

Aluminium and aluminium alloys — Anodizing —

Part 2: Determination of mass per unit area (surface density) of anodic oxidation coatings — Gravimetric method

The European Standard EN 12373-2:1998 has the status of a
British Standard

ICS 25.220.10; 77.120.10

National foreword

This British Standard is the English language version of EN 12373-2:1998. It supersedes BS 6161-1:1984, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee STI/32, Anodic oxidation coatings on aluminium, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 5 and a back cover.

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English version

Aluminium and aluminium alloys — Anodizing — Part 2: Determination of mass per unit area (surface density) of anodic oxidation coatings — Gravimetric method

Aluminium et alliages d'aluminium —
Anodisation — Partie 2: Détermination de la masse
par unité de surface (masse surfacique) des
couches anodiques — Méthode gravimétrique

Aluminium und Aluminiumlegierungen —
Anodisieren — Teil 2: Bestimmung der Masse je
Flächeneinheit (flächenbezogene Masse) von
anodisch erzeugten Oxidschichten —
Gravimetrisches Verfahren

This European Standard was approved by CEN on 5 November 1998.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 132, Aluminium and aluminium alloys, the Secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 1999, and conflicting national standards shall be withdrawn at the latest by May 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

It is based upon ISO 2106:1982.

In this standard, annex A is informative.

EN 12373, *Aluminium and aluminium alloys — Anodizing*, comprises the following parts:

- *Part 1: Method for specifying decorative and protective anodic oxidation coatings on aluminium;*
- *Part 2: Determination of mass per unit area (surface density) of anodic oxidation coatings — Gravimetric method;*
- *Part 3: Determination of thickness of anodic oxidation coatings — Non-destructive measurement by split-beam microscope;*
- *Part 4: Estimation of loss of absorptive power of anodic oxidation coatings after sealing by dye spot test with prior acid treatment;*
- *Part 5: Assessment of quality of sealed anodic oxidation coatings by measurement of admittance;*
- *Part 6: Assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution without prior acid treatment;*
- *Part 7: Assessment of quality of sealed anodic oxidation coatings by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution with prior acid treatment;*
- *Part 8: Determination of the comparative fastness to ultra-violet light and heat of coloured anodic oxidation coatings;*

- *Part 9: Measurement of wear resistance and wear index of anodic oxidation coatings using an abrasive wheel wear test apparatus;*
- *Part 10: Measurement of mean specific abrasion resistance of anodic oxidation coatings using an abrasive jet test apparatus;*
- *Part 11: Measurement of specular reflectance and specular gloss of anodic oxidation coatings at angles of 20°, 45°, 60° or 85°;*
- *Part 12: Measurement of reflectance characteristics of aluminium surfaces using integrating-sphere instruments;*
- *Part 13: Measurement of reflectivity characteristics of aluminium surfaces using a goniophotometer or an abridged goniophotometer;*
- *Part 14: Visual determination of image clarity of anodic oxidation coatings — Chart scale method;*
- *Part 15: Assessment of resistance of anodic oxidation coatings to cracking by deformation;*
- *Part 16: Check for continuity of thin anodic oxidation coatings — Copper sulfate test;*
- *Part 17: Determination of electric breakdown potential;*
- *Part 18: Rating system for the evaluation of pitting corrosion — Chart method;*
- *Part 19: Rating system for the evaluation of pitting corrosion — Grid method.*

Contents

	Page
Foreword	2
1 Scope	3
2 Principle	3
3 Reagents	3
4 Apparatus	3
5 Procedure	3
6 Expression of results	3
7 Test report	4
Annex A (informative) Recommended method for the drying of samples	5

1 Scope

This part of this European Standard specifies a gravimetric method for determining the mass per unit area (surface density) of anodic oxidation coatings on aluminium and its alloys.

The method is applicable to all oxide coatings formed by anodizing aluminium and its alloys, either cast or wrought, and is suitable for most aluminium alloys except those in which the copper content is greater than 6 %.

NOTE 1 A high content of copper in the alloy can lead to increased dissolution of the basis aluminium.

NOTE 2 If the thickness is known with sufficient precision (from, for example, the method specified in EN 12373-3¹⁾), determination of the mass per unit area (surface density) of the coating will enable its apparent density to be calculated. Conversely, if the conditions of application of the coating, and its density, are known, the determination of its mass per unit area (surface density) may permit calculation of the average mass and an approximate evaluation of the thickness (see note to clause 6).

2 Principle

The anodic oxidation coating on a weighed test piece of known surface area is dissolved, without significantly attacking the basis metal, using a mixture of phosphoric acid and chromium(VI) oxide of specified concentration.

After dissolution of the coating, the test piece is reweighed, and the loss in mass calculated. The mass loss is related to the unit area covered by the coating and is expressed in milligrams of coating per square decimetre of surface.

NOTE The test is destructive.

3 Reagents

Use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

Phosphoric acid/chromic acid solution, prepared as follows:

- phosphoric acid, ρ_{20} 1,7 g/ml: 35 ml;
- chromium(VI) oxide: 20 g;
- distilled water: to make up to 1 l.

WARNING NOTE Chromium(VI) solutions are toxic and should be handled with care.

4 Apparatus

Usual laboratory apparatus and glassware, together with the following.

Laboratory balance, capable of weighing to an accuracy of 0,1 mg.

5 Procedure

5.1 Preparation of test piece

The surface of the test piece to be tested shall have an area of between 0,25 dm² and 1 dm², and the mass of the test piece shall not exceed 100 g. If the surface is dirty or impregnated with oil, grease or similar material, this shall be removed with the aid of a suitable organic solvent (see annex A).

To measure the mass of the coating on one surface only of the test piece, the coating on the other surface shall be removed by a mechanical or chemical process, leaving the significant surface intact. Alternatively, a protective agent, resistant to attack by the acidic test solution, shall be applied on the reverse surface of the test piece.

5.2 Performance of the test

Calculate the area of the surface covered by an anodic oxidation coating, weigh the test piece to the nearest 0,1 mg and immerse it in the phosphoric acid/chromic solution (see clause 3) for 10 min at 95 °C to 100 °C with efficient stirring. Rinse the test piece in distilled water, dry and reweigh (see annex A). Repeat the immersion, drying and weighing until no further loss in mass is observed.

NOTE The freshly made reagent will normally allow complete dissolution of the coating within 10 min. Its dissolving power diminishes with use; in general, 1 l of solution is capable of dissolving 12 g of coating before the diminution becomes noticeable.

6 Expression of results

Calculate the mass per unit area of surface (surface density) of the coating, ρ_A , in milligrams per square decimetre, using the equation:

$$\rho_A = \frac{m_1 - m_2}{A} \quad (1)$$

where

- m_1 is the mass, in milligrams, of the test piece before dissolution of the coating;
- m_2 is the mass, in milligrams, of the test piece after dissolution of the coating;
- A is the area, in square decimetres, effectively covered by the coating of which the mass is measured (without taking into account edges or other uncoated parts).

NOTE Where required, the average thickness of the coating, δ , in micrometres, can be estimated using the equation:

$$\delta = \frac{\rho_A}{\rho \times 10} \quad (2)$$

where

- ρ_A is the mass per unit area (surface density), in milligrams per square decimetre, of the coating;
- ρ is the density, in grams per cubic centimetre, of the coating.

¹⁾ See foreword.

The density of the coating depends on the specific alloy, the anodizing process and the sealing process. This density can vary, in normal processing conditions, between $2,3 \text{ g/cm}^3$ and 3 g/cm^3 .

For thin oxide coatings on aluminium and on alloys without copper, produced under direct current in sulfuric acid solution, at a temperature of approximately $20 \text{ }^\circ\text{C}$, the density can be assumed to be equal to $2,6 \text{ g/cm}^3$ for sealed coatings and $2,4 \text{ g/cm}^3$ for unsealed coatings.

The method gives only an approximate value of the thickness because of uncertainty about the density value.

The estimation of thickness is most accurate for thin coatings ($10 \text{ }\mu\text{m}$ and less).

7 Test report

The test report shall include at least the following information:

- a) the type and identification of the product tested;
- b) a reference to this European Standard;
- c) the result of the test (see clause 6);
- d) anything unusual noticed during the determination;
- e) any operations not included in the procedure described in this European Standard, or considered to be optional;
- f) the date of the test.

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Annex A (informative)

Recommended method for the drying of samples

A.1 Degrease the test piece by gentle agitation for 30 s in a suitable organic solvent at room temperature; remove; leave for 5 min in the ambient atmosphere (pre-drying); place in a drying oven preheated to 60 °C and leave for 15 min with the coated surfaces standing upright.

WARNING NOTE Where organic solvents are used, carry out the degreasing operation and the pre-drying in a well-ventilated area to minimize exposure to solvent vapour.

A.2 Allow the test piece to cool for 30 min over silica gel in a closed desiccator.

A.3 After the acid treatment and rinsing (see 5.2), repeat operations **A.1** and **A.2**, omitting the treatment in the organic solvent.

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