INTERNATIONAL STANDARD

ISO 10111

First edition
2000-06-15

Metallic and other inorganic coatings — Measurement of mass per unit area — Review of gravimetric and chemical analysis methods

Revêtements métalliques et autres revêtements inorganiques — Mesurage de la masse surfacique — Présentation des méthodes d'analyse gravimétrique et chimique

Reference number
ISO 10111:2000(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2000

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale

Contents

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10111 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Test methods*.

Annex A forms a normative part of this International Standard.

INTERNATIONAL STANDARD **ISO 10111:2000(E)**

Metallic and other inorganic coatings — Measurement of mass per unit area — Review of gravimetric and chemical analysis methods

WARNING — The methods referred to in this International Standard can involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this International Standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 Scope

1.1 General

This International Standard outlines general methods for determining the average surface density over a measured area of anodic oxide or of a coating deposited autocatalytically, mechanically, by chemical conversion, by electrodeposition, by hot dip galvanizing and by vacuum using gravimetric and other chemical analysis procedures that have attained some degree of national or international standardization.

A variety of procedures are described and include:

- gravimetric procedures for chemical or electrochemical dissolution of the coating or the substrate to determine the coating surface density;
- analytical procedures that utilize dissolution of the coating for photometric or volumetric determination of the coating surface density;
- non-destructive instrumental physical analysis of the coating to determine the surface density.

With the exception of the gravimetric method described in ISO 3892:—, this International Standard does not give the measurement uncertainties of the methods cited.

1.2 Sources

The stripping methods cited in annex A are described in specifications in the open literature or have been used routinely by at least one laboratory.

1.3 Restrictions

The procedures described can be used for many coating-substrate combinations. They cannot be used where neither the coating nor substrate material can be completely removed, one from the other by chemical or physical means and there is a constituent common to both that is not readily separated (e.g. nickel phosphorus alloy on nickel).

NOTE The measurement of very thin coatings on very small pieces can result in a reduction in accuracy and a lack of repeatability. Several measurements using a combination of different procedures on similar samples might overcome this situation.

1.4 Limitations

In principle, the gravimetric procedures can be used to measure very thin coatings or to measure coatings over small areas, but not thin coatings over small areas. The limits depend on the required accuracy; e.g., 2,5 mg/cm^2 of coating might require 1 cm^2, but 0,1 mg/cm^2 of coating would require 25 cm^2 to obtain 2,5 mg of coating. These limitations do not apply to the chemical analysis methods.

The gravimetric method does not indicate the presence of bare spots or sites with thicknesses lower than the specified minimum in the measuring areas. In addition, the single value obtained from each measuring area is the mean thickness of that area. There can be no further mathematical analysis of this single value, for example, for statistical process control purposes.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 2079:1981, *Surface treatment and metallic coatings — General classification of terms.*

ISO 2080:1981, *Electroplating and related processes — Vocabulary*.

ISO 2081:1986, *Metallic coatings — Electroplated coatings of zinc on iron or steel*.

ISO 2082:1986, *Metallic coatings — Electroplated coatings of cadmium on iron or steel*.

ISO 2093:1986, *Electroplated coatings of tin — Specification and test methods*.

ISO 2106:1982, *Anodizing of aluminium and its alloys — Determination of mass per unit area (surface density) of anodic oxide coatings — Gravimetric method*.

ISO 3892:—1), *Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods.*

ISO 4522-1:1985, *Metallic coatings — Test methods for electrodeposited silver and silver alloy coatings — Part 1: Determination of coating thickness*.

ISO 4524-1:1985, *Metallic coatings — Test methods for electrodeposited gold and gold alloy coatings — Part 1: Determination of coating thickness*.

ISO 7587:1986, *Electroplated coatings of tin-lead alloys — Specification and test methods*.

ISO 8407: 1991, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*.

3 Terms and definitions

For the purposes of this standard, the terms and definitions given in ISO 2079 and ISO 2080 apply.

---

1) To be published. (Revision of ISO 3892:1980)

4 Principle

The mass of a coating over a measured area is determined by:

a) weighing the test specimen before and after dissolving the coating in a reagent or electrolyte that does not attack the substrate or

b) weighing the coating after dissolving the substrate in a reagent that does not attack the coating or

c) dissolving both the coating and the substrate, or the coating alone, and quantitatively analysing the resulting solution.

The surface density of the coating is calculated from the mass and area measurements and its thickness is based on the mass, area and density of the coating material.

5 Special equipment

Certain specialized chemical, electrochemical and chemical analysis equipment is required for some of the specific methods referred to in Table A.1. (See clauses 8 and 9.)

6 Preparation of test specimen

6.1 Size

The specimen shall be large enough to permit area and mass measurement of adequate accuracy. (See clauses 8 and 9.)

6.2 Shape

The shape of the test specimen should be such that the surface area can be readily measured without difficulty. A rectangular or circular test specimen is usually suitable.

6.3 Edge condition

If the area to be measured is small and has to be known accurately, the edges may need to be dressed to remove smeared coating, to remove loose burrs and to provide well-defined and (for rectangles) straight edges. This should be considered for areas less than 100 mm^2.

One method of dressing the edges of a rectangular specimen involves clamping the specimen between two plastic or metal blocks with the edge of the specimen flush with the edges of the blocks and then grinding and polishing the edges metallographically.

6.4 Heat treatment

If the substrate has to be dissolved in such a way as to leave the coating intact, it may be desirable to first treat the test specimen so that the coating will not curl up tightly or fall apart. Some gold deposits of 1,5 mg/cm^2 ($< 0{,}9$ µm) will fall apart when their substrates are dissolved, but will support themselves after heat treatment at 120 °C for 3 h. If the thickness of a coating (instead of its surface density) is being determined, a heat treatment that could change the density of the coating material shall not be used.

7 Measurement of coated area

7.1 Measurement method

Since the accuracy of the area measurement shall be greater than the desired accuracy of the surface density measurement, the method of measuring the area depends on the desired accuracy and the specimen size.

The measurement uncertainty of the gravimetric method is normally less than 5 % over a wide range of thicknesses (see ISO 3892).

7.2 Surface measuring equipment

The area can be measured with a planimeter, but it is usually determined by linear measurements. Often a micrometer or vernier calliper is used. For large areas, however, a rule may do.

For maximum accuracy, a measuring microscope shall be used.

It may be difficult to measure directly the area of threaded articles with sufficient accuracy in which case the area shall be determined from drawings or published tables.

7.3 Number of measurements

Because circular or rectangular specimens will not be perfectly circular or rectangular, each dimension shall be measured in three places. For a rectangle, the length of each edge and the length and width through the centre shall be measured and an average obtained for each dimension.

NOTE In the case of a cylinder, one would normally measure the diameter and length. In specifications for metallic coated wire (fencing) that has been electroplated or coated by other processes, the length of the wire specimen is not measured, but is, in effect, calculated from the mass (which is measured anyway), the radius and the density of the substrate material as follows:

$$l = \frac{m}{\pi r^2 \rho_s}$$

where

l is the length;

m is the mass;

r is the radius;

ρ_s is the density of the substrate.

8 Determination of mass of coating by chemical analysis

The chemical analysis method is very general. Both coating and substrate are dissolved in a suitable reagent and then the resulting solution is analysed for the coating material. For each coating-substrate-reagent combination, there may be several analytical methods.

9 Gravimetric determination of mass of coating

9.1 Specimen size

Since the measurement uncertainty of the mass measurement shall be less than the desired measurement uncertainty of the surface density measurement, the test specimen shall be large enough for the coating to be weighed with the desired accuracy.

9.2 Gravimetric analysis equipment

A balance is required for gravimetric analysis, but the required sensitivity of the balance depends on the size of the test specimen, the coating thickness (coating mass) and the required accuracy of the measurement. The analytical balance shall be capable of weighing to an accuracy of 0,1 mg for weighing the test pieces under examination before and after dissolution of the coatings. For anodic and cathodic dissolution, a constant d.c. source is necessary.

9.3 Procedure

9.3.1 General

The mass of coating may be determined by:

a) weighing the test specimen before and after dissolving the coating (see annex A) and taking the difference (see 9.3.2) or

b) dissolving the substrate (see annex A) and weighing the coating directly (see 9.3.3).

NOTE The first time a gravimetric method is used, it should be evaluated in accordance with 9.3.2.2 and 9.3.3.2.

9.3.2 Difference method

9.3.2.1 First, clean the test specimen of any foreign material, then rinse it with alcohol (methanol, ethanol, isopropanol) or other suitable solvent, blow it dry with clean air and weigh it. Immerse the specimen in the appropriate reagent (see annex A) to dissolve the coating either chemically or electrochemically, rinse it with water, then with alcohol, blow it dry with clean air and weigh it again. The loss of mass is the mass of the coating.

9.3.2.2 To determine if any dissolution of the substrate has occurred, repeat the process with the stripped substrate making sure that the substrate is immersed in the reagent for the same length of time as before. Any loss of mass enables one to make a judgement of a possible error due to any dissolution of the substrate with the coating during the stripping process.

9.3.3 Direct weighing method

9.3.3.1 Dissolve the substrate in the appropriate reagent (see annex A). Rinse the coating with water and then alcohol (methanol, ethanol, isopropanol) or other suitable solvent, blow it dry with clean air and weigh it.

9.3.3.2 To determine if any dissolution of the coating occurred, submit the isolated coating to the same stripping process making sure that the coating is immersed in the stripping reagent for the same length of time as it was during the stripping process. Any loss of mass enables one to make a judgement of a possible error due to any dissolution of the coating with the substrate during the stripping process.

10 Calculations

10.1 Surface density

Calculate the surface density, ρ_{A}, in milligrams per square centimetre, from the following equation:

$$\rho_{\mathrm{A}} = \frac{m}{A}$$

where

m is the mass of coating, in milligrams;

A is the area, in square centimetres.

10.2 Thickness

Calculate the thickness, d, in micrometres, from the following equation:

$$d = 10 \times \frac{\rho_{\mathrm{A}}}{\rho_{\mathrm{C}}}$$

where

ρ_{A} is the surface density, in milligrams per square centimetre;

ρ_{C} is the density of the coating in grams per cubic centimetre.

NOTE The density of a coating metal is usually not the same as the published values of bulk or wrought metal. For example, the density of electrodeposited gold is generally less than 19,3 g/cm^3 and sometimes as low or lower than 17 g/cm^3. The densities of some electrodeposited metals are given in [1].

If there is any uncertainty about the numerical value of the density used to calculate the thickness in micrometres from the surface density in milligrams per square centimetre, the density value used shall be stated.

Annex A
(normative)

Reagents for selective dissolution of metal layers

With many of the reagents given in Table A.1, there may be some dissolution of the layer other than the one being stripped. Often the dissolution is not significant, but the possibility should be tested for as described in 9.3.2 and 9.3.3.

Dissolution shall be carried out at room temperature unless otherwise indicated.

All test pieces shall be rinsed and dried before weighing.

Table A.1 — Reagents for selective dissolution of metallic and other inorganic coatings using both chemical and electrochemical methods
(non-destructive instrumental methods do not require any reagents)

Coating	Substrate	Reagents[a]	Comments[b]
Aluminium	Steel	(1) 20 parts by mass NaOH + 80 parts water (2) concentrated HCl	See [1]. Immerse for a few minutes (avoid a longer time) at about 90 °C. While rinsing, scrub with a sponge to remove loose material. Drain off water, immerse for 3 s in concentrated HCl at room temperature, scrub again in running water, repeat the entire process until there is no visible reaction in HCl. Two or three cycles are required normally.
Aluminium	Steel	(1) 200 g $SbCl_3$ in 1 l concentrated HCl (2) 100 g $SnCl_2{\cdot}H_2O$ in 1 l concentrated HCl + few granules of tin	See [1] and [2]. Mix equal volumes of (1) and (2), immerse until evolution of hydrogen stops (about 1 min to 4 min), keep below 38 °C, rinse and scrub with a soft cloth.
Anodized aluminium	Aluminium	35 ml 85 % H_3PO_4 + 20 g CrO_3 per litre	See ISO 2106:1982 and [3] and [4]. Immerse at room temperature, rinse, dry, weigh and repeat until constant mass loss.
Anodized magnesium HAE	Magnesium	300 g/l CrO_3	See [5]. Immerse at room temperature, rinse, dry, weigh and repeat until mass loss is less than 3,9 mg/dm^2. Keep a piece of commercially pure aluminium in solution but not in contact with magnesium.
Brass	Steel	500 g/l CrO_3 + 50 g/l H_2SO_4	Immerse at room temperature with mild agitation. Used routinely in one or more laboratories.
Cadmium	Steel	300 g/l NH_4NO_3	See ISO 2082:1986, 10.1.2 and [6]. Immerse.
Cadmium	Steel	200 g Sb_2O_3 in 1 l concentrated HCl	See [7]. Immerse till evolution of gas practically stops.

Table A.1 *(continued)*

Coating	Substrate	Reagents[a]	Comments[b]
Cadmium	Steel	20 g Sb_2O_3 in 800 ml concentrated HCl + 200 ml water	See ISO 2082:1986, 10.1.2 and [7] and [8]. Immerse till evolution of gas practically stops.
Cadmium	Steel	5 % $(NH_4)S_2O_8$ + 10 % by volume of concentrated NH_4OH solution	See [7]. Immerse.
Chromate	Aluminium	(1) $NaNO_2$ (2) 1 part by volume water + 1 part concentrated HNO_3	See [9]. Immerse in molten $NaNO_2$ at 326 °C to 354 °C for 2 min, rinse in cold water, then in (2) for 30 s at room temperature.
Chromate (aged)	Aluminium and its alloys	(1) 98 % $NaNO_3$ + 2 % NaOH solution (2) 1 part by volume 65 % to 70 % mass fraction HNO_3 + 1 part water	See ISO 3892:—, 4.5.2, and [10]. Immerse in (1) for 2 min to 5 min at 370 °C to 500 °C (some coatings may require the higher temperature). Rinse in water, immerse in (2) for 15 s to 30 s at room temperature.
Chromate (fresh)	Aluminium alloys	1 part by volume water + 1 part 65 % to 70 % mass fraction HNO_3	See ISO 3892:—, 4.5.1 and [10]. Immerse for 1 min at room temperature within 3 h of application of coating.
Chromate (aged) and (fresh)	Aluminium alloys	(1) 500 ml 1 % H_2SO_4 (2) $(NH_4)_2S_2O_8$	See [10]. Immerse in (1) for 10 min at boiling point, evaporate to about 50 ml. Treat with (2) (concentration not critical) to oxidize Cr(III) to Cr(VI). Determine photometrically at wavelength (λ) = 445 nm.
Chromate	Tin or zinc	(1) 100 g KNO_3 + 100 g KCl in 1 l of water (2) 100 g NaCl or KCl in 1 l of water	See [11]. Immerse chromates on tin in (1), rinse, dry and determine gravimetrically. Immerse chromates on zinc in (2), rinse, dry and determine gravimetrically.
Chromate	Tin or zinc	(1) 100 g KNO_3 + 100 g KCl in 1 l of water (2) 100 g NaCl or KCl in 1 l of water	See [12]. Immerse chromates on tin in (1), rinse, dry and determine volumetrically. Immerse chromates on zinc in (2), rinse, dry and determine volumetrically.
Chromate	Cadmium or zinc	50 g/l NaCN or KCN + 5 g/l NaOH	See ISO 3892:—, 4.4. Dissolve cathodically at 15 A/dm^2 at room temperature.
Chromium	Nickel or steel	12 g/l NaOH	See [13]. Chromium dissolves anodically at about 20 mA/cm^2.
Copper	Nickel	(1) Dissolve 200 g Na_2S in 750 ml water, heat to boiling with 20 g S, dilute to 1 l. (2) 20 % NaCN	See [14]. Immerse in (1). When copper becomes black copper sulfide and begins to peel off, rinse and immerse in (2) to dissolve copper sulfide.

Table A.1 *(continued)*

Coating	Substrate	Reagents[a]	Comments[b]
Copper	Nickel or steel	500 g/l CrO_3 + 50 g/l H_2SO_4	See [15] and [13]. Dissolves about 1,2 µm/min.
Copper	Zinc alloys	1 part concentrated HCl + 4 parts water	Dissolves zinc alloy substrate. Cool initial reaction to prevent dissolution of copper. Used routinely in one or more laboratories
Gold	Steel, copper, nickel or Fe-Ni-Co	1 to 3 parts by volume water + 1 part concentrated HNO_3	See ISO 4524-1:1985. Substrate dissolved by immersion. Heat as required. Keep free of halides. Nickel may passivate – make contact with nickel wire to increase area of nickel.
Lead–tin alloys	Steel		See ‘terne plate’.
Nickel	Brass	90 % H_3PO_4	See [16]. Immerse at 180 °C to 190 °C, do not add water. 2,5 µm nickel dissolve in about 10 min.
Nickel	Brass	500 g/l CrO_3 + 50 g/l H_2SO_4	Dissolves brass substrate by immersion at room temperature with mild agitation. Used routinely in one or more laboratories
Nickel	Steel	(1) Fuming HNO_3 with mild agitation, or (2) 1 part fuming + 1 part concentrated HNO_3 (3) 10 % CrO_3	See [17] and [13]. Attack of steel is insignificant. Transfer quickly to CrO_3 to remove HNO_3, then rinse with water. Nickel dissolves more rapidly in (2).
Nickel	Steel	(1) Sodium *meta*-nitrobenzene sulfonate 65 g + NaOH 10 g + NaCN 100 g + water to 1 l (2) Sodium nitrobenzoic acid 65 g + NaOH 20 g + NaCN 100 g + water to 1 l	See [16]. Immerse in (1) or (2) at 75 °C to 85 °C. 7 µm nickel dissolve in about 30 min. Copper undercoat, if used, is also dissolved by these solutions.
Nickel or nickel over copper	Zinc alloys	1 part concentrated HCl + 4 parts water	See [14]. Dissolves zinc alloy substrate. Cool initial reaction to prevent dissolution of copper. Check for dissolution of nickel. To remove copper from nickel, see copper on nickel.
Phosphate (amorphous)	Aluminium and its alloys	1 part by volume water + 1 part 65 % to 70 % mass fraction HNO_3	See ISO 3892:—. Immerse for 1 min at room temperature.
Phosphate (crystalline)	Aluminium and its alloys	65 % mass fraction HNO_3 + 35 % mass fraction H_2O	See ISO 3892:— and [18]. Immerse 5 min at 75 °C, or 15 min at room temperature.

Table A.1 *(continued)*

Coating	Substrate	Reagents[a]	Comments[b]
Phosphate	Cadmium	4 % mass fraction triethanolamine + 12 % mass fraction Na_4EDTA[c] + 9 % mass fraction NaOH + 75 % mass fraction water	See [18]. Immerse for 5 min at 70 °C.
Phosphate	Cadmium or zinc	20 g $(NH_4)_2CrO_7$ in 25 % to 30 % mass fraction NH_4OH	See ISO 3892:—. Immerse for 3 min to 5 min at room temperature.
Phosphate	Steel	20 g/l Sb_2O_3 in concentrated HCl	[19]. Immerse at room temperature, rub off any loose material.
Phosphate	Steel	(1) 4 % mass fraction triethanolamine + 12 % mass fraction Na_4EDTA + 9 % mass fraction NaOH + 75 % mass fraction water (2) 5 % mass fraction CrO_3 + 95 % mass fraction water	[18]. (1) Immerse at 70 °C for 5 min. (2) Immerse at 70 °C for 15 min.
Phosphate (manganese, zinc or iron base)	Steel	50 g/l CrO_3	See ISO 3892:— and [20]. Immerse for at least 15 min at 75 °C ± 5 °C, rinse, dry, weigh and repeat till mass is constant.
Phosphate (zinc base)	Steel	100 g/l NaOH + 90 g/l EDTA tetra-sodium salt + 4 g/l triethanolamine	See ISO 3892:—. Immerse for 5 min at 70 °C ± 5 °C.
Phosphate (zinc base)	Steel	180 g/l NaOH, + 90 g/l NaCN	See [20]. Immerse for at least 10 min, rinse dry, weigh and repeat till mass is constant.
Phosphate (zinc base)	Zinc	(1) 2,2 % mass fraction $(NH_4)_2Cr_2O_7$ + 27,4 % mass fraction NH_3 + 70,4 % mass fraction water (2) 5 % mass fraction CrO_3 + 95 % mass fraction water	See [18]. Immerse in (1) or (2) at 25 °C for 5 min.
Silver	Nickel brass	19 parts by volume concentrated H_2SO_4 + 1 part by volume concentrated HNO_3	See [21]. Immerse at 80 °C.
Silver	Copper alloys	(1) 19 parts by volume concentrated H_2SO_4 + 1 part by volume concentrated HNO_3 (2) concentrated H_2SO_4	See ISO 4522-1:1985 and [22]. Immerse at 60 °C to 70 °C until silver dissolves, dip in concentrated H_2SO_4, rinse.
Silver	Nickel and steel	90 g NaCN + 15 g NaOH, + 1 000 ml water	See ISO 4522-1:1985. Dissolve anodically at 2 V to 4 V

Table A.1 *(continued)*

Coating	Substrate	Reagents[a]	Comments[b]
Silver	Corrosion resisting steel	30 g/l NaCN	See [21]. Dissolve anodically at 3 V to 4 V.
Silver	Tin alloys	30 g/l NaCN	See ISO 4522-1:1985. Dissolve anodically at 4 V.
Terne plate	Long terne sheet (steel)	(1) 100 g/l NaOH (2) 1 volume concentrated HCl + 3 volumes water	See [23]. Strip anodically in NaOH solution, 77 °C to 88 °C, 12 A/dm^2, reverse current 5 s to 15 s, rinse, dip in HCl solution for 1 s to 2 s, rinse, dip in HCl solution for 1 s to 2 s, rinse.
Terne plate	Long terne sheet (steel)	200 g/l $AgNO_3$	See [23]. Immerse, silver replaces terne metal, remove by scrubbing in water and examine for residual terne metal.
Tin	Copper alloys	concentrated HCl	See ISO 2093:1986 and [22] and [24]. Immerse and simmer at 95 °C or higher until tin is dissolved.
Tin	Steel	40 % NaOH	Immerse and heat until evolution of gas stops. Used routinely in one or more laboratories
Tin	Steel	120 g $SbCl_3$ in 1 l concentrated HCl	See [21]. Immerse until evolution of gas stops and then wait 15 s to 30 s.
Tin	Steel	20 g Sb_2O_3 in 1 l concentrated HCl)	See ISO 2093:1986 and [25]. Immerse until 1 min after evolution of gas stops.
Tin–lead alloy	Copper	10 ml concentrated HNO_3 + 15 g urea + 10 ml H_2O_2 (10 volumes) + 80 ml water	See [26]. Alloy dissolves at about 0,1 μm/min. Copper dissolves at about 0,5 mg·dm^{-2}·min^{-1}.
Tin–lead alloy	Copper and copper alloys	50 ml H_2O_2 6 % mass fraction + 50 ml fluoboric acid (HBF_4) 40 % mass fraction	See ISO 7587:1986. Immerse in freshly prepared solution.
Tin–lead alloy	Nickel and steel	20 g/l Sb_2O_3 + 1 000 ml concentrated HCl	See ISO 7587:1986. Immerse.
Tin–nickel alloy	Copper and copper alloys	concentrated H_3PO_4	See [27]. Immerse at 180 °C to 200 °C.
Tin–nickel alloy	Steel	20 g/l NaOH + 30 g/l NaCN	See [27]. Dissolve anodically at near boiling point. If current density is too high, coating passivates and gas is evolved. To reactivate, make cathodic for a few seconds.

Table A.1 *(continued)*

Coating	Substrate	Reagents[a]	Comments[b]
Zinc	Steel	(1) 20 g Sb_2O_3 or 32 g $SbCl_3$ in 1 l concentrated HCl (2) 5 ml of (1) in 100 ml concentrated HCl	See [28]. Immerse in solution (2). Keep below 38 °C until violent evolution of hydrogen has stopped and only a few bubbles are being evolved.
Zinc	Steel	20 g Sb_2O_3 + 50 g $SnCl_2$ in 100 g concentrated HCl	See ISO 8407:1991. Immerse in solution. Keep below 38 °C until violent evolution of hydrogen has stopped and only a few bubbles are being evolved.
Zinc	Steel	20 g Sb_2O_3 + 800 ml concentrated HCl + 200 ml water	See ISO 2081:1986 and [8]. Immerse until effervescence ceases.
Zinc	Steel	3,2 g $SbCl_3$ or 2 g Sb_2O_3 in 500 ml concentrated HCl + water to 1 l	See [29]. Immerse until vigorous reaction virtually ceases, brush off loose deposit.
Zinc	Steel	1 to 1 HCl	See [28]. Immerse in 1 to 1 HCl until violent evolution of hydrogen has stopped.
Zinc	Steel	5 g $(NH_4)_2S_2O_8$ + 10 ml NH_4OH (ρ_{20} 0,880) + 90 ml water	See [6]. Immerse.
Zinc	Steel	10 ml formaldehyde 30 % mass fraction + 500 ml concentrated HCl + 500 ml water	See ISO 2081:1986. Immerse.
Zinc	Steel	300 g/l NH_4NO_3	See ISO 2081:1986. Immerse.
Zinc	Steel	500 g concentrated HCl + 1 g prop-2-yn-1-ol (C_3H_4O) + 500 ml water	See ISO 2081:1986. Immerse.

[a] The relative densities at 20 ºC (ρ_{20}) of the acids referred to in this table are as follows:

- hydrochloric acid HCl, 1,18;
- nitric acid HNO_3, 1,42;
- phosphoric acid H_3PO_4, 1,75;
- sulfuric acid H_2SO_4, 1,84.

[b] See bibliography for references in brackets.

[c] EDTA = ethylene diaminetetraacetic acid.

Bibliography

[1] ASTM A428M-95, *Standard Test Method for Weight [Mass] of Coating on Aluminum-Coated Iron or Steel Articles*.

[2] United States Standard, *Standards for Anodically Coated Aluminum Alloys for Architectural Applications*, 2nd ed. The Aluminum Association, June 1965.

[3] ASTM B137-95, *Standard Test Method for Measurement of Coating Mass Per Unit Area on Anodically Coated Aluminum*.

[4] DIN 50944:1979 2), *Testing of inorganic non-metallic coatings on pure aluminium alloys — Determination of substances by chemical dissolution*.

[5] United States Standard MIL-M45202C, *Anodic Treatment of Magnesium Alloys*.

[6] BS 3382:Parts 1 & 2:1961, *Specification for electroplated coatings on threaded components. Cadmium on steel components. Zinc on steel components*.

[7] CLARKE, S.G., Tests of thickness of protective cadmium coatings on steel. *J. Electrodepositors' Tech. Soc*. 1932-33, **VIII** (11).

[8] BS 1706:1990, *Method for specifying electroplated coatings of zinc and cadmium on iron and steel*.

[9] ASTM B449-93 (1998), *Standard Specification for Chromates on Aluminum*.

[10] DIN 50939:1996, *Corrosion protection — Chromating aluminium — Principles and testing*.

[11] DIN 50988-1:1984 3), *Measurement of coating thickness, determination of mass per unit area of zinc, and tin coatings on ferrous materials by dissolution of the coating material — Gravimetric method*.

[12] DIN 50988-2:1988, *Measurement of coating thickness, determination of mass per unit area of zinc, and tin coatings on ferrous materials by dissolution of the coating material — Volumetric method*.

[13] BRENNER, A., Methods of stripping plated coatings. *Monthly review* (AES), November 1933.

[14] BROWN, H.E., Determination of plate thickness on zinc base alloy diecasting. *Plating*, 1951, **38**, p.556.

[15] READ, H.J. and LORENZ, F.E., Methods for testing thickness of electrodeposits, III. Comparison of methods for acid copper on steel. *Plating*, 1951, **38**, p. 946.

[16] BS 3382:Parts 3 & 4:1965, *Specification for electroplated coatings on threaded components. Nickel or nickel plus chromium on steel components. Nickel or nickel plus chromium on copper and copper alloy (including brass) components.*

[17] READ, H.J. and LORENZ, F.E., Methods for testing thickness of electrodeposits, II. Comparison of methods for nickel on steel. *Plating*, 1951, **38**, p. 225.

[18] DIN 50942:1996, *Phosphating metals — Principles and testing.*

[19] BS 3189:1991, *Method for specifying phosphate conversion coatings for metals*.

---

2) Standard now withdrawn; replaced by DIN/EN 12373-2:1999.

3) Standard now withdrawn; replaced by DIN/EN/ISO1460:1992.

[20] United States Standard DOD-P-16232F, *Phosphate Coatings, Heavy, Manganese or Zinc Base (for Ferrous Metals)*.

[21] United States Standard RR-T-51D. *Tableware and Flatware — Silverplated*.

[22] BS 3382:Parts 5 & 6:1967, *Specification for electroplated coatings on threaded components. Tin on copper and copper alloy (including brass) components. Silver on copper and copper alloy (including brass) components.*

[23] ASTM A309-94a, *Standard Test Method for Weight and Composition of Coating on Terne Sheet by the Triple Spot Test.*

[24] BS 1872:1984, *Specification for electroplated coatings of tin*.

[25] CLARKE, S.G., A rapid test of thickness of tin coatings on steel. *Analyst*, 1934, **59**, p. 525.

[26] PRICE, J.W., Determination of thickness of tin-lead alloy coatings on copper wire. *J. Soc. Chem. Industry*, 1944, **63** (10).

[27] BS 3597:1984, *Specification for electroplated coatings of 65/35 tin/nickel alloy*.

[28] ASTM A90-95a. *Standard Test Method for Weight [Mass] Coating on Iron and Steel Articles with Zinc or Zinc-Alloy Coatings*.

[29] BS 729:1971[4], *Specification for hot dip galvanized coatings on iron and steel articles*.

[30] ISO 1456, *Metallic coatings — Electrodeposited coatings of nickel plus chromium and of copper plus nickel plus chromium*.

[31] ISO 1458, *Metallic coatings — Electrodeposited coatings of nickel*.

[32] ISO 1460, *Metallic coatings — Hot dip galvanized coatings on ferrous materials — Gravimetric determination of the mass per unit area.*

[33] ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*.

[34] ISO 2179, *Electroplated coatings of tin-nickel alloy — Specification and test methods*.

[35] ISO 3882, *Metallic and other inorganic coatings — Review of methods of measurement of thickness*.

[36] ISO 4521, *Metallic coatings — Electrodeposited silver and silver alloy coatings for engineering purposes*.

[37] ISO 4523, *Metallic coatings — Electrodeposited gold and gold alloy coatings for engineering purposes*.

[38] ISO 4525, *Metallic coatings — Electroplated coatings of copper plus nickel plus chromium on plastics materials*.

[39] ISO 4526, *Metallic coatings — Electroplated coatings of nickel for engineering purposes*.

[40] ISO 4527, *Metallic coatings — Autocatalytic (electroless)nickel-phosphorus alloy coatings — Specification and test methods*.

[41] ISO 6158, *Metallic coatings — Electroplated coatings of chromium for engineering purposes*.

---

4) Standard now withdrawn.

[42] United States Standard, FED-STD-151B, Test methods for metals, Method 513.1. *Weight of Coating on Hot Dip Tin Plate and Electrolytic Tin Plate*.

[43] SAFRANEK, W.H., *The Properties of Electrodeposited Metals and alloy*s, 2nd ed. American Electroplaters' and Surface Finishers' Society, 1986.

ICS 25.220.20; 25.220.40

Price based on 15 pages