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**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*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of 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.

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# 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<sup>2</sup> of coating might require 1 cm<sup>2</sup>, but 0,1 mg/cm<sup>2</sup> of coating would require 25 cm<sup>2</sup> 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:—<sup>1)</sup>, *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.

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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

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### 6.1 Size

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

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### 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<sup>2</sup>.

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<sup>2</sup> (< 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:

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

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where

$l$  is the length;

$m$  is the mass;

$r$  is the radius;

$\rho_s$  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,  $\rho_A$ , in milligrams per square centimetre, from the following equation:

$$\rho_A = \frac{m}{A}$$

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:

$$d = 10 \times \frac{\rho_A}{\rho_C}$$

where

$\rho_A$  is the surface density, in milligrams per square centimetre;

$\rho_C$  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<sup>3</sup> and sometimes as low or lower than 17 g/cm<sup>3</sup>. 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 <sup>a</sup>	Comments <sup>b</sup>
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 <sub>3</sub> in 1 l concentrated HCl (2) 100 g SnCl <sub>2</sub> ·H <sub>2</sub> O 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 <sub>3</sub> PO <sub>4</sub> + 20 g CrO <sub>3</sub> 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 <sub>3</sub>	See [5]. Immerse at room temperature, rinse, dry, weigh and repeat until mass loss is less than 3,9 mg/dm <sup>2</sup> . Keep a piece of commercially pure aluminium in solution but not in contact with magnesium.
Brass	Steel	500 g/l CrO <sub>3</sub> + 50 g/l H <sub>2</sub> SO <sub>4</sub>	Immerse at room temperature with mild agitation. Used routinely in one or more laboratories.
Cadmium	Steel	300 g/l NH <sub>4</sub> NO <sub>3</sub>	See ISO 2082:1986, 10.1.2 and [6]. Immerse.
Cadmium	Steel	200 g Sb <sub>2</sub> O <sub>3</sub> in 1 l concentrated HCl	See [7]. Immerse till evolution of gas practically stops.