INTERNATIONAL STANDARD

ISO 4999

Sixth edition
2014-07-01

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

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

Reference number
ISO 4999:2014(E)

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Foreword

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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).

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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 12, *Continuous mill flat rolled products*.

This sixth edition cancels and replaces the fifth edition (ISO 4999:2011), which has been technically revised.

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

1 Scope

This International Standard is applicable to cold-reduced carbon steel sheet of commercial, drawing, and structural qualities coated by a continuous hot-dip terne (lead alloy) coating process. It includes the group of products commonly known as terne plate or terne sheets (or, in the USA, as terne coated).

Terne sheets are used where ease of solderability, a degree of corrosion resistance, or amenability to stamping, pressing, or deep drawing would be advantageous.

Terne (lead alloy) coated steel sheet can be ordered in one of two ordering conditions:

a) condition A: steel ordered to satisfy mechanical property requirements;

b) condition B: steel ordered to make an identified part.

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

ISO 7438, *Metallic materials — Bend test*

ISO 16162, *Cold-rolled steel sheet products — Dimensional and shape tolerances*

ISO 16163, *Continuously hot-dipped coated steel sheet products — Dimensional and shape tolerances*

3 Terms and definitions

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

3.1 Quality applications

3.1.1
commercial
intended for general fabricating purposes where sheet is used in the flat condition, or for bending or moderate forming

3.1.2
drawing
intended for parts where drawing or severe forming might be involved

3.1.3
deep drawing
intended for parts where severe forming or severe drawing might be involved

3.1.4
deep drawing/aluminum killed (non-aging)
intended for fabricating parts where particularly severe drawing or forming might be involved or essential freedom from aging is required

3.1.5
extra-deep drawing (stabilized)
intended for applications requiring interstitial-free steel (IF) which is non-aging and has maximum formability

3.1.6
structural quality
structural quality which is available in several grades and classes

Note 1 to entry: See Tables 2 and 6.

3.2
aluminum killed
steel which has been deoxidized with sufficient aluminum to prevent the evolution of gas during solidification

3.3
stabilized interstitial-free steel
extra-low-carbon steel in which all interstitial elements are combined with titanium and/or equivalent elements

Note 1 to entry: Stabilized steel is sometimes referred to as interstitial-free steel.

3.4
terne
lead alloy
any lead-based alloy in commercial use for the hot-dip coating of steel sheet

Note 1 to entry: Tin is the most common alloying element, but antimony is also used commercially, as are combinations of both elements.

Note 2 to entry: If a specific alloy composition is required, it shall be by agreement between the manufacturer and purchaser.

3.5
skin pass
light cold rolling of the coated steel sheet

Note 1 to entry: The purpose of the skin pass is to produce a higher degree of surface smoothness and thereby improve the surface appearance. The skin pass also temporarily minimizes the occurrence of a surface condition known as stretcher strain (Luder’s lines) or fluting during the fabrication of finished parts. The skin pass also controls and improves flatness. Some increase in hardness and some loss in ductility will result from skin passing.

3.6
differential coating
coating having a coating mass on one surface significantly different from the coating mass on the other surface

3.7
lot
50 t or less of sheet of the same grade rolled to the same thickness and coating condition

4 Dimensions

4.1 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 can be slit from wide sheet and will be considered as sheet. Slit sheet is not available from all producers.

4.2 The thickness of hot-dip terne (lead alloy) coated steel sheet can be specified as a combination of base metal and metallic coating, or as base metal alone. The purchaser shall indicate on the order which specification method is required. In the event that the purchaser does not indicate any preference, the thickness as a combination of the base metal and coating will be provided. Annex B describes the requirements for specifying the thickness as base metal alone.

5 Conditions of manufacture

5.1 Chemical composition

The chemical composition (heat analysis) shall not exceed the values given in Tables 1, 2, and 3. On request, a report of the heat analysis shall be made to the purchaser.

A verification analysis can be made by the purchaser to verify the specified analysis of the product and shall take into consideration any normal heterogeneity. The sampling method and deviation limits shall be agreed upon between the interested parties at the time of ordering.

The product analysis tolerances are shown in Table 3 and Table 4.

The processes used in making the steel and in manufacturing terne (lead alloy) sheet are left to the discretion of the manufacturer. When requested, the purchaser shall be informed of the steelmaking process used.

Table 1 — Chemical composition (heat analysis) — Commercial and drawing qualities

Mass fractions in percent

Quality		C max.	Mn max.	P max.	S max.	Ti max.
Designation	**Name**					
T0 01	Commercial	0,15	0,60	0,035	0,035	—
T0 02	Drawing	0,10	0,50	0,025	0,035	—
T0 03	Deep drawing	0,08	0,45	0,03	0,03	[a]
T0 04	Deep drawing aluminium killed	0,06	0,50	0,025	0,035	[a]
T0 05	Extra-deep drawing stabilized	0,02	0,25	0,02	0,02	0,15[a]

[a] For interstitial-free steels only, the value of 0,15 % titanium and 0,10 % maximum for niobium and vanadium are acceptable to ensure that the carbon and nitrogen are fully stabilized.

Table 2 — Chemical composition (heat analysis) — Structural quality

Mass fractions in percent

Grade	C max.	Mn max.	P max.	S max.
TCR220	0,15	1,20	0,035	0,035
TCR250	0,20	1,40	0,035	0,035
TCR320	0,20	1,50	0,035	0,035
TCH550	0,20	1,50	0,035	0,035

Table 3 — Limits on additional chemical elements[a]

Mass fractions in percent

Elements	Heat analysis max.	Product analysis max.
Cu[b] Ni[b]	0,20 0,20	0,23 0,23
Cr[bc]	0,15	0,19
Mo[bc]	0,06	0,07
Nb[e] V[de] Ti[e]	0,008 0,008 0,008	0,018 0,018 0,018

a Each of the elements listed in this table shall be included in the report of the heat analysis. When the amount of copper, nickel, chromium, or molybdenum present is less than 0,02 %, the analysis can be reported as "<0,02 %".

b The sum of copper, nickel, chromium, and molybdenum shall not exceed 0,50 % on heat analysis. When one or more of these elements are specified, the sum does not apply; in which case, only the individual limits on the remaining elements will apply.

c The sum of chromium and molybdenum shall not exceed 0,16 % on heat analysis. When one or more of these elements are specified, the sum does not apply; in which case, only the individual limits on the remaining elements will apply.

d Analysis greater than 0,008 % can be supplied after agreement between the producer and consumer.

e For interstitial-free steels only, the value of 0,15 % titanium and 0,10 % maximum for niobium and vanadium are acceptable to ensure that the carbon and nitrogen are fully stabilized.

Table 4 — Product analysis tolerances

Mass fractions in percent

Element	Maximum of specified element	Tolerance over maximum specified
C	0,20	0,04
Mn	1,50	0,05
P	0,05	0,01
S	0,035	0,01

NOTE The above maximum tolerance is the allowable excess over the specified requirement and not of the heat analysis.

5.2 Mechanical properties

5.2.1 Commercial and drawing qualities

Terne (lead alloy) coated sheet of designations T0 02, T0 03, T0 04, and T0 05 are supplied under the following two ordering conditions.

a) Ordering condition A: Steel, when ordered according to its mechanical properties, at the time the steel is made available for shipment, shall satisfy the applicable requirements of Table 5.

b) Ordering condition B: Steel, when ordered to make an identified part, shall be supplied with a commitment for satisfactory manufacturing performance within a properly established breakage allowance, which shall have been agreed upon previously between the interested parties. In these cases, the part name, the details of fabrication, and special requirements (such as freedom from stretcher strain or fluting) shall be specified.

Prolonged storing of the sheet can result in a change in mechanical properties (increase in hardness and decrease in elongation) leading to a decrease in formability. To minimize this effect, qualities of designations T0 04 and T0 05 should be specified.

Table 5 — Mechanical properties other than structural quality

Quality		R_m max.[a]	A min.[b]		$\overline{r}$ [cde]	$\overline{n}$ [cde]
Designation	**Name**	MPa	L_o = 50 mm	L_o = 80 mm	min.	min.
T0 01	Commercial	—	—	—	—	—
T0 02	Drawing	430	24	23	—	—
T0 03	Deep drawing	410	26	25	—	—
T0 04	Deep drawing aluminium killed	410	29	28	—	—
T0 05	Extra-deep drawing stabi-lized	350	37	36	1,4	0,17

R_m = tensile strength

A = percentage elongation after fracture

L_o = gauge length on test piece

$\overline{r}$ = index of drawability

$\overline{n}$ = index of stretchability

NOTE 1 MPa = 1 N/mm^2

[a] The minimum tensile strength for qualities T0 02, T0 03, T0 04, and T0 05 would normally be expected to be 270 MPa. All tensile strength values are determined to the nearest 10 MPa.

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

[c] $\overline{r}$ and $\overline{n}$ values are only applicable to thickness *a* 0,5 mm. For thickness >2,0 mm, the $\overline{r}$ value is reduced by 0,2.

[d] $\overline{r}$ can also be written as r-bar and $\overline{n}$ can also be written as n-bar.

[e] $\overline{r}$ and $\overline{n}$ values can be modified or excluded from this specification, by agreement between the producer and the purchaser.

5.2.2 Structural quality

The mechanical properties, at the time the steel is made available for shipment, shall satisfy the requirements of Table 6.

Table 6 — Mechanical properties — Structural quality

Grade	R_e min. MPa	R_m min. MPa	A min, %[a]		Coating bend test 180° bend-mandrel diameter	
			L_o = 50 mm	L_o = 80 mm	e < 3 mm	e ≥ 3 mm
TCR220	220	300	22	20	1a	2a
TCR250	250	330	20	18	1a	2a
TCR320	320	400	16	14	3a	3a
TCH550	550	[b]	—	—	—	—

R_e = yield strength — can be either R_{eL} or R_{eH} but not both

R_{eL} = lower yield strength

R_{eH} = higher yield strength

R_m = tensile strength

A = percentage elongation after fracture

L_o = gauge length on test piece

a = thickness of bend test piece

e = thickness of steel sheet, in millimetres

NOTE 1 MPa = 1 N/mm^2.

NOTE 2 R_{eL} can be measured by 0,5 % total elongation proof strength (proof strength under load) or by 0,2 % offset when a definite yield phenomenon is not present.

[a] Use either L_o = 50 mm or L_o = 80 mm.

[b] For grade TCH550, the yield strength approaches the tensile strength and, since there is no hesitation of the pointer or drop of the beam, the lower yield strength (R_{eL}) shall be taken as the strength at 0,5 % total elongation under load in accordance with ISO 6892-1.

5.2.3 Fabrication qualities

Terne (lead alloy) coated steel sheet is available in several fabrication qualities.

— Commercial: intended for general fabrication purposes where sheet is used in the flat condition or for bending or moderate forming.

— Drawing: intended for fabricating parts where drawing or severe forming might be involved.

— Deep drawing: intended for fabricating parts where severe drawing or severe forming might be involved.

— Deep drawing/aluminium killed (non-aging): intended for fabricating parts where particularly severe drawing or forming might be involved or essential freedom from aging is required.

— Extra-deep drawing (stabilized interstitial-free): intended for applications requiring interstitial-free steel (IF), which is non-aging and has maximum formability.

5.3 Coating

5.3.1 Coating mass

The coating mass limits shall conform to the limits for the designations shown in Table 7. The coating mass is the total amount of coating on both sides of the sheet, expressed in grams per square metre.

Table 7 — Coating designations and limits

Coating designation	Minimum coating mass limits, g/m² (total for 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
170	170	125
260	260	215
335	335	275
NOTE 1 The coating mass, in grams per square metre, refers to the total coating on both surfaces. Because of the many variables and changing conditions that are characteristic of continuous hot-dip coating, the coating mass is not always evenly divided between the two surfaces of a sheet, neither is the coating evenly distributed from edge to edge. However, it can normally be expected that no less than 40 % of the single-spot test limit will be found on either surface.		
NOTE 2 "No minimum" means that there are no established minimum check limits for triple-spot and single-spot tests.		
NOTE 3 The coating thickness can be estimated from the coating mass by using the following relationship: 100 g/m² total for both sides ≅ 0,006 8 mm total for both sides.		

5.3.2 Coating adherence

The coated sheet shall be capable of being bent in any direction, in accordance with the mandrel requirements of Table 8 for commercial and drawing qualities and Table 6 for structural quality, without flaking of the coating on the outside of the bend. Flaking of the coating within 7 mm from the edge shall not be cause for rejection.

Table 8 — Coating bend test requirements, excluding structural quality

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	

5.4 Weldability

Terne sheet is suitable for welding, soldering, or brazing if appropriate methods and procedures are selected, with special attention to the heavier coatings. When the mass fraction of carbon increases above 0,15 %, spot welding becomes increasingly difficult. Because the heat of welding might have a significant effect on lowering the strength of grade 550, this grade is not recommended for welding.

WARNING — When sheet is subjected to joining techniques involving heat, suitable precautions shall be taken to avoid toxic effects.

5.5 Surface treatments

5.5.1 Mill passivation

A chemical treatment can be applied to minimize the hazard of wet-storage stains during shipment and storage. However, the inhibiting characteristics of the treatment are limited and if a shipment is received wet, the material shall be used immediately.

5.5.2 Oiling

Oiling prevents marring and scratching of the soft surface during handling or shipping and helps to minimize the hazard of storage stains. The order should specify whether the sheet should be oiled or non-oiled.

5.6 Coated coil joining

Continuous hot-dip coating lines can use various methods to join coil ends. The shipment of coils containing joined ends can be permitted if agreed upon between the manufacturer and purchaser.

5.7 Dimensional and shape tolerances

5.7.1 Dimensional and shape tolerances applicable to the coated steel sheet shall be as given in ISO 16163. The tolerances for thickness apply to products whose thickness is a combination of base metal and coating.

5.7.2 When the base metal thickness is specified, the thickness tolerances of ISO 16163 shall apply to the average product thickness calculated in accordance with Annex B. The tolerances for thickness of the base metal shall be as given in ISO 16162.

6 Sampling

6.1 Chemical composition

The manufacturer shall test each heat to determine compliance with the requirements of Tables 1, 2, and 3.

6.2 Tensile test

When required, one representative transverse test shall be taken from each lot for shipment, to verify compliance with the requirements of Tables 5 and 6. Transverse test pieces shall be taken midway between the centre and the edge of the sheet as rolled.

6.3 Coating tests

6.3.1 Coating mass

The producer shall develop a testing plan with a frequency sufficient to adequately characterize the lot of material and ensure conformance with specification requirements.

The purchaser can conduct verification tests by securing a sample piece approximately 300 mm in length by the as-coated width from which three test specimens will be taken, one from the mid-width position and one from each side, not closer than 25 mm from the side edge. The minimum area of each specimen shall be 1 200 mm^2.

6.3.2 Triple-spot test

The triple-spot test result shall be the average coating mass found on three specimens taken in accordance with 6.3.1.

6.3.3 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 that has been slit from wide coil shall be subject to a single-spot test only.

6.4 Coating adherence

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

7 Test methods

7.1 Tensile test

The tests shall be conducted in accordance with the methods specified in ISO 6892-1. Transverse test pieces shall be taken midway between the centre and edge of the sheet as rolled. The base metal thickness shall be used to calculate the cross-sectional area needed for the tensile test; however, for orders specifying thickness “as base metal only”, there are two permissible methods for determining the base metal thickness.

a) Option A — Determine the actual base metal thickness by direct measurement of the substrate of a specimen whose coating has been removed.

b) Option B — Calculate the base metal thickness by subtraction of the average coating thickness for the appropriate coating designation included in Annex B from the actual coated thickness of the test specimen.

7.2 Coating properties

7.2.1 Coating mass

The manufacturer shall conduct tests for coating mass using the methods given in Annex A.

7.2.2 Coating adherence

Bend tests shall be conducted in accordance with the methods specified in ISO 7438.

8 Designation system

The designation system includes the coating name, coating mass designation, coating surface condition, surface treatment, and base metal quality or grade of structural steel.

8.1 Coating mass

The letters T0 are used to indicate the terne (lead alloy) coating.

Coating mass designations are 001, 050, 075, 100, 120, 170, 260, and 335.

The coating is expressed as the total mass on both surfaces, in grams per square metre. The coating mass specified should be compatible with the desired service life, the thickness of the base metal, and the forming requirements involved.

NOTE For differential coatings, the standard would be top surface before bottom surface. An example of a differential coating designation is: T0170S100C02.

8.2 Coating surface conditions

— N (normal) — as-coated surface

— S (skin passed) — smooth surface

8.3 Surface treatments

— C — mill passivation

— O — oiled

— CO — mill passivation and oiled

8.4 Base metal designations

— T0 01 — commercial

— T0 02 — drawing

— T0 03 — deep drawing

— T0 04 — drawing aluminium killed

— T0 05 — extra-deep drawing stabilized

— structural quality grades in accordance with Table 6

8.5 Examples

8.5.1 An example of a complete designation for a uniform coating is T0120NC01

— T0 — terne (lead alloy) coating

— 120 — coating mass designation

— N — normal surface condition

— C — mill passivation

— 01 — base metal quality

8.5.2 An example of a complete designation for a differential coating is T0120100NC01

— T0 — terne (lead alloy) coating

— 120 — coating mass designation — top surface

— 100 — coating mass designation — bottom surface

— N — normal surface condition

— C — mill passivation

— 01 — base metal quality

9 Retests

9.1 Machining and flaws

If any test piece shows defective machining or develops flaws, it shall be discarded and another test piece shall be substituted.

9.2 Elongation

If the percentage elongation of any test piece is less than that specified in Table 6, and if any part of the fracture is outside the middle half of the gauge length as scribed before the test, the test shall be discarded and a retest shall be carried out.

9.3 Additional tests

If a test does not give the specified results, two additional tests shall be carried out at random on the same lot. Both tests shall conform to the requirements of this International Standard.

10 Resubmission

10.1 The manufacturer can resubmit, for acceptance, the products that have been rejected during earlier inspection because of unsatisfactory properties after they have been subjected to a suitable treatment (for example selection, heat treatment) which, upon request, will be indicated to the purchaser. In this case, the tests should be carried out as if they applied to a new lot.

10.2 The manufacturer has the right to submit the rejected products to a new examination for compliance with the requirements for another quality or grade.

11 Workmanship

The terne (lead alloy) coated steel sheet in cut lengths shall be free from amounts of laminations, surface flaws, and other imperfections that are detrimental to subsequent processing. Processing for shipment in coils does not afford the manufacturer the opportunity to observe readily or to remove defective portions, as is possible for cut-length products.

12 Inspection and acceptance

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

12.2 Steel that is reported to be defective after arrival at the user's works shall be set aside, properly and correctly identified, and adequately protected.

13 Coil size

When hot-dip terne coated steel sheet is ordered in coils, a minimum or range of acceptable inside diameter(s) (ID) shall be specified. In addition, the maximum outside diameter (OD) and the maximum acceptable coil mass shall be specified.

14 Marking

Unless otherwise stated, the following minimum requirements for identifying the steel shall be legibly stenciled 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 (i.e. ISO 4999:2014);

c) the designation (coating, coating mass, coating condition, surface treatment, and quality or grade);

d) order number;

e) product dimensions;

f) lot number;

g) mass.

15 Information to be supplied by the purchaser

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

a) the number of this International Standard (i.e. ISO 4999:2014);

b) the name and designation of the material, i.e. T0, base metal quality, coating surface condition, coating mass designation, coating type, and surface treatment;

EXAMPLE Terne (lead alloy) coated steel sheet, commercial quality, normal (as-coated) surface, T0 100, mill passivated, T0100NC01.

c) dimensions: for cut lengths, thickness (combination of base metal and coating or base metal alone), width, length, and bundle mass and the total quantity required; for coils, thickness (combination of base metal and coating or base metal alone), width, minimum or range of inside diameter, maximum outside diameter, and acceptable maximum coil mass and quantity required;

NOTE 1 When the base metal thickness alone is specified, see Annex B for details.

NOTE 2 When the method of specifying thickness is not specified, the combination of base metal and coating will be provided.

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

e) ordering condition A or B (see 5.2.1);

f) surface treatment, mill passivation and/or oiled (see 5.5);

g) the report of heat analysis, if required (see 5.1);

h) the coil size requirements (see Clause 13);

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 Clause 12);

k) when hot-dip terne alloy-coated steel sheet is ordered in coils, a minimum or range of acceptable inside diameters(s) (ID) shall be specified. In addition, the maximum outside diameter (OD) and the maximum acceptable coil mass shall be specified.

NOTE A typical ordering description is as follows:

International Standard ISO 4999:2014, terne sheet, T0 100, drawing quality, skin pass, coating designation 100, mill passivation (T0100SC02), 0,46 mm × 1 200 mm × 2 400 mm, 20 000 kg, to fabricate drawn fuel tanks, part number 7201.

Annex A
(normative)

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 that is 50 mm^2 (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 can 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 another 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 can 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 mol/l).

Prepare and standardize so that the solution is 0,1 mol/l.

A.3.1.2 Erloglaucine indicator.

Prepare a 0,1 % (mass fraction) solution of erloglaucine 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 l.

A.3.1.5 Potassium iodate, standard volumetric solution (1 ml = 0,003 g Sn, 0,05mol/l).

Prepare and standardize so that the solution will be 0,05 mol/l.

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 l of freshly boiled distilled water.

A.3.1.9 Tin(II) chloride, solution.

Dissolve 150 g of tin(II) chloride dihydrate ($SnCl_2{\cdot}2H_2O$) in 1 l of hydrochloric acid, diluted 1:2 (volume fraction).

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 can 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. (See Caution 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, m, represents the mass of coating, together with some iron dissolved from the steel sheet.

CAUTION — A suitable face shield shall 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 can 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(lI) 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 erloglaucine (A.3.1.2) as indicator. Calculate the mass, m_{Fe}, in grams, of iron dissolved from the test specimen using Formula (1):

$$m_{Fe} = 5 m_1 V_1 \qquad (1)$$

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 hotplate, 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). Then 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, by 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 can be run using one of the other test specimens. Calculate the mass, m_{Sn}, in grams, of tin stripped from the test specimen using Formula (2):

$$\%\ Sn = m_{Sn} = \frac{5 m_2 V_2}{2} \qquad (2)$$

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 pour it into 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 weighted Gooch crucible, while washing with sulfuric acid, diluted 1:19 (volume fraction). 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 Formula (3):

$$m_{Pb} = 5(m_3 - m_4) \times 0{,}6831 \quad (3)$$

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 Formula (4):

$$m_c = 400\,(m - m_{Fe}) \quad (4)$$

where

m 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 mm^2 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 Formula (5):

$$\%\ Sn = \frac{m_{Sn}}{m_{Sn} + m_{Pb}} \times 100 \quad (5)$$

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.3.3.3) from 100 %.

A.4 Procedure B — Electrolytic stripping

A.4.1 Reagents

A.4.1.1 Hydrochloric acid, diluted 1:3 (volume fraction).

Mix one part of chemically pure or reagent-grade hydrochloric acid (ρ 1,19 g/ml) with three 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 l.

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 The test specimens can 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 l beaker containing the stripping solution, which can be used for a number of specimens. The cathodes, consisting of two pieces of lead sheet, can 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 mm^2 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 l.

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.5.1.1; see note to A.5.2.2). The 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 it.

NOTE The silver nitrate solution can 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 mm^2 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 l with 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.6.1.1) 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 mm^2 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 % (mass fraction).

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

A.7.1.2 Sodium peroxide, granulated.

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 hotplate. Place the specimen in the dish; make 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.

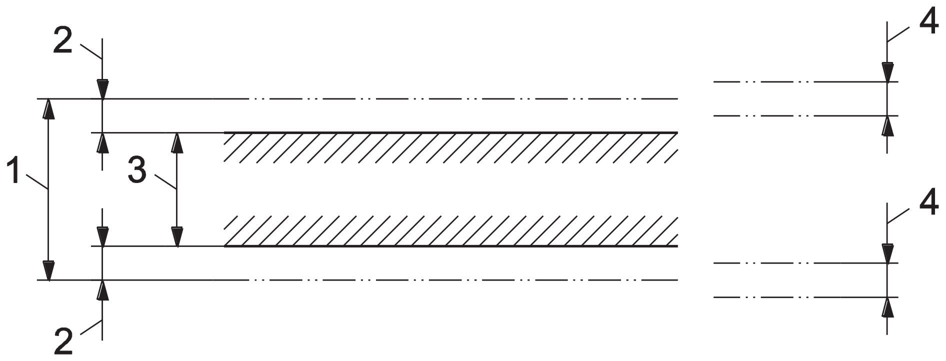
A.7.3 Calculation

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 mm^2 in area is used. For specimens of other sizes, a suitable correction factor shall be applied.

Annex B
(informative)

Orders requiring base metal thickness

When specified by the purchaser, the ordered thickness shall be the base metal thickness. In these cases, the average coated-product thickness shall be calculated as the base metal thickness plus average thickness for each surface (see Table B.1) of the coating mass, as indicated in Figure B.1. Thickness tolerance tables apply to the average coated-product thickness.



Key

1 average coated-product thickness
2 average coating thickness
3 base metal thickness
4 thickness tolerance

Figure B.1 — Calculation of the average coated-product thickness

Table B.1 — Average thickness for coating mass (total for both sides)

Coating designation	Average coating thickness[a] for calculation, mm
050	0,006
075	0,009
100	0,012
120	0,014
170	0,020
260	0,031
335	0,040

a Coating mass data derived from actual production results.

Bibliography

[1] ASTM A308/A308M, *Standard Specification for Steel Sheet, Terne (Lead-Tin Alloy) Coated by the Hot-Dip Process*[1)]

1) This document is recognized by ISO/TC 17/SC 12 to cover a subject similar to that of this International Standard. This information is given for the convenience of users of this International Standard and constitutes neither an endorsement of the document by ISO/TC 17/SC 12 or ISO, nor a statement regarding its degree of equivalence with this International Standard.

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