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British Standard Methods of test for
**Anodic oxidation coatings on
aluminium and its alloys**

**Part 3. Assessment of sealing quality by measurement of the
loss of mass after immersion in phosphoric-chromic
acid solution**

[ISO title: Anodizing of aluminium and its alloys — Assessment of quality of sealed anodic coatings by measurement of the loss of mass after immersion in phosphoric-chromic acid solution]

Méthodes d'essai des couches anodiques sur aluminium et alliages d'aluminium
Partie 3. Evaluation de la qualité du colmatage par mesurage de la perte de masse
après immersion en solution phospho-chromique

Verfahren zur Prüfung von anodisch erzeugten Oxidschichten auf Aluminium und Aluminiumlegierungen
Teil 3. Beurteilung der Güte der Verdichtung durch Messung des Massenverlustes nach Tauchbad
in einer Phosphor-Chromsäure-Lösung

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National foreword

This revision of this Part of BS 6161 has been prepared under the direction of the Surface Coatings (other than Paints) Standards Committee and supersedes the 1981 edition which is withdrawn. It is identical with ISO 3210-1983 'Anodizing of aluminium and its alloys — Assessment of quality of sealed anodic coatings by measurement of the loss of mass after immersion in phosphoric-chromic acid solution', published by the International Standards Organization (ISO). ISO 3210 was prepared by Subcommittee 2 'Anodized aluminium' of Technical Committee 79 'Light metals and their alloys' with the active participation and approval of the UK.

This revision differs from the 1981 edition as follows.

- (a) A correction has been made in respect of an incorrectly specified reagent.
- (b) The procedure has been more rigorously described and provision made for more accurate weighing of the test specimen.
- (c) A procedure has been included for drying the test specimen before and after acid treatment.

The individual Parts of this standard are intended to replace progressively the appendices of BS 1615 : 1972 'Anodic oxidation coatings on aluminium' and, when all the test methods required for the purposes of that standard have been issued as Parts of BS 6161, BS 1615 will be revised accordingly.

Further Parts of this standard are in preparation; at present the following Parts have been published:

- Part 1 Determination of mass per unit area of anodic oxide coatings (gravimetric method)
- Part 2 Determination of thickness of anodic oxide coatings: non-destructive measurement by split-beam microscope
- Part 3 Assessment of sealing quality by measurement of the loss of mass after immersion in phosphoric-chromic acid solution
- Part 4 Assessment of sealing quality by measurement of the loss of mass after immersion in acid solution
- Part 5 Estimation of loss of absorptive power of sealed coatings: dye spot test with prior acid treatment
- Part 6 Assessment of sealing quality by measurement of admittance or impedance
- Part 7 Accelerated test of light fastness of coloured anodic oxidation coatings using artificial light
- Part 8 Determination of the fastness to ultraviolet light of coloured anodic oxide coatings.

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'Part of BS 6161'.

Cross-reference

International standard	Corresponding British Standard
ISO 2143-1981	BS 6161 Methods of test for anodic oxidation coatings on aluminium and its alloys Part 5 : 1982 Estimation of loss of absorptive power of sealed coatings: dye spot test with prior acid treatment (Identical)

Additional information. With reference to clause 5, water complying with BS 3978 'Water for laboratory use', is suitable.

WARNING. Chromium (VI) oxide, specified in 5.2, causes severe burns and, in contact with combustible material, may cause fire. Avoid inhaling dust. Prevent contact with the eyes and skin.

NOTE. In 8.5, line 4, delete 'immediatly' and substitute 'immediately'.

This Part of BS 6161 describes a method of test only and should not be used or quoted as a specification defining sealing quality. Reference to this Part should state that the method of test used is in accordance with BS 6161 : Part 3.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

British Standard Methods of test for

Anodic oxidation coatings on aluminium and its alloys

Part 3. Assessment of sealing quality by measurement of the loss of mass after immersion in phosphoric-chromic acid solution

1 Scope

This International Standard specifies a method for assessing the quality of sealed anodic coatings on aluminium and its alloys by measurement of the loss of mass after immersion in phosphoric-chromic acid solution.

2 Field of application

This International Standard specifies a referee method to be used to assess the quality of sealed coatings.

The method is applicable to anodic coatings intended for exposure to the weather, or for protective purposes in corrosive media, and where resistance to staining is important.

The method is not applicable to

- hard-type anodic coatings which normally are not sealed;
- anodic coatings that have been sealed only in dichromate solutions;
- anodic coatings that have undergone a treatment to render them hydrophobic.

The method is destructive and may serve as a referee method in case of doubt or dispute regarding the results of the test of loss of absorptive power (see ISO 2143).

3 Reference

ISO 2143, *Anodizing of aluminium and its alloys — Estimation of loss of absorptive power of anodic oxide coatings after sealing — Dye spot test with prior acid treatment.*

4 Principle

The test is based upon the observation that an unsealed coating of aluminium oxide is rapidly dissolved in acid media, whereas a well-sealed coating of aluminium oxide withstands long immersion without appreciable attack.

5 Reagents

The reagents used shall be of recognized analytical grade. The water used shall be distilled or deionized water.

5.1 Phosphoric acid ($\rho_{20} = 1,7$ g/ml).

5.2 Chromium(VI) oxide.

6 Apparatus

Ordinary laboratory apparatus and

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

7 Preparation of test piece

From the material to be tested, cut a test piece such that there is an area of about 1 dm² (minimum 0,5 dm²) of significant surface area. Normally, its mass should not exceed 200 g.

On hollow extrusions, take the sample from the end of the sections where the total surface area has an anodic coating due to the throwing power of the anodizing electrolyte.

NOTE — In special cases (certain types of jiggling, small hollow sections, etc.), it will be necessary to remove the anodic oxide coating from the inside surface and to carry out the test on the coating on the outer surface of the extrusion.

8 Procedure

8.1 Measure the total coated area of the test piece (excluding cut edges and other uncoated surfaces).

NOTE — The test solution does not attack bare metal; hence, it is unnecessary to take bare surfaces into account.

Remove any surface bloom from the test piece by rubbing with a dry cloth.

8.2 Degrease the test piece in a suitable organic solvent at room temperature according to the method described in clauses A.1 and A.2 of the annex.

WARNING NOTE — This operation should be carried out in a well-ventilated hood to prevent inhalation of the solvent vapour.

8.3 Then dry the test piece thoroughly and immediately weigh it to the nearest 0,1 mg (mass m_1).

8.4 Completely immerse the test piece standing upright in a aqueous solution containing per litre,

35 ml of phosphoric acid (5.1);

20 g of crystallized chromium(VI) oxide (5.2).

Leave there for exactly 15 min at a constant temperature of 38 ± 1 °C.

NOTE — Uniformity of the temperature within the solution is very important. This may be achieved by using a water-bath or by continuously stirring.

The test solution may be re-used but shall be discarded after 10 dm² of anodized surface have been treated per litre of solution. Do not use test solution which has been in contact with materials other than anodized aluminium and its alloys.

8.5 Take the test piece from the solution and rinse thoroughly first under running water and then in deionized or distilled water. Then dry it as indicated in the annex and immediately weigh it to the nearest 0,1 mg (mass m_2).

8.6 During all operations 8.2 to 8.5, avoid touching the test piece with bare hands.

Take extreme care that the two drying operations in 8.3 and 8.5 are always carried out in the same reproducible way. Avoid heating to temperatures above 60 °C.

9 Expression of results

The loss in mass per surface area, ρ_A , expressed in milligrams per square decimetre, is given by the equation

$$\rho_A = \frac{m_1 - m_2}{A}$$

where

m_1 is the mass, in milligrams, of the test piece before immersion;

m_2 is the mass, in milligrams, of the test piece after immersion;

A is the surface area, in square decimetres, in contact with the solution, excluding the edges.

The maximum permissible loss in mass for the coating under test shall be subject to agreement between the interested parties.

10 Test report

The test report shall contain at least the following information :

- a) the type and identification of the product tested;
- b) the reference to this International Standard;
- c) the result of the test;
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the date of the test.

Annex

Suggested method for drying of samples before and after acid treatment

A.1 After degreasing the test piece by gentle agitation for 30 s in a suitable organic solvent at room temperature, leave for 5 min in the surrounding atmosphere (pre-drying) and then place in a drying oven preheated to 60 °C and leave for exactly 15 min with the anodized surfaces standing upright.

WARNING NOTE — In the case where chlorinated solvents are used, carry out the degreasing operation in perchloroethylene and the pre-drying in a well-ventilated hood to prevent inhalation of the solvent vapour.

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

A.3 After the acid treatment and thorough rinsing, repeat the operations A.1 and A.2 in exactly the same way but omitting the use of organic solvent.

Publications referred to

See national foreword.

BS 6161 : Part 3 : 1984 ISO 3210-1983

This British Standard, having been prepared under the direction of the Surface Coatings (other than Paints) Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 December 1984.

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First published October 1981

First revision December 1984

ISBN 0 580 14041 5

British Standards Institution

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The following BSI references relate to the work on this standard: Committee reference SRC/4 Draft for comment 82/54131 DC

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Surface Coatings (other than Paints) Standards Committee (SRC/-) to Technical Committee SRC/4 upon which the following bodies were represented:

Aluminium Federation
Aluminium Window Association
Association of Builders' Hardware Manufacturers
British Anodizing Association
British Lock Manufacturers' Association

British Metal Finishing Suppliers' Association
Institute of Metal Finishing
Institution of Chemical Engineers
Institution of Corrosion Science and Technology
Metal Finishing Association
Metal Window Federation Limited
Ministry of Defence
Royal Institution of Chartered Surveyors

Amendments issued since publication

Amd. No.	Date of issue	Text affected

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