
**Metallic coatings — Measurement of
coating thickness — Coulometric method
by anodic dissolution**

*Revêtements métalliques — Mesurage de l'épaisseur — Méthode
coulométrique par dissolution anodique*

iTeh Standards
(<https://standards.iteh.ai>)
Document Preview

[ISO 2177:2003](https://standards.iteh.ai/catalog/standards/iso/f2445510-80bf-48c6-aa6a-f90744358163/iso-2177-2003)

<https://standards.iteh.ai/catalog/standards/iso/f2445510-80bf-48c6-aa6a-f90744358163/iso-2177-2003>



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.

iTeh Standards
(<https://standards.iteh.ai>)
Document Preview

[ISO 2177:2003](https://standards.iteh.ai/catalog/standards/iso/f2445510-80bf-48c6-aa6a-f90744358163/iso-2177-2003)

<https://standards.iteh.ai/catalog/standards/iso/f2445510-80bf-48c6-aa6a-f90744358163/iso-2177-2003>

© ISO 2003

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.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Instrumentation	2
6 Electrolyte	2
7 Factors affecting the measuring accuracy	3
7.1 Coating thickness	3
7.2 Current variation	3
7.3 Area variation	3
7.4 Agitation (if required)	4
7.5 Alloy layer between coating and substrate	4
7.6 Purity of coatings	4
7.7 Condition of test surface	4
7.8 Density of coating material	4
7.9 Cleanliness of the cell	4
7.10 Cleanliness of electrical connections	4
7.11 Calibration standards (if used)	4
7.12 Non-uniform dissolution	5
8 Procedure	5
8.1 General	5
8.2 Preparation of test surface	5
8.3 Cell application	5
8.4 Electrolysis	5
8.5 Undercoats	5
8.6 Examination after test	6
8.7 Coatings on cylindrical specimens	6
9 Expression of results	6
10 Measurement uncertainty	7
11 Test report	7
Annex A (informative) Typical electrolytes	8
Annex B (informative) Types of instrument	13

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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2177 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Test methods*.

This third edition cancels and replaces the second edition (ISO 2177:1985), which has been technically revised.

iteh Standards
(<https://standards.iteh.ai>)
Document Preview

[ISO 2177:2003](https://standards.iteh.ai/catalog/standards/iso/f2445510-80bf-48c6-aa6a-f90744358163/iso-2177-2003)

<https://standards.iteh.ai/catalog/standards/iso/f2445510-80bf-48c6-aa6a-f90744358163/iso-2177-2003>

Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution

1 Scope

This International Standard describes a coulometric method, by anodic dissolution, for measuring the thickness of metallic coatings. It is only applicable to conductive coatings.

Typical combinations of coatings and substrates that can be tested by this method are given in Table 1. Other combinations may be tested with electrolytes in current use (see Annex A), or new electrolytes may be developed for them but, in both cases, it is necessary to verify the suitability of the complete system.

This International Standard is also applicable to multi-layer systems, e.g. Cu-Ni-Cr (see also 8.5).

It may be used to measure the thickness of coatings applied by various means, if due account is taken of special features such as the presence of an alloy layer. In some cases, the presence and thickness of diffusion layers can also be measured. It can also be used to measure the thickness of coatings on cylindrical specimens and wires (see 8.7).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

<https://standards.iteh.ai/catalog/standards/iso/f2445510-80bf-48c6-aa6a-f90744358163/iso-2177-2003>

ISO 2064:1996, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

ISO 2080, *Surface treatment, metallic and other inorganic coatings — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064 and ISO 2080 as well as the following apply.

3.1

measuring area

area of the significant surface over which a single measurement is made

NOTE The measuring area for this method is the area enclosed by the sealing ring of the cell.

4 Principle

A well-defined area of the coating is subjected to anodic dissolution using a suitable electrolyte. The virtually complete dissolution of the coating is detected by a change in cell voltage. The coating thickness is calculated from the quantity of electricity used, in coulombs, which in turn may be calculated from:

- a) the time interval between the start and the end of the test, if it is conducted at constant current density;
- b) the integrated quantity of electricity used in dissolving the coatings.

5 Instrumentation

5.1 Suitable instruments can be constructed from readily available components but proprietary instruments are usually used (see Annex B).

5.2 Proprietary direct-reading instruments are available for use with electrolytes recommended by the manufacturer. Other instruments record the quantity of electricity, in coulombs, used in dissolving the coating from the measuring area (see 3.1), usually in arbitrary units, from which the thickness is calculated using factors or tables.

With direct reading instruments, the calculation of thickness from current density is made electronically.

5.3 The performance of the instrument shall be checked using specimens of known coating thickness. If the instrument readings agree to within $\pm 5\%$ of the known thicknesses of the specimens, the instrument may be used without further adjustment; otherwise, the cause of the discrepancy shall be removed. However, proprietary instruments shall always be calibrated in accordance with the manufacturer's instructions.

Suitable specimens of known coating thickness shall consist of the same type of coating and substrate as the specimen to be measured, and they shall have an accuracy of 5 % or better. In measuring alloy coatings, the use of correct specimens is of particular importance.

6 Electrolyte

The electrolyte shall have a known, adequate shelf-life and shall be such that:

- a) there is no reaction with the coating metal in the absence of an impressed flow of current;
- b) the coating dissolves anodically at an efficiency as close to 100 % as possible;
- c) a detectable change in electrode potential occurs as the coating is penetrated and an increasing area of substrate is exposed;
- d) the test area exposed in the test cell is completely wetted.

The electrolyte shall be chosen with reference to the coating and substrate materials, the current density and electrolyte circulation within the test cell.

NOTE Typical electrolytes that have been found satisfactory for use with one type of test apparatus for testing various electrodeposited coatings on specific substrates are described in Annex A.

For proprietary instruments, the electrolytes shall normally be chosen on the recommendations of the manufacturer.

Table 1 — Typical combinations of coatings and substrates that can be tested by the coulometric method

Coating	Substrate (underlying material)							
	Al ^a	Cu and Cu alloys	Ni	Ni-Co-Fe alloys	Ag	Steel	Zn	Non-metals
Cd	x	x	x	—	—	x	—	x
Cr	x	x	x	—	—	x	—	x
Cu	x	Only on brass and copper-beryllium	x	—	—	x	x	x
Au	x	x	x	x	x	x	—	—
Pb	x	x	x	x	x	x	—	x
Ni	x	x	—	x	—	x	—	x
Ni, autocatalytic ^b	x	x	x	x	—	x	—	x
Ag	x	x	x	—	—	x	—	x
Sn	x	x	x	—	—	x	—	x
Sn-Ni alloys	—	x	—	—	—	x	—	x
Sn-Pb alloys ^c	x	x	x	x	—	x	—	x
Zn	x	x	x	—	—	x	—	x
^a The detection of the change in the cell voltage may be difficult with some aluminium alloys. ^b The coulometric method can only be used if the phosphorus or boron contents of these coatings are within certain limits. ^c The method is sensitive to alloy composition.								
NOTE See Clause 5 for instrumentation.								

7 Factors affecting the measuring accuracy

7.1 Coating thickness

Generally, the accuracy is lower than optimum for coating thicknesses greater than 50 µm and less than 0,2 µm, unless special equipment is used.

With coatings thicker than 50 µm, there may be an appreciable amount of bevelling or undercutting as the anodic dissolution proceeds. The amount of bevelling is largely dependent on the method employed for stirring the electrolyte. Undercutting may be eliminated, or reduced, by increasing the rate of dissolution, i.e. by increasing the current density used in the test.

7.2 Current variation

For instrumentation using the constant-current and time-measuring technique, current variation causes errors. For instruments using a current-time integrator, too large a change in current may change the anode current efficiency and interfere with the end-point thus causing an error.

7.3 Area variation

The accuracy of the thickness measurement will be no better than the accuracy to which the measuring area is known. Area variations due to gasket wear, gasket pressure, etc., may lead to measurement errors. Much greater accuracy can be obtained if the electrolytic cells are so designed that sealing rings consistently give

well defined measuring areas. In some cases it may be advantageous to measure the area after it has been de-plated and compensate accordingly.

NOTE Error due to variations of the measuring area may, in some instances, be minimized by modifying the instrument calibration using coating thickness calibration standards. Such standards should create similar test conditions to those during actual tests, particularly if testing curved surfaces.

7.4 Agitation (if required)

Inadequate agitation and over-agitation can cause a false end-point.

7.5 Alloy layer between coating and substrate

The measurement of coating thickness by the coulometric method assumes implicitly that a sharply defined interface exists between the coating and the substrate. If an alloy layer exists between the coating and the substrate as, for example, in the case of coatings applied by hot dipping, the coulometric end-point can occur at some point within the alloy layer, thus giving falsely high values for the thickness of the unalloyed coatings. See B.1.2.

NOTE It is possible to use a potential recording chart recorder to detect the potential step where the alloy commences and also when the pure substrate is reached by the dissolution process.

7.6 Purity of coatings

Materials that co-deposit with a coating metal (including alloying metals) can change the effective electrochemical equivalent of the coating metal, the anode current efficiency, and the coating density.

7.7 Condition of test surface

Oil, grease, paint, corrosion products, polishing ingredients, conversion coatings, passivity of nickel coatings, etc., can interfere with the test.

7.8 Density of coating material

Because the coulometric method measures intrinsically mass per unit area, variations in density from the normal density of the coating metal will cause corresponding variations in linear thickness measurements. Normal variations of the composition of an alloy result in small, but significant changes in alloy density and its electrochemical equivalent.

7.9 Cleanliness of the cell

Deposition of metal can take place on the cathode in some electrolytes. This deposit can alter the cell voltages or block the cell aperture. It is, therefore, essential to keep the cathode clean.

7.10 Cleanliness of electrical connections

In the case of instruments other than the constant current type, if the electrical connections are not clean, the current/potential relationship will be disturbed and false end-points obtained.

7.11 Calibration standards (if used)

Measurements made using calibration standards are subject to the additional error of the standards. If the thickness of alloy coatings is being determined, it is usually necessary to use coating standards and to test them using the same procedure.

NOTE The coating of the standards might not be similar to that being tested, e.g. zinc produced by an acid and by an alkali electroplating bath.

7.12 Non-uniform dissolution

If the rate of dissolution is not uniform over the measuring area, a premature end-point can be obtained, and yield falsely low results. Hence, the surface shall be examined after the test to verify that most of the coating has dissolved. On some substrates however, a visible, but insignificant, portion of the coating may remain.

The presence of other material in the coating, the roughness of the coating surface and interface and the presence of porosity in the coating can cause fluctuation of the cell voltage. Such fluctuations can prematurely affect the end-point.

8 Procedure

8.1 General

If commercial equipment is used, follow the manufacturer's instructions with respect to the operating procedure for measurement, the electrolytes and, if necessary, calibration (see 5.3). Appropriate attention shall be given to the factors listed in Clause 7.

NOTE If using instruments that require a pre-set voltage, it should be noted that the actual value is dependent on the particular metallic coating, current density, electrolyte concentration and temperature and circuit resistance, e.g. lead-out connections. For these reasons, it is considered advisable to first perform an evaluation test.

8.2 Preparation of test surface

The test surface (see 7.7) shall be cleaned immediately prior to beginning the test.

NOTE It may be necessary to activate the test surface by mechanical or chemical means, but care should then be taken to avoid removal of metal.

8.3 Cell application

Press the electrolytic cell, fitted with its flexible sealing ring, on to the coating so that a known area is exposed to the test electrolyte. If the cell body is metallic, e.g. stainless steel, it normally forms the cell cathode; if not, insert a suitable cathode (incorporated in some instruments as part of the electrolyte agitation mechanism).

8.4 Electrolysis

The electrolyte in contact with the surface being de-plated shall be continually replenished using the most efficient agitation possible. The agitation shall commence prior to the beginning of the test.

NOTE While simple stirring can be adequate for large areas, it is of little use for areas of 3 mm² or less; hence pulse or pumped agitation should be used. Ultrasonic agitation is also a possibility.

Make the connections, and operate the agitator as appropriate. Continue electrolysis until dissolution of the coating is complete, as indicated by a change in the anode potential or cell voltage, or by the operation of the automatic cut-out.

8.5 Undercoats

When one or more undercoats are to be measured, after measuring the top coating ensure that the top coat has first been completely removed from the whole of the measuring area. Remove the electrolyte from the cell using a suitable suction device, and thoroughly rinse out the cell with distilled or de-mineralized water.

Do not disturb the cell at any time during these operations. Abandon the test if the slightest displacement has occurred.

Reset the instrument controls for the next coating, introduce the appropriate electrolyte and continue the test as before.

8.6 Examination after test

After completion of the test(s), remove the electrolyte from the cell, rinse out with water, lift off the cell and examine the specimen to ensure that the removal of the coating is complete over the area enclosed by the seal (see 7.12).

8.7 Coatings on cylindrical specimens

If the surface area is too small to use the normal cell with a flexible gasket, replace the cell by an electrolyte container and an appropriate holding device, using an agitator if necessary. This device shall be adjustable and shall be pre-set to allow a known length of specimen to be immersed. For direct reading instruments, especially those with alternative cell sizes, calculate the length of the specimen to be immersed so that the same known surface area is presented to the cathode as for a test cell.

NOTE 1 In the majority of applications, the same electrolyte can be used, but, in order to obtain the optimum instrument sensitivity and accuracy, the operating conditions might have to be modified, e.g., the cut-off voltage and deplating current.

NOTE 2 An exact de-plating area is necessary for accuracy and the main source of error is due to the meniscus and current field at the electrolyte surface. In the case of large diameter cylinders, e.g., wires, the immersed cylinder end should be shielded from the current and electrolyte. The area of any exposed end should not exceed 2 % of the whole area.

9 Expression of results

The coating thickness, d , in micrometres, is given by the equation:

$$d = 100k \times \frac{QE}{A\rho} \tag{1}$$

where

- k is the current efficiency of the dissolution process (equal to 100 in the case of 100 % efficiency);
- Q is the quantity of electricity, in coulombs, passed in dissolving the coating; if an integrating meter is not used, calculate Q from equation (2);
- E is the electrochemical equivalent, in grams per coulomb, of the coating metal for the conditions of the test;
- A is the area, in square centimetres, from which the coating is dissolved, i.e. the measuring area;
- ρ is the density, in grams per cubic centimetre, of the coating.

$$Q = It \tag{2}$$

where

- I is the current, in amperes;
- t is the test duration, in seconds.