same quality rolled to the same thickness and condition.

7.1.2 Bend test

One representative sample for the bend test (applicable only to quality 001) shall be taken from each lot (see 7.1.1) of sheet for shipment.

7.2 Coating tests

7.2.1 Mass of coating

The producer shall make such tests and measurements as he deems necessary to ensure that the material produced complies with the values in table 2. The purchaser may verify the mass of coating by use of the following sampling method:

Three specimens shall be cut, one from the mid-width position, and one from each side not closer than 25 mm from the side edge. The minimum specimen area should be 2 000 mm^2.

7.2.2 Bend test

One representative sample shall be taken from each lot of sheet for shipment. The specimens shall be taken for the coated bend test, not closer than 25 mm from the side edge. The minimum specimen width shall be 50 mm.

8 Test methods

8.1 Mechanical property tests

8.1.1 Tensile test (base metal)

The tensile test shall be performed in accordance with ISO 6892. Transverse or longitudinal test pieces shall be taken mid-way between the centre and edge of the sheet as rolled. Since the tensile test is for determination of properties of the base metal, ends of test pieces should be stripped of the coating to measure base metal thickness for calculation of cross-sectional area.

8.1.2 Bend test (base metal) (applicable only to quality 001)

The transverse bend test piece, stripped of coating in a suitably inhibited acid, shall withstand being bent through 180° in the direction shown in figure 1, around the inside diameter as shown in table 3, without cracking on the outside of the bent portion. The bend test shall be performed at ambient temperature as specified in ISO 7438.

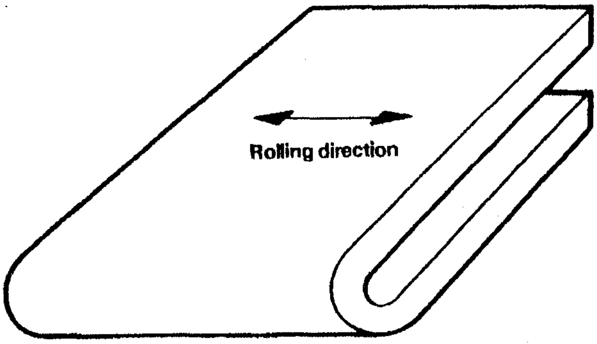


Figure 1 — Transverse bend test piece (after bending)

8.2 Coating tests

8.2.1 Triple spot test

The triple spot test result is the average coating mass found on the three pieces taken according to 7.2.1. The test is normally carried out by stamping out a known area of sheet and calculating the coating mass from the loss in mass after removing the terne (lead alloy) coating in suitably inhibited acid. (See annex A for suggested methods.) Procedures other than those in annex A should be permitted upon agreement by manufacturer and puchaser.

8.2.2 Single spot test

The single spot test result shall be the minimum coating mass found on any one of the three specimens used for the triple spot test. Material slit from wide coil shall be subject to a single spot test.

8.2.3 Bend test (coating)

Bend test pieces taken after coating (before additional processing) shall withstand being bent through 180° in either direction without flaking of the coating on the outside of the bend. The radius of the bend is determined by the number of pieces of the same thickness (or mandrel equivalent) as shown in table 4. Flaking of coating within 7 mm from the edge of the test specimen shall not be cause for rejection.

9 Retests

If a test does not give the required results, two more tests shall be carried out at a random on the same lot. Both retests must conform to the requirements of this International Standard, otherwise, the lot may be rejected.

10 Workmanship

The terne sheet in cut lengths shall be free from laminations, surface flaws and other imperfections that are detrimental to subsequent appropriate processing. Processing for shipment in coils does not afford the producer the opportunity to observe readily or to remove defective portions, as can be carried out on the cut length product.

11 Inspection and acceptance

11.1 While not usually required for products covered by this International Standard, when the purchaser specifies that inspection and tests for acceptance be observed prior to shipment from the manufacturer's works, the manufacturer shall afford the purchaser's inspector all reasonable facilities to determine that the steel is being furnished in accordance with this International Standard.

11.2 Terne sheet that is reported to be defective after arrival at the user's works shall be set aside, properly and correctly identified and adequately protected. The supplier shall be notified in order that he may properly investigate.

12 Coil size

When terne sheet is ordered in coils, a minimum inside diameter (I.D.) or range of acceptable inside diameters shall be specified. In addition, the maximum outside diameter (O.D.) and maximum acceptable coil mass shall be specified.

13 Marking

Unless otherwise stated, the following minimum requirements for identifying the terne sheet shall be legibly stencilled on the top of each lift or shown on a tag attached to each coil or shipping unit:

a) the manufacturer's name or identifying brand;

b) the number of this International Standard;

c) the quality designation;

d) the coating designation;

e) the order number;

f) the product dimensions;

g) the lot number;

h) the mass.

14 Information to be supplied by the purchaser

To specify adequately requirements under this International Standard, inquiries and orders shall include the following information:

a) the number of this International Standard;

b) the name, coating designation, coating condition and quality of material (for example, terne sheet (T0120N02): (see 1.3, 1.4 and clause 4);

c) the dimensions of the product (the thickness includes the coating) and quantity required;

d) the application (name of part or intended usage), if possible (see 5.5);

e) for drawing qualities T0 02, T0 03, T0 04, whether ordered to mechanical properties (see 5.6) or to fabricate an identified part (see 5.5);

f) oiled if required (see 5.7);

g) the coil size requirements (see clause 12);

h) the report of cast analysis, if required (see 5.3.1);

i) details of fabrication or special requirements (fluting or coating performance);

j) inspection and tests for acceptance prior to shipment from the producer's works, if required (see 11.1).

NOTE 2 A typical ordering description is as follows:

ISO 4999, terne sheet, T0100N02, drawing quality, coating designation 100—0,46 × 1 200 × 2 400 mm, 20 000 kg, to fabricate drawn fuel tanks ≠ 7201.

Table 2 — Coating designations and limits

Coating designation	Minimum coating mass limits, g/m² (total both sides)	
	Triple spot test check limits	Single spot test check limits
001	No minimum	No minimum
050	50	40
075	75	60
100	100	75
120	120	90

NOTE — "No minimum" means that there are no established minimum check limits for triple spot and single spot tests.

Table 3 — Mechanical properties

Quality		R_m max. 1) N/mm²	A min. 2)		180°, bend mandrel diameter, for all thicknesses
Designation	Name		L_o = 50 mm	L_o = 80 mm	
T0 01	Commercial				1a
T0 02	Drawing	430	24	23	—
T0 03	Deep drawing	410	26	25	
T0 04	Deep drawing special killed	410	29	28	

R_m = tensile strength

A = percentage elongation after fracture

L_o = gauge length on test piece

a = thickness of bend test piece

1 N/mm² = 1 MPa

1) Minimum tensile strength for qualities T0 02, T0 03 and T0 04 would normally be expected to be 260 N/mm². All tensile strength values are determined to the nearest 10 N/mm².

2) For material up to and including 0,6 mm in thickness, the elongation values in the table shall be reduced by 2. For thicknesses up to 2 mm, use either L_o = 50 mm or L_o = 80 mm.

Table 4 — Coating bend test requirements

180° bend mandrel diameter, for all thicknesses and all coating designations	
Commercial quality	**Drawing qualities**
1*a*	0 (flat on itself)
a = thickness of bend test piece	

Table 5 — Thickness tolerances for coils and cut lengths

Values in millimetres

Specified widths	Thickness tolerances[1]), over and under, for specified thicknesses						
	0,3 up to and including 0,4	over 0,4 up to and including 0,6	over 0,6 up to and including 0,8	over 0,8 up to and including 1,0	over 1,0 up to and including 1,2	over 1,2 up to and including 1,6	over 1,6 up to and including 2,0
up to and including 1 200	0,04	0,05	0,07	0,08	0,09	0,11	0,13
over 1 200	0,05	0,06	0,08	0,09	0,10	0,12	0,14

The thickness tolerances for sheet in coil form are the same as for sheets supplied in cut lengths but in cases where welds are present, the tolerances shall be double those given over a length of 15 m in the vicinity of the weld.

1) Thickness is measured at any point on the sheet not less than 25 mm from a side edge.

Table 6 — Width tolerances for coils and cut lengths, not resquared

Values in millimetres

Specified widths	Tolerance
Up to and including 1 200	$^{+5}_{0}$
Over 1 200	$^{+7}_{0}$

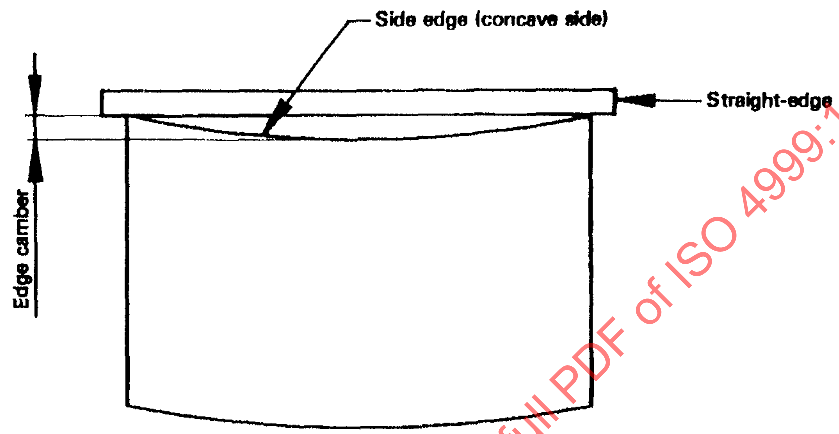
Table 7 — Length tolerances for cut lengths, not resquared

Values in millimetres

Specified lengths	Tolerance
Up to and including 3 000	$^{+20}_{0}$
Over 3 000 up to and including 6 000	$^{+30}_{0}$
Over 6 000	$^{+0,5\ \%}_{0}$

Table 8 — Camber tolerances

Form	Camber tolerance
Coils	20 mm in any 5 000 mm length
Cut lengths	0,4 % × length



Camber is the greatest deviation of a side edge from a straight line, the measurement being taken on the concave side with a straight edge.

Figure 2 — Measurement of camber

Table 9 — Out-of-square tolerance for cut lengths, not resquared

Dimensions	Out-of-square tolerance
All thicknesses and all sizes	1,0 % × width

Table 10 — Out-of-square tolerances for resquared material

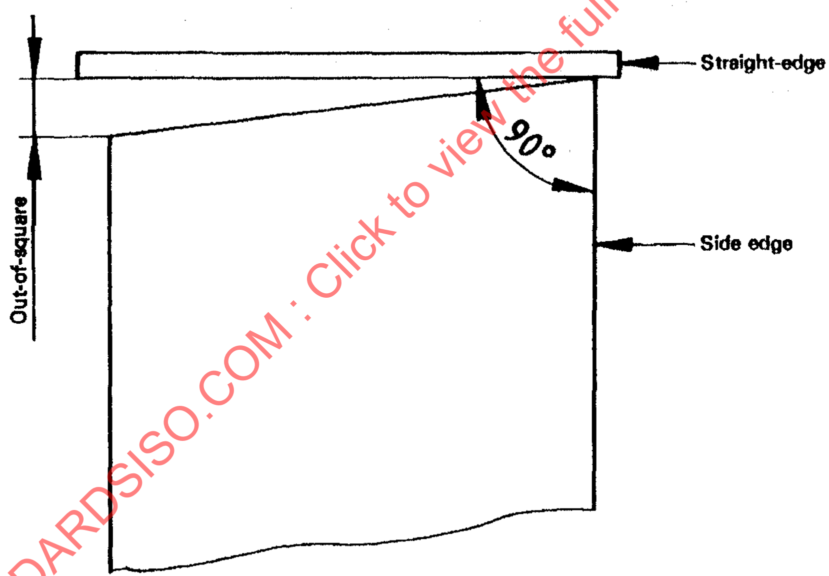
Values in millimetres

Specified lengths	Specified widths	Out-of-square tolerance
Up to and including 3 000	Up to and including 1 200	$^{+2}_{0}$
	Over 1 200	$^{+3}_{0}$
Over 3 000	All widths	$^{+3}_{0}$

NOTES

1 See figure 3.

2 When measuring material ordered to resquared tolerances, consideration may have to be given to extreme variations in temperature.



Out-of-square is the greatest deviation of an end edge from a straight line at right angles to a side and touching one corner, the measurement being taken as shown in figure 3. It can also be measured as one-half the difference between the diagonals of the cut length sheet.

Figure 3 — Measurement of out-of-square

Table 11 — Standard flatness tolerances for cut lengths

Values in millimetres

Specified thicknesses	Specified widths	Flatness tolerance[1)]
Up to and including 0,7	Up to and including 1 200	15
	Over 1 200	18
Over 0,7 up to and including 1,2	Up to and including 1 200	12
	Over 1 200	15
Over 1,2	Up to and including 1 200	10
	Over 1 200	12

These tolerances are only applicable to sheet up to and including 5 000 mm length when their thickness is 1,6 mm or less. This table also applies to sheet cut to length from coil by the purchaser when adequate flattening procedures are performed. Tolerances for sheet exceeding 5 000 mm in length are subject to agreement.

1) Maximum deviation from a flat horizontal surface. With the sheet lying under its own weight on a flat surface, the maximum distance between the lower surface of the sheet and the flat horizontal surface is the maximum deviation from flatness.

Table 12 — Special flatness tolerances for cut lengths, roller-levelled or stretcher-levelled

Values in millimetres

Specified thicknesses	Specified widths	Flatness tolerance[1)]
Up to and including 0,7	Up to and including 1 200	6
	Over 1 200	7
Over 0,7 up to and including 1,2	Up to and including 1 200	5
	Over 1 200	6
Over 1,2	Up to and including 1 200	4
	Over 1 200	5

Tolerances for sheet exceeding 5 000 mm in length are subject to agreement.

1) Maximum deviation from a flat horizontal surface. With the sheet lying under its own weight on a flat surface, the maximum distance between the lower surface of the sheet and the flat horizontal surface is the maximum deviation from flatness (see figure 4).

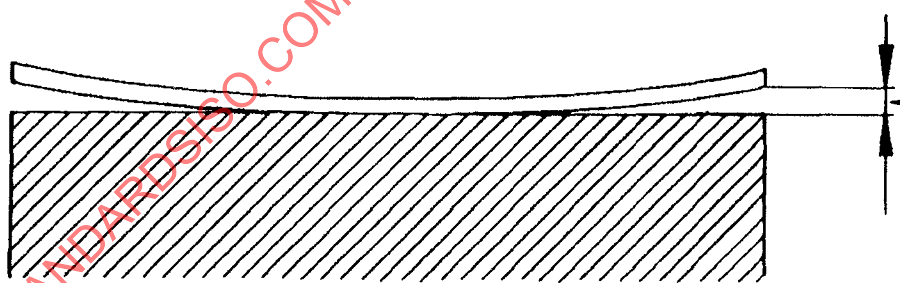


Figure 4 — Measurement of flatness

When terne sheet is specified to stretcher-levelled standard of flatness and not resquared, the allowances over specified dimensions in width and length given in table 13 apply. Under these conditions, the allowances for width and length are added by the manufacturer to the specified width and length and the tolerances given in table 6 and table 7 apply on the basis of the new size established. The camber tolerances in table 8 do not apply.

When sheet is not to have grip or entry marks within the specified length, the purchaser shall specify "grip or entry marks outside specified length". When sheet may have grip or entry marks within the specified length, the purchaser shall specify "grip or entry marks inside specified length".

Table 13 — Width and length allowances for material with stretcher-levelled standard of flatness

Values in millimetres

Specified lengths	Allowances over specified dimensions		
	Width	Length	
		Grip or entry marks outside specified length	Grip or entry marks inside specified length
Up to and including 3 000	19	100	75
Over 3 000 up to and including 4 000	25	100	75
Over 4 000	32	125	100
Allowances for sheet exceeding 5 000 mm in length are subject to agreement.			

Annex A
(informative)

Determination of mass and composition of coating on terne (lead alloy) coated sheet

A.1 General

A.1.1 This annex covers the determination of the mass and composition of coating on terne sheets by the triple-spot method. Five procedures are described, as follows:

a) Procedure A: stripping with sulfuric acid.

b) Procedure B: electrolytic stripping.

c) Procedure C: stripping with silver nitrate solution.

d) Procedure D: stripping with hydrochloric acid and antimony(III) chloride.

e) Procedure E: stripping with sodium hydroxide and sodium peroxide.

A.1.2 If the percentage of tin or lead, or both, in the coating is required, stripping with sulfuric acid is the preferred procedure. Electrolytic stripping, and stripping with silver nitrate or antimony(III) chloride, or sodium hydroxide and sodium peroxide, are convenient procedures where a large number of determinations of mass of coating are to be made.

A.2 Test specimens

A.2.1 Test specimens shall be stamped or cut from the sheet, usually from the centre and near both edges.

A.2.2 Specimens shall be 50 mm ± 0,25 mm square or 56,42 mm ± 0,25 mm in diameter, except that for material narrower than 50 mm in width, test specimens shall be of such a length that the area of the specimen is equal to 2 500 mm^2. The mass of coating in grams on a specimen 50 mm square (2 500 mm^2 in area) when multiplied by 400 is equal to the mass of coating in grams per square metre of sheet. When it is not possible to secure a specimen of 2 500 mm^2 in area, a smaller size may be used, but it is recommended that a specimen of not less than 2 000 mm^2 be used.

A.2.3 The specimens shall be clean; if necessary, they shall be washed with solvent naphtha or other suitable solvent, then with alcohol, and dried thoroughly.

A.3 Procedure A — Stripping with sulfuric acid

A.3.1 Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A.3.1.1 Cerium(IV) sulfate, standard volumetric solution (1 ml ≙ 0,005 6 g Fe, 0,1 N).

Prepare and standardize so that the solution will be 0,1 N.

A.3.1.2 Erioglaucine indicator.

Prepare a 0,1 % (*m*/*m*) solution of erioglaucine A in water.

A.3.1.3 Hydrochloric acid, ρ 1,12 g/ml.

Mix 500 ml of chemically pure or reagent grade hydrochloric acid (ρ 1,19 g/ml) with 400 ml of distilled water.

A.3.1.4 Mercury(II) chloride solution (50 g/l).

Dissolve 50 g of mercury(II) chloride ($HgCl_2$) in water and dilute to 1 litre.

A.3.1.5 Potassium iodate, standard volumetric solution (1 ml ≙ 0,003 g Sn, 0,05 N).

Prepare and standardize so that the solution will be 0,05 N.

A.3.1.6 Potassium permanganate, saturated solution.

A.3.1.7 Sodium hydrogen carbonate, saturated solution.

Saturate freshly boiled distilled water with sodium hydrogen carbonate ($NaHCO_3$).

A.3.1.8 Sodium hydrogen carbonate, dilute solution.

Dissolve about 10 g of sodium hydrogen carbonate ($NaHCO_3$) in 1 litre of freshly boiled distilled water.

A.3.1.9 Tin(II) chloride solution.

Dissolve 150 g of tin(II) chloride dihydrate ($SnCl_2.2H_2O$) in 1 litre of hydrochloric acid, diluted 1 + 2 ($V + V$).

A.3.1.10 Starch solution.

A.3.1.11 Sulfuric acid, ρ 1,84 g/ml, chemically pure or reagent grade.

A.3.2 Procedure for stripping

Accurately determine the mass of each test specimen in grams. Wrap a stiff platinum or nickel wire around each specimen in such a manner that it may be held firmly in an acid solution in a horizontal position. Using a 400 ml beaker, heat 60 ml of the sulfuric acid (A.3.1.11) to 250 °C. (**Caution**, see below.) Immerse each specimen for about 1 min in the hot acid; then remove and momentarily immerse in 50 ml of distilled water contained in a 600 ml beaker. Rub the surface of the specimen with a rubber-tipped glass rod while washing with about 50 ml of distilled water from a wash bottle. If the coating has not been completely removed, again immerse in the acid and repeat the procedure. Thoroughly dry and reweigh the specimen. The loss in mass, Δm, represents the mass of coating, together with some iron dissolved from the steel sheet.

CAUTION — A suitable face shield should be worn in order to protect the operator from accidental splashing or popping of hot sulfuric acid.

A.3.3 Chemical analysis

A.3.3.1 Test solution

Cool the sulfuric acid solution in which the specimen was stripped and combine with the washings obtained in the 600 ml beaker while stripping the specimen. Pour the solution into a 500 ml volumetric flask, and rinse the beaker with hydrochloric acid (A.3.1.3). Add the rinsings to the flask and dilute to the 500 ml mark with hydrochloric acid (A.3.1.3), again diluting to the mark after cooling, if necessary. The solution may now be analysed for iron and, if desired, lead and tin.

A.3.3.2 Determination of iron

Transfer a 100 ml aliquot of the solution in the 500 ml volumetric flask (see A.3.3.1) to a 600 ml beaker. Add 1 ml to 2 ml of the saturated potassium permanganate solution (A.3.1.6) to oxidize the iron and tin, and heat to boiling. Cool and add a slight excess of ammonia. Filter, and wash the precipitate with hot water. Dissolve the precipitate in the original beaker with hydrochloric acid (A.3.1.3) and hot water. Evaporate the solution to a volume of about 10 ml and reduce with the tin(II) chloride solution (A.3.1.9). Add 10 ml of the mercury(II) chloride solution (A.3.1.4) and 400 ml of water. Titrate with the standard volumetric cerium(IV) sulfate solution (A.3.1.1), using erioglaucine (A.3.1.2) as indicator. Calculate the mass, m_{Fe}, in grams, of iron dissolved from the test specimen using the formula

$$m_{Fe} = 5 m_1 V_1$$

where

V_1 is the volume, in millilitres, of the standard volumetric cerium(IV) sulfate solution required for the titration;

m_1 is the mass, in grams, of iron equivalent to 1 ml of the standard volumetric cerium(IV) sulfate solution.

A.3.3.3 Determination of tin

Transfer a 200 ml aliquot of the solution in the 500 ml volumetric flask (see A.3.3.1) to a 300 ml Erlenmeyer flask. Add 3 g of iron in the form of fine wire or thin sheet and 1 g of powered antimony. Fit the flask with a one-hole rubber stopper containing a glass tube bent twice at right angles, with the end of the short bend projecting through the stopper, the other end being long enough to reach almost to the bottom of a beaker placed on a level with the flask. Pour about 300 ml of the dilute sodium hydrogen carbonate solution (A.3.1.8) into this beaker. Place the flask on a hot-plate, with the glass tube extending into the beaker containing the dilute sodium hydrogen carbonate solution. After boiling the solution in the flask for about 5 min, remove the beaker containing the dilute sodium hydrogen carbonate solution and substitute another containing about 50 ml of the saturated sodium hydrogen carbonate solution (A.3.1.7). Move the beaker and the flask to a cool place. This will cause a small amount of the saturated sodium hydrogen carbonate solution to enter the flask and exclude the air. Finally cool the solution to about 10 °C. Add several millilitres of the starch solution (A.3.1.10) and titrate with the standard volumetric potassium iodate solution (A.3.1.5). It is desirable to run a duplicate analysis for tin, adding the standard volumetric potassium iodate solution quickly to a point slightly less than the end point found in the previous determination, then finishing the titration more slowly. This duplicate analysis may be run using one of the other test specimens. Calculate the mass, m_{Sn}, in grams, of tin stripped from the test specimen using the formula

$$m_{Sn} = \frac{5m_2V_2}{2}$$

where

V_2 is the volume, in millilitres, of the standard volumetric potassium iodate solution required for the titration;

m_2 is the mass, in grams, of tin equivalent to 1 ml of the standard volumetric potassium iodate solution.

A.3.3.4 Determination of lead

After ascertaining that all the lead(II) chloride is in the solution, remove a 100 ml aliquot from the 500 ml volumetric flask (see A.3.3.1) and place in a 400 ml beaker. Add 10 ml of the sulfuric acid (A.3.1.11), cover, and evaporate to fumes of sulfur trioxide. Cool and dilute to 200 ml with water. Allow to settle and then filter on a weighed Gooch crucible, washing with sulfuric acid, diluted 1 + 19 ($V + V$). Dry and ignite at a dull red heat. Cool and reweigh. Calculate the mass, m_{Pb}, in grams, of lead stripped from the test specimen using the formula

$$m_{Pb} = 5(m_3 - m_4) \times 0{,}6831$$

where

m_3 is the mass, in grams, of the Gooch crucible and ignited precipitate;

m_4 is the mass, in grams, of the Gooch crucible.

A.3.4 Calculations

A.3.4.1 Mass of coating

Calculate the mass of coating, in grams per square metre, using the formula

$$400(\Delta m - m_{Fe})$$

where

Δm is the loss in mass, in grams, from the specimen (see A.3.2);

m_{Fe} is the mass, in grams, of iron dissolved from the test specimen (see A.3.3.2) where a specimen of 2 500 mm² in area is used. For specimens of other sizes, a suitable correction factor shall be applied.

A.3.4.2 Percentage of tin in coating

Calculate the percentage, by mass, of tin in the coating using the formula

$$\frac{m_{Sn}}{m_{Sn} + m_{Pb}} \times 100$$

where

m_{Sn} is the mass, in grams, of tin stripped from the test specimen (see A.3.3.3);

m_{Pb} is the mass, in grams, of lead stripped from the test specimen (see A.3.3.4).

A.3.4.3 Percentage of lead in coating

Calculate the percentage of lead in the coating by subtracting the percentage of tin (see A.3.4.2) from 100 %.

A.4 Procedure B — Electrolytic stripping

A.4.1 Reagents

A.4.1.1 Hydrochloric acid, diluted 1 + 3 ($V + V$).

Mix 1 part of chemically pure or reagent grade hydrochloric acid (ρ 1,19 g/ml) with 3 parts of water.

A.4.1.2 Sodium hydroxide solution (100 g/l).

Dissolve 100 g of sodium hydroxide (NaOH) in distilled water and dilute to 1 litre.

A.4.2 Procedure for stripping

After cleaning the test specimens as described in A.2.3, weigh each specimen separately. Strip anodically in the sodium hydroxide solution (A.4.1.2) at a temperature of 77 °C to 80 °C for 10 min to 12 min, or until the coating is completely removed, using a current of 4 A. Reverse the current for 5 s to 15 s and then turn it off. Remove the test specimens and wash with water. Dip in hydrochloric acid (A.4.1.1) for 1 s to 2 s and rinse again. Dry and reweigh the specimens separately.

NOTE 3 The test specimens may be conveniently supported vertically by their bottom edges with two hook-shaped pieces of copper wire fastened to a copper bar laid across a 2 litre beaker containing the stripping solution, which may be used for a number of specimens. The cathodes, consisting of two pieces of lead sheet, may be supported on each side of the test specimen by the edge of the beaker. A 6 V automobile battery or a low-voltage rectifier are suitable sources of current, a series resistance being utilized for adjusting the current to 4 A.

A.4.3 Calculation

The loss in mass, in grams, multiplied by 400 is equal to the coating mass, in grams per square metre of sheet, when a specimen of 2 500 mm² in area is used. For specimens of other sizes, a suitable correction factor shall be applied.

A.5 Procedure C — Stripping with silver nitrate solution

A.5.1 Reagent

A.5.1.1 Silver nitrate solution (200 g/l).

Dissolve 200 g of silver nitrate ($AgNO_3$) in distilled water and dilute to 1 litre.

A.5.2 Procedure for stripping

A.5.2.1 After cleaning as described in A.2.3, weigh each test specimen separately. Then place in a beaker containing 200 ml of the silver nitrate solution (A.5.1) (see note 4). Reaction is fairly rapid. The terne (lead alloy) coating goes into solution and metallic silver is deposited on the surface of the steel. Remove the silver by washing the test specimen with a stream of water from a wash bottle and rubbing with a rubber-tipped glass rod.

A.5.2.2 Examine for lead spots. If any are found, repeat the immersion until all the coating is removed. Then thoroughly wash and dry the test specimen and reweigh.

NOTE 4 The silver nitrate solution may be used repeatedly as long as lead is removed. The number of specimens that can be stripped with 200 ml of solution depends on the amount of coating on the sheets. The solution is used cold and replaced with fresh solution when the reaction becomes too slow. After the first specimen is stripped, the silver nitrate solution is discoloured. Most of the silver can be recovered from the used solution and washings, if desired.

A.5.3 Calculation

The loss in mass, in grams, multiplied by 400 is equal to the coating mass, in grams per square metre of sheet, when a specimen of 2 500 mm² in area is used. For specimens of other sizes, a suitable correction factor shall be applied.

A.6 Procedure D — Stripping with hydrochloric acid and antimony(III) chloride

A.6.1 Reagent

A.6.1.1 Antimony(III) chloride — hydrochloric acid solution.

Dissolve approximately 40 g of antimony(III) chloride ($SbCl_3$) in chemically pure or reagent grade hydrochloric acid (ρ 1,19 g/ml) and dilute to 1 litre with the hydrochloric acid.

A.6.2 Procedure for stripping

After cleaning as described in A.2.3, weigh each test specimen separately. Immerse in the cold antimony(III) chloride-hydrochloric acid solution (A.6.1) until the coating is removed and the reaction ceases. The reaction will leave the specimen coated with antimony. Wash thoroughly to remove the loosely adherent antimony, dry, and reweigh.

A.6.3 Calculation

The loss in mass, in grams, multiplied by 400 is the mass of coating, in grams per square metre of sheet, when a specimen of 2 500 mm² in area is used. For specimens of other sizes, a suitable correction factor shall be applied.

A.7 Procedure E — Stripping with sodium hydroxide and sodium peroxide

A.7.1 Reagents

A.7.1.1 Sodium hydroxide solution, 5 % (*m*/*m*).

Dissolve 50 g of sodium hydroxide (NaOH) in 1 litre of water.

A.7.1.2 Sodium peroxide, granulated.[1)]

A.7.2 Procedure for stripping

After cleaning as described in A.2.3, weigh each test specimen separately. Fill a porcelain dish (approximately 120 mm in diameter) to about three-quarters capacity with the sodium hydroxide solution (A.7.1.1) and heat to 50 °C on a hot-plate. Place the specimen in the dish, making sure it is completely immersed. Slowly add the granulated sodium peroxide (A.7.1.2) until the solution becomes clear. Turn the test specimen over to expose the other side and add sodium peroxide until the solution again becomes clear. Stripping is complete when no more lead is detectable on the specimen. Discard the used sodium hydroxide solution. Carefully rinse the specimen with water, dry and reweigh.

A.7.3 Calculation

The loss in mass, in grams, multiplied by 400 is the mass of coating, in grams per square metre of sheet, when a specimen of 2 500 mm² in area is used. For specimens of other sizes, a suitable correction factor shall be applied.

1) It is important to use only granulated sodium peroxide.