

Specification for

Chromic acid anodizing of aluminium and wrought aluminium alloys

This European Standard EN 2101:1991 has the status of a
British Standard

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National foreword

This British Standard has been prepared under the direction of the Aerospace Standards Policy Committee and is the English language version of EN 2101:1991 "*Aerospace series — Chromic acid anodizing of aluminium and wrought aluminium alloys*", published by the European Committee for Standardization (CEN).

EN 2101 was produced as a result of international discussion in which the UK took an active part.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

EUROPEAN STANDARD

EN 2101

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English version

Aerospace series
Chromic acid anodizing of aluminium and wrought
aluminium alloys

Série aérospatiale
Anodisation chromique de l'aluminium et des
alliages d'aluminium corroyés

Luft- und Raumfahrt Chromsäure-Anodisieren
von Aluminium und
Aluminium-Knetlegierungen

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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

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Ref. No. EN 2101:1991 E

Foreword

This European Standard has been prepared by the European Association of Aerospace Manufacturers (AECMA).

After inquiries and votes carried out in accordance with the rules of this Association, this Standard has successively received the approval of the National Association and the Official Services of the member countries of AECMA, prior to its presentation to CEN.

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1 Scope and field of application

This standard specifies the required characteristics for the performance of chromic acid anodizing with or without sealing as well as quality assurance of the coating obtained.

It applies to parts in aluminium and wrought aluminium alloys of category 1 and 2 (see clause 4) used in aerospace construction when reference is made to this standard.

2 Purpose of anodizing

2.1 Type A: Unsealed anodizing

It is used either as a surface preparation before paint application or as a preparation to a macrographic examination (structural condition, presence of metallurgical defects).

2.2 Type B: Sealed anodizing

It is intended for corrosion protection. It shall be with or without colouring and used with or without additional painting.

3 References

ISO 1463:1982, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method.*

ISO 2085:1976, *Anodizing of aluminium and its alloys — Check of continuity of thin anodic oxide coatings — Copper sulphate test.*

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

ISO 2143:1981, *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.*

ISO 2360:1982, *Non-conductive coatings on non-magnetic basis metals — Measurement of coating thickness — Eddy current method.*

ISO 2376:1972, *Anodization (anodic oxidation) of aluminium and its alloys — Insulation check by measurement of breakdown potential.*

ISO 3768:1976, *Metallic coatings — Neutral salt spray test (NSS test).*

EN 2334, *Aerospace series — Acid chromate pickle for aluminium alloys¹⁾.*

4 Material categories

4.1 Category 1

Pure aluminium, clad alloys, alloys characterised by the absence of copper or with a copper content limited to 1 %.

4.2 Category 2

Non-clad alloys containing > 1 % copper:

- Category 2 A: solution heat treated, quenched and naturally aged condition
- Category 2 B: solution heat treated, quenched plus artificially aged condition.

5 Supporting jig

The supporting jig (e.g. in aluminium alloy or titanium) shall provide effective electrical contact with the parts. This contact is preferably achieved at several points to ensure better current distribution.

6 Processing sequence

6.1 Cleaning (see Annex A)

The cleaning method used shall be appropriate for the contamination experienced on the materials treated. Solvent degreasing followed by cleaning in an alkaline bath is generally the most effective method.

¹⁾ In preparation at the date of publication of this standard.

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6.2 Pickling (see Annex A)

The pickling operation shall remove natural oxides in order to obtain correct anodizing; it shall neither degrade the metallurgical properties of the material nor the material fatigue behaviour, nor initiate pitting.

It shall not degrade the dimensional tolerances and surface roughness specified.

As a general rule, pickling is conducted in a sulphuric-chromic acid bath, but in exceptional cases, for certain alloys of category 2, alkaline pickling may be used.

6.3 Rinsing

All rinsing operations before anodizing shall be effective and complete. For example, they may be performed by immersion followed by running water spray. It is recommended that rinsing in ordinary water is followed by rinsing in deionized or distilled water.

6.4 Anodizing

6.4.1 Electrolyte

Aqueous solution of chromic acid (99,5 % min. CrO_3) at a concentration of 30 g/l to 150 g/l to which certain additional agents such as oxalic acid may be added.

The impurity content shall be less than:

- Chlorides: 200 mg/l expressed as NaCl,
- Sulphates: 500 mg/l expressed as H_2SO_4 .

The chromic acid anodizing bath shall be made up with deionized water of a resistivity of more than $10^5 \Omega\cdot\text{cm}$ unless sufficiently pure ordinary water is available.

6.4.2 Treatment conditions

The anodizing parameters (temperature, voltage, time) shall be adapted to the material and its category in accordance with the bath composition.

The control system shall make it possible to maintain the treatment temperature within a tolerance of $\pm 2^\circ\text{C}$ in a suitably agitated bath.

NOTE The treatment of materials of different categories in one anodizing batch shall be avoided.

6.5 Unsealed anodizing

In this case, it is necessary to take great precautions to avoid contamination of the oxide coating during handling.

If a paint finish is required, it shall be applied as soon as possible and 16 h max. after anodizing.

6.6 Sealed anodizing

The sealing quality stipulated in this standard requires the use of water having a resistivity greater than $10^5 \Omega\cdot\text{cm}$ when made up.

The pH shall be between 5,5 and 6,9.

The temperature of the sealing bath shall not be less than 97°C .

For alloys of category 2, sealing shall preferably be applied with the addition in deionized water of not less than 30 mg/l potassium dichromate, with a resistivity greater than $10^5 \Omega\cdot\text{cm}$.

6.7 Removal of the anodic coating (see Annex A)

The method used to remove the anodic coating shall be such that, when reanodized, the dimensions and surface roughness shall conform with the values specified.

7 Quality assurance

The required quality is achieved by carrying out at the same time tests on the bath efficiency and inspection of treated parts as follows:

7.1 Check for bath efficiency

The following tests shall be conducted for qualification of a new installation and for continuous quality monitoring:

7.1.1 Chemical analysis of the bath on make up and during operation (see clause 6.4.1).

7.1.2 Measurement of resistivity and pH of the sealing bath (see clause 6.6).

7.1.3 Corrosion tests

These tests (see Table) are applicable for anodized and sealed parts only.

Not less than 5 rectangular specimens of the minimum dimensions of 60 mm × 120 mm, thickness optional, representative of the treated parts (material, heat treatment, surface finish) shall be anodized and sealed. If it is impossible to produce rectangular specimens, the surface exposed to corrosion for each specimen shall be practically equivalent.

7.1.4 The frequency of inspection of bath efficiency is laid down by the official services or at the discretion of the quality assurance authority.

7.2 Inspection of treated parts

Where it is not practicable to perform the quality control on processed parts, test pieces which are representative of a part or of a batch of parts which have been processed the same way and at the same time shall be used.

7.2.1 Batching

A batch consists of all parts treated at the same time in the same bath.

7.2.2 Inspection (see Table)

The quality assurance authority specifies the number of parts to be inspected per batch or parts to be subjected to individual inspection.

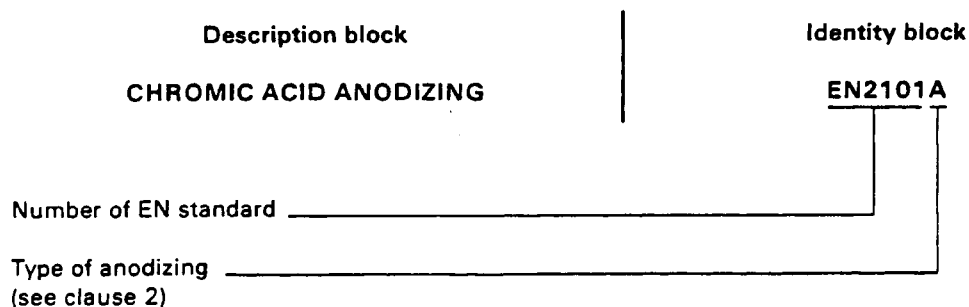
7.3 Test report

The test report shall state the following:

- Reference to this standard
- Type of anodizing (type A or B)
- Category of the material and its heat treatment
- Date of anodizing
- Batch number
- Test results.

8 Designation

The following designation shall be indicated on the relevant documents:



NOTE If necessary, the originator code 19005 may be introduced between the description block and the identity block.

Table

| Nature of test | Test method | Anodizing | | | | | |
|-------------------------------------|---|--|-----------------------------|--------------------------|-------------------|-----|-------------------|
| | | Sealed | | | Unsealed | | |
| | | Material category | | Material category | Material category | | Material category |
| Appearance | Visual | 1 | 2A | 2B | 1 | 2A | 2B |
| Minimum thickness (µm) ^a | ISO 1463 ^c ISO 2360 ISO 2106 | 2,5 | 1,5 | 1 | 2,5 | 1,5 | 1 |
| Loss of absorptive power | ISO 2143 | No persistence of colourant stain (intensity if stain: 0 or 1) | — | — | — | — | No test |
| Corrosion resistance ^b | Annex B ISO 3768 ^c or Annex C | — | No indelible residual stain | No corrosion after 500 h | 300 h | — | — |
| Coating continuity | ISO 2085 ^d | No black point | — | — | — | — | No test |
| Average insulation (V) ^a | ISO 2376 | 300 | 200 | 150 | 200 | 150 | 100 |

^a Unsatisfactory results of thickness and insulation measurements are not a cause for rejection.

— For sealed anodizing:

If the results of corrosion testing are satisfactory, or if there are no particular electrical insulation requirements.

— For unsealed anodizing:

Acceptance or rejection is left to the discretion of the Official Services or of competent Authority who shall consider the results depending upon anodizing process conditions and the intended use of the anodized part.

^b Except at points of electrical contact during anodizing or at points of marking.

^c Reference method.

^d Without thickness restriction.

Annex A Surface preparation recommendations

A.1 Scope

This annex specifies the cleaning and pickling processes to be used as preliminary treatment. Any other process giving at least equivalent results is acceptable.

A.2 Degreasing

A.2.1 Organic solvents

- a) Hot solvents non-water washable

Halogenated hydrocarbons in the liquid or vapour phase such as trichlorethylene, perchlorethylene, trichlorotrifluoroethane and 1-1-1 trichloroethane.

- b) Cold solvents non-water washable

Solvents in the liquid phase such as those listed under a) and white spirit. It can be applied by immersion, spraying or manually. When the parts are degreased by immersion in the solvents, at least 3 successive immersions shall be applied.

When the solvent is contaminated by grease, it shall be replaced.

A.2.2 Alkaline

Used alone or in addition to preliminary solvent degreasing. The degreasing baths used shall be inhibited to avoid attack of metal surfaces. They may be used hot or cold with or without electrical current.

A.3 Cleaning by abrasive blasting

A.3.1 Rough cleaning

Blasting abrasives of a grit size of more than 100 μm with or without water.

This cleaning is not applicable to thin parts or when the required surface roughness does not allow it.

A.3.2 Fine cleaning

Blasting of abrasives of a grit size of less than or equal to 100 μm with or without water.

A.4 Pickling

A.4.1 Acid

A.4.1.1 Sulphuric-chromic acid

Parts shall be processed in accordance with EN 2334.

A.4.1.2 Other baths

Baths containing e.g. fluorides may be used.

A.4.2 Alkaline

Bath composition:

- Sodium hydroxide : 26 g/l to 50 g/l
- Sodium heptonate or gluconate : 0,75 g/l to 1,0 g/l

Conditions of use:

- Temperature : 40 °C to 65 °C.
- Rate of attack : 0,6 $\mu\text{m}/\text{min}$ to 1,5 $\mu\text{m}/\text{min}$.

The reaction is very vigorous and an immersion time of 15 s to 1 min is generally sufficient.

This bath is not applicable to close tolerance parts, nor to assemblies joined by riveting or resistance welding or if a surface polish is required.

A.4.3 De-smutting

The deposit formed after alkaline pickling and certain acid pickling operations is eliminated by immersion in a aqueous solution of 30 % to 50 % by volume of nitric acid (ρ 20 °C = 1,4 g/cm³) at room temperature.

A.5 Stripping of anodic coatings

The following aqueous solution may be used at a temperature of 97 °C to 100 °C:

- Phosphoric acid (85 % by mass) : 25 ml/l to 35 ml/l
- Chromic acid : 20 g/l to 25 g/l

Annex B Inspection of the absorbing ability by anthraquinone violet drop test

B.1 Scope

This annex describes the method of inspection of the absorbing ability of the oxide films obtained by anodizing by anthraquinone violet drop test.

B.2 Field of application

This method is applicable to all sealed oxide coatings produced on wrought aluminium and aluminium alloys. As the estimation is based on the persistence of a colouration, the method cannot apply to dyed coatings of dark shades such as: deep black, deep blue, deep purple.

B.3 Equipment

No special equipment.

B.4 Operation method

A given surface of the anodized specimen is carefully degreased with a suitable solvent (e.g. benzol, trichloroethylene) and if necessary with a light abrasive (e.g. magnesia paste diluted in water) so as to obtain complete cleaning of the surface.

A few drops of a 1 % anthraquinone violet solution are deposited on an area of about 1 cm² of the surface prepared in this way.

It is left to act for 5 min.

The surface is cleaned by rubbing, under running water, using a cotton swab or light abrasive (magnesia, pearl white) for 2 min, and then in a neutral soap solution. It is thoroughly rinsed and dried.

B.5 Interpretation of results

If no residual indelible stain on the tested area persists, the coating has lost its absorptive power and this indicates a correctly sealed coating.

Annex C Inspection of corrosion resistance by repeated immersion test in salt solution

C.1 Scope and field of application

This annex specifies the method of inspection of corrosion resistance of the sealed oxide films obtained by anodizing by repeated immersion tests in salt solution.

C.2 Salt solution (Reagent)

This consists of a 30 g/l sodium chloride solution in distilled or deionized water to which a buffer mixture of:

- Chemically pure disodium phosphate : 0,19 g
- Boric acid : 1,25 g

is added.

To produce a pH value of approximately 8, add a quantity of saturated sodium carbonate solution in distilled water.

The necessary quantity of this solution can be determined either with the pH-meter or on an aliquot portion poured into a test tube rinsed several times with the reagent, to which 0,5 ml phenolphthalein is added per 10 ml reagent; it is obtained when the solution takes on a pink colouration.

C.3 Equipment

The equipment consists of an oven maintained throughout at a temperature of between 33 °C and 37 °C with a humidity of 80 % to 95 % containing the following:

- An appropriate set-up by which the complete operations of alternating immersion and removal can be obtained automatically and continuously for each part or specimen attached to this set-up by clamps made from insulating material.

— One or several glass or plastic containers holding the reagent.

C.4 Operating method

C.4.1 Preparation of the surfaces to be inspected

The specimens defined in clause 7.1.3 of this standard or the parts are carefully degreased with a volatile solvent vapour according to the conditions of Annex A, clause A.2.1, a).

C.4.2 Test method

There shall be only one part or specimen per container. Each part or specimen shall be completely immersed in the reagent (see C.2) with at least 10 mm of reagent in contact with the test surfaces.

The volume of the reagent shall be at least of 4 ml per cm² of the test surface.

A complete cycle takes 4 h : 2 h immersion and 2 h removal.

The reagent level in the containers is kept constant by additions of distilled or deionized water.

The reagent is renewed after careful cleaning of the containers every 2 days during the first week and every 10 days subsequently.

C.5 Results

After the specified number of hours of tests, the parts or specimens shall meet the requirements specified in the Table.

National appendix NA (informative)

The United Kingdom participation in the preparation of this European Standard was entrusted by the Aerospace Standards Policy Committee (ACE/-) to Technical Committee ACE/44 upon which the following bodies were represented:

Metal Finishing Association
 Ministry of Defence
 Oil and Colour Chemists' Association
 Paintmakers' Association of Great Britain
 Society of British Aerospace Companies Ltd.

National appendix NB (informative)

The British Standards corresponding to the international standards referred to in the text are as follows:

| International standard | Corresponding British Standard |
|------------------------|---|
| ISO 1463:1982 | BS 5411 <i>Methods of test for metallic and related coatings</i> Part 5:1984 <i>Measurement of local thickness of metal and oxide coatings by the microscopical examination of cross-sections</i> (Identical) |
| ISO 2106:1982 | BS 6161 <i>Methods of test for anodic oxidation coatings on aluminium and its alloys</i> Part 1:1984 <i>Determination of mass per unit area (surface density) of anodic oxidation coatings (gravimetric method)</i> (Identical) |
| ISO 2143:1981 | Part 5:1982 <i>Estimation of loss of absorptive power of sealed coatings: dye spot test with prior acid treatment</i> (Identical) |
| ISO 2360:1982 | BS 5411 <i>Methods of test for metallic and related coatings</i> Part 3:1984 <i>Eddy current method for measurement of coating thickness of non-conductive coatings on non-magnetic basis metals</i> (Identical) |
| ISO 3768:1976 | BS 5466 <i>Methods for corrosion testing of metallic coatings</i> Part 1:1977 <i>Neutral salt spray test (NSS test)</i> (Identical) |

There is no British Standard corresponding to EN 2334 referred to in the text. A British Standard identical in title and number will be published in due course.

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