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Aerospace series — Hexavalent chromium free anodizing of aluminium and aluminium alloys



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National foreword

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Aerospace series - Hexavalent chromium free anodizing of aluminium and aluminium alloys

Série aérospatiale - Anodisation sans chrome hexavalent de l'aluminium et des alliages d'aluminium Luft- und Raumfahrt - Hexavalentes chromfreies Anodisieren von Aluminium und Aluminiumlegierungen

This European Standard was approved by CEN on 24 September 2016.

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European foreword

This document (EN 4827:2017) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

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

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of an identical text or by endorsement, at the latest by August 2017, and conflicting national standards shall be withdrawn at the latest by August 2017.

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1 Scope

This European Standard defines the requirements for hexavalent chromium free anodizing of aluminium and aluminium alloys for corrosion protection, bonding and painting.

Hard anodizing is not covered by this European Standard.

The purpose of this European Standard is to give design, quality and manufacturing requirements. It does not give complete in-house process instructions; these shall be given in the manufacturers detailed process instructions.

2 Normative references

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

EN 2284, Aerospace series — Sulphuric acid anodizing of aluminium and wrought aluminium alloys

EN 3665, Aerospace series — Test methods for paints and varnishes — Filiform corrosion resistance test on aluminium alloys

EN 4704, Aerospace series — Tartaric-Sulphuric-Acid anodizing of aluminium and aluminium wrought alloys for corrosion protection and paint pre-treatment (TSA)

EN 4707, Aerospace series — Acid pickling of aluminium and aluminium alloy without hexavalent chromium

EN 6072, Aerospace series — Metallic materials — Test methods — Constant amplitude fatique testing

EN ISO 2409, Paints and varnishes — Cross-cut test (ISO 2409)

EN ISO 9227, Corrosion tests in artificial atmospheres — Salt spray tests (ISO 9227)

EN ISO 1463, Metallic and oxide coatings — Measurement of coating thickness — Microscopical method (ISO 1463)

EN ISO 2085, Anodizing of aluminium and its alloys — Check for continuity of thin anodic oxidation coatings — Copper sulfate test (ISO 2085)

EN ISO 2360, Non-conductive coatings on non-magnetic electrically conductive basis materials — Measurement of coating thickness — Amplitude-sensitive eddy-current method (ISO 2360)

EN ISO 9220, Metallic coatings — Measurement of coating thickness — Scanning electron microscope method (ISO 9220)

3 Purpose of process

The anodizing is an electrochemical process voltage controlled allowing transforming the metal surface in a microporous oxide layer made of alumina. The aim of this treatment is to ensure a protection against the corrosion, and/or to be used as an adhesion base before bonding or before painting. This anodizing is generally sealed for protection corrosion application (with or without painting or bonding) and can stay unsealed when the part is bonded or painted.

This specification is applicable on aluminium and aluminium alloys generally on single parts.

Hard anodizing and plasma electrolytic anodizing dedicated to wear protection are not covered by this specification.

3.1 Applicability

3.1.1 Type A: unsealed anodizing

It shall be used either as surface preparation before the application of painting/bonding or any other finishing.

3.1.2 Type B: sealed anodizing

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

See Table 1.

Table 1 — Different application cases

	Unsealed (type A)			Sealed (type B)		
	Unpainted	Painted	Bonding (structural)	Unpainted	Painted	
Sulfuric acid anodizing (SAA) EN 2284					Applicable	
Thin film sulfuric acid anodizing (TFSAA)	Not applicable		N . 1 11	A 11 11		
Tartaric sulfuric acid anodizing (TSA) EN 4704		Not applicable Applicable	Applicable	Not defined yet with chromate free sealing		
Boric sulfuric acid anodizing (BSAA)						
Phosphoric acid anodizing (PAA)			Applicable Net you	Not applicable	Not applicable	
Sulfuric phosphoric acid anodizing (PSA)			Applicable	Not applicable		

3.2 Limitations

All processes that can compromise the anodic film such as forming, or heat-treatment shall be performed prior to surface preparation of the parts to be anodized.

Anodizing shall not be applied:

- in electric conductivity zones/areas;
- for tubes, pipes and open holes with a length to diameter ratio higher than 10:1 (unless using specific cathode);
- for trapped holes with a length to a diameter ratio greater than 5:1;
- for parts or assemblies (e.g. spot-welded and riveted), which can permanently entrap treatment solutions;
- for components which can permanently entrap treatment solutions, except components that can be adequately masked.

NOTE The formation of oxide layer influences the dimensions of the part and is to be considered for close tolerance parts.

4 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

4.1

de-anodizing

process, which removes the anodic oxide

4.2

smut

precipitations of alloying elements (e.g. Cu, Fe, Zn, Si) on the surface of parts after a process step normally after alkaline etching step

4.3

Mechanically Disturbed Layer

MDL

layer that is present at the surface resulting from the rolling process of the material

4.4

pit

surface corrosion defect at which the anodic coating is penetrated

Note 1 to entry: Typical characteristics of corrosion pits are:

- rounded or irregular or elongated geometry,
- comet tail or line or halo that emerges from the cavity,
- some corrosion by-products inside pits (on aluminium specimens the by-product may be granular, powdery or amorphous and white, grey or black in colour).

To be considered as a corrosion pit, a surface cavity must exhibit at least two of the above characteristics.

4.5

process instruction

document that describes the application scopes, detailed process (key parameters, detailed steps, etc.), quality management, environmental and safety regulations, etc.

4.6

alloys

all aluminium alloys, that are treated with the chromate free anodizing process in the specific shop

4.7

re-anodizing

repetition of the anodizing process step after complete de-anodizing

4.8

sealing

chromate free sealing (of the anodized layers) is applied to close the pores produced by the acid anodizing process.

Note 1 to entry: It is usually applied in hot demineralized water bath with or without additives at different temperatures. Sealing improves the corrosion resistance performance of the anodic film.

4.9.

batch

unless otherwise specified, it comprises parts of the same type (i.e. shape, size, material), processed at the same time in the same bath

5 Protection system classification

5.1 System types

Anodizing layer is classified by the two following types:

- Type A: unsealed anodizing: It shall be used as surface preparation before the application of painting/ bonding or any other finish.
- Type B: sealed anodizing: It is intended for corrosion protection. It shall be with or without dyeing and used with or without additional painting.

5.2 Layer thicknesses

See Table 2.

Table 2 — Layer thicknesses corresponding to the class type

Class type	Typical thickness	Anodizing process		
Class 1 < 1 um		Phosphoric acid anodizing (PAA) Sulfuric phosphoric acid anodizing (PSA) ^a		
Class 2	2 μm to 8 μm	Tartaric sulfuric acid anodizing (TSA) Boric sulfuric acid anodizing (BSAA) Thin film sulfuric acid anodizing (TFSAA)		
Class 3	8 μm to 25 μm	Sulfuric acid anodizing (SAA)		
^a ≤ 5 µm for some Aluminium alloys under agreement between purchaser and supplier.				

6 Process requirements

6.1 Information for the processor

- type and class designation,
- substrate standard reference and heat treatment,
- areas to be anodized.
- anodized thickness measuring points,
- electrical contact points or areas where these are inadmissible,
- specification for testing on parts and/or samples.

6.2 Condition of parts prior to the treatment

Welding, soldering/brazing, mechanical operations and heat treatments shall have been completed.

- the parts shall be free of oil, grease, marking inks and other surface contaminations;
- the surface shall be free from precipitations or smut from alloying elements or pre-processes indicated by the bright and uniform appearance of the surface;
- mechanically disturbed layer shall be removed either by mechanical or chemical processes;
- in case of re-anodizing all residuals from the previous anodizing shall be completely removed.

6.3 Process conditions

6.3.1 Tooling

The tools, bars, electrical contact systems, and metal masking tooling must be free of corrosion or any other damage which may be detrimental to the treatment during use. The part racks and tools must be designed and set up in such a manner as to:

- avoid any retention of air or treatment solution in the parts,
- facilitate neutralization and removal of solutions during rinsing operations,
- the electrical contacts must be kept in good condition for the correct passage of the current
- avoid any accidental contact between the parts to be treated and the tank equipment or electrodes, and between the different parts during all the process.
- electrical contact points should be defined between purchaser and processor;
- the fixturing tools (e.g. in aluminium alloy or titanium) must provide effective electrical contact with the parts;
- the contact is preferably achieved at several points in order to ensure better current distribution.

6.3.2 Masking

The parts shall be at least degreased prior to masking.

Component areas which must not be coated shall be masked with suitable material.

6.3.3 Surface pre-treatment

Surface preparation means any method able to eliminate completely all surface contaminations.

In case of chemical pre-treatment, the final step prior to anodizing shall be acidic pickling, preferably chromate-free.

Anodizing must be performed immediately after pickling (in accordance with aluminium and aluminium alloys pickling standard EN 4707).

6.3.4 Anodizing

During the anodizing process:

- distance between part and electrode must be defined to have the requested anodic layer thickness without electrical arc;
- parts should be fully immersed;
- the parts shall not be subjected to any tensile, flexure, torsion or other stress;
- the process shall be performed in such a way that parts do not dry between single process steps (pre-treatment, anodizing, etc.);
- the anodizing parameters (temperature, voltage, time) shall be adapted to the material and its requirements in accordance with the bath composition.
- in case of re-anodizing, the former protection will have to be totally removed before (chemically or mechanically) (see 6.5).

6.3.5 Anodizing post treatments

After the anodizing procedure:

- parts have to be adequately rinsed with water according to 6.4;
- afterwards, the parts shall be either:
 - dried immediately afterwards and painted within 16h; this time can be extended in accordance with customer requirements. In this case, it is recommended to handle the parts output anodizing treatment with gloves.
 - dried immediately afterwards and bonded within 8h; this time can be extended in accordance with customer requirements. In this case, it is recommended to handle the parts output anodizing treatment with gloves.
 - sealed with chromate free solution to achieve the desired corrosion resistance of the anodic film.

6.4 Water quality

All water used for bath make-up and addition of the anodizing and for the final rinsing step as well as for the sealing process shall be demineralized.

6.4.1 Anodizing bath

The water shall comply with the following requirements:

— pH value at 25 °C: 5,0 to 8,0

— total residue [mg/l]: ≤ 5

— conductivity [μ S/cm]: ≤ 2

6.4.2 Sealing and dyeing bathes

The water shall comply with the following requirements:

— pH value at 25 °C: 5,0 to 8,0

— total residue [mg/l]: ≤ 5

— conductivity [μ S/cm]: ≤ 2

— Silica content [mg/l]: ≤ 1

6.4.3 Rinsing bath

The water shall comply with the following requirements:

— pH value at 25 °C: 5,0 to 8,0

— total residue [mg/l]: ≤ 5

— conductivity [μ S/cm]: ≤ 20

6.5 Re-anodizing

One re-anodizing is allowed; any further re-anodizing shall be agreed between the interested parties.

7 Engineering requirements

7.1 General

The anodizing is expected to meet the main requirements summed-up in Table A.1.

7.2 Visual aspects

The film shall be free of powdery areas, burnings, blisters and discontinuities such as scratches, ruptures or other damages.

7.3 Film thickness

The thickness of the film can be checked by eddy current method (EN ISO 2360) which may be correlated with optical microscope (EN ISO 1463), scanning electronic microscope (SEM) (EN ISO 9220), or equivalent examinations.

7.4 Corrosion prevention performance of unpainted parts

When tested in accordance to EN ISO 9227 the anodized and sealed post-treated specimens' corrosion performances (see Table A.1) shall be determined following:

- number of pits/dm² (see 4.4),diameter of pits,
- exposure time,
- aluminium grade.

The area of 2 mm from the specimen edges and marks shall not be evaluated. It is recommended to use samples with surface $\geq 1 \text{ dm}^2$.

7.5 Paint adhesion on anodic film

Unless otherwise agreed with the purchaser, the damage of painted specimens when tested in accordance with EN ISO 2409 shall not exceed at initial state and after 14 days water immersion.

Classification 1 max. on unsealed anodizing;

and

Classification 2 max. on sealed anodizing.

The fulfilment of these requirements shall be demonstrated using two sets of specimen, whereby the second set is required only when the maximum open time (time between anodizing and paint application) applied at the shop exceeds 16 h.

- Set 1: specimen shall be painted to max 16 h after anodizing taking into account storage under ambient conditions of the shop (standard);
- Set 2: shall be painted after the maximum open time agreed with the customer.

7.6 Corrosion prevention performance of painted parts

7.6.1 Filiform corrosion

The damage of painted specimens shall be determined according the maximum length of the longest filament on either side of the scratches at 40 days of exposure when tested in accordance with EN 3665. There shall not be any corrosion, extending further than max. 1,25 mm on either side of the scratch at 125 days of exposure when tested in accordance with EN ISO 9227.

7.6.2 Corrosion resistance

When tested in accordance to EN ISO 9227 the painted specimens' corrosion performances (see Table A.1) shall be determined following:

- number of pits,
- exposure time,
- paint system.

7.7 Fatigue requirements

Unless otherwise specified, the fatigue specimen shall be manufactured and tested in accordance with EN 6072.

For each process, all the fatigue test values shall be plotted on the Wöhler curves (mean and minimum curves).

- at least 50 % of the samples shall be on or above the mean curve;
- 90 % of the samples shall be above the minimum curve.

7.8 Bonding check

See Table A.1.

8 Quality requirements

8.1 Process approval

The processor shall carry out:

- the anodizing on pre-production parts and/or samples determined by agreement between the processor and the purchaser;
- the tests specified in this European Standard, unless otherwise agreed between the processor and the purchaser.

The process chart defined in the manufacturers process instructions shall not be changed without any previous agreement from the purchaser.

8.2 General points

During all the process operations, the operating conditions must be within the parameters defined in the processors detailed process instructions.

Regular controls must be performed on the facilities and the products used and on conformance of the application process conditions to the requirements of this document and of the manufacturers detailed process instructions.

All records of process parameters, tests and inspection results shall be registered under the control of the quality assurance of the workshop.

All process operators shall be adequately trained.

In order to verify the above specified design requirements and maintain the process with appropriate performances, the process needs to be checked by:

- periodic tests on samples coated under the same conditions as the parts,
- periodic bathes chemical analysis,
- parts acceptance controls.

The minimum tests and controls are defined in the paragraphs below. The frequency and nature of other tests shall be determined by agreement between the processor and the purchaser.

8.3 Periodic tests

The Table B.1 synthesizes all the periodic tests on samples.

8.4 Periodic chemical analysis

The bathes composition shall be in the limits as specified in the manufacturers detailed process instruction. The periodicity of the analysis shall be defined considering the ageing, contamination and the efficiency of the bathes, the production rate and the re-adjustment or refilling of the bathes.

8.5 Parts acceptance controls

8.5.1 Controls before treatment

A visual inspection must be performed in order to ensure that the parts to be treated have no defects such as cracks, stripes, pits, corrosion or other defects which can be harmful for parts, for anodizing appearance and performance.

8.5.2 Controls during treatment

At the end of the degreasing phases, the rinsing phase must be followed by the water break test to evaluate the wettability (and therefore the cleanliness) of the surfaces.

This test consists in covering the parts with a uniform water film. This water film must cover the parts during at least 30 s without any film discontinuity; otherwise, degreasing phases have to be repeated.

8.5.3 Controls after anodizing

The Table B.3 synthesizes the minimum parts acceptance controls after anodizing.

Annex A

(normative)

Engineering requirements

See Table A.1.

Table A.1

Characteristics		55		Class 3 (SAA)			
Visual		The film shall be free of powdery areas, burnings, blisters and discontinuities such as scratches, ruptures or other damages.					
Anodizing thickness		≤ 1 µm ^a	2 μm to 8 μm	8 μm to 25 μm			
C. II	Sealed with hot water	Not applicable	≥ 96 h ≤ 2 pits/dm² (see 4.4)	≥ 500 h ≤ 2 pits/dm² (see 4.4)			
Salt spray corrosion	Painted	According to manufacturer requirements					
resistance in accordance with EN ISO 9227	Sealed in 2 steps:1st step sealed with inhibitors - 2nd step sealed with hot water	Not applicable	0 pits/dm² (see 4.4) ≥ 500 h	0 pits/dm² (see 4.4) ≥ 500 h			
Painting adhesi		On unsealed anodizing: classification 1 max.					
state and after 14 days water immersion (according EN ISO 2409)		On sealed anodizing: classification 2 max.					
Fatigue performance		According to manufacturer requirements					
Bonding performance: peeling test		According to manufacturer requirements					
Bonding performance: Shearing test		According to manufacturer requirements					
a ≤ 5 μm for	some Aluminium	alloys under agreement	between purchaser and supplier.				

Annex B (normative)

Quality requirements

B.1 Quality requirements

See Table B.1.

Table B.1 — Quality requirements

Characteristics		Class 1 (PAA, PSA)	Class 2 (TFSAA, TSA, BSAA)	Class 3 (SAA)	Minimum periodicity	Material	Samples Number	Dimension
Visual		The film shall be free of powdery areas, burnings, blisters and discontinuities such as scratches, ruptures or other damages.				All samples		
Anodizing thickness		≤1	2 μm to 8 μm	8 μm to 25 μm				According to manufacturer requirements
Layer continuit	ty	According to m	According to manufacturer requirements ^a					
Sealing control (see Clause B.2 and Table B.2)		Not applicable	Intensity of the 0, 1 or 2 accordin Clause B.2.					
	Sealed with hot water	Not applicable	≥ 96 h ≤ 2 pits/dm² (see 4.4)	≥ 500 h ≤ 2 pits/dm² (see 4.4)	According manufactur	According to	nanufacturer	
Salt spray corrosion resistance in accordance with EN ISO 9227	Sealed in 2 steps: 1st step sealed with inhibitors - 2nd step sealed with hot water	Not applicable	≥ 500 h no pit (see 4.4)	≥ 500 h no pit (see 4.4)		manufacturer requirements		Minimum surface of 1 dm ²
	Painted	According to m	anufacturer requirements					
Painting adhesion at initial state and after 14 days water immersion (according EN ISO 2409)		On unsealed anodizing: classification 1 max.						
		On sealed anod	izing: classificati	on 2 max.	According to manufacturer requirements			According to manufacturer requirements
Bonding control: peeling test		According to m	anufacturer requ	irements				

This test shall be applied in a non-critical area when there is a doubt about the continuity of the layer on a part or on a specimen (localised dissolution of oxide layer).

B.2 Interpretation of the results of the dye-spot test

Clean the surface to be tested, removing any grease with a piece of cotton wool soaked in, for example, acetone or ethanol $96\,\%$.

Apply one drop of dye solution A or dye solution B to the spot, and allow it to remain for 1 min.

Wash off the drop of dye and clean the surface of the test area thoroughly by rubbing with a clean cloth soaked in water and light abrasive, such as magnesia whiting or an equivalent abrasive, for 20 s. Rinse thoroughly and dry.

Examine the test area and assess the intensity of the stain by comparison with the examples illustrated in Table B.2.

Dye solution A

Aqueous solution containing 5 g of Sanodye Blue 2LW formerly Sanodal Blue 2LW (Colour Index Mordant Blue 69) per litre, adjusted, at approximately 23 °C, to a pH of 5.0 ± 0.5 with dilute sulfuric acid solution or with dilute sodium hydroxide solution.

Dye solution B

Aqueous solution containing 10 g of Sanodal Red B3LW (Colour Index Acid Red 331) per litre, adjusted, at approximately 23 °C, to a pH of 5.7 ± 0.5 with dilute sulfuric acid solution or with dilute sodium hydroxide solution.

Table B.2 — Interpretation of the results of the dye-spot test

SANODYE BLUE 2LW	SANODAL RED B3LW	Intensity of the stain	Loss of absorptive power
		5	none
		4	very weak
		3	weak
0		2	medium
- 10		1	strong
		0	total

 ${\bf Table~B.3-Minimum~parts~acceptance~controls~after~anodizing}$

Туре	Frequency	Method and criteria
Visual inspection	100 %	The parts have to be inspected by naked eyes under constant light conditions. Checking of powdery areas or dusting is made with the help of a white and dry rag, which is slightly rubbed on the part to check.
		All relevant areas of the parts have to be covered by the anodic film. The anodic film shall be uniform, free of powdery areas, burnings, blisters and discontinuities such as scratches, ruptures or other damages. There shall be no water stains on surfaces to which a coating is to be applied.
Thickness of the anodic film	After agreement between manufacturer and customer	The thickness of the film can be checked by eddy current method (EN ISO 2360), which may be correlated with optical microscope (EN ISO 1463), scanning electronic microscope (SEM) (EN ISO 9220) or equivalent examinations. The thickness of the anodizing layer shall be in the range required in 5.2.
		The breakdown voltage can be investigated for dedicated application in accordance with EN ISO 2376.
Continuity of the sealed anodic film	1 part per batch, in case of failed test 100 % of the batch should be checked	The continuity of the film can be determined in accordance with EN ISO 2085 only on sealed anodizing (type B). This test shall be applied in a non-critical area.
Corrosion resistance	After agreement between manufacturer and customer	Salt spray corrosion resistance in accordance with EN ISO 9227. The results shall be in accordance with requirements (see Table A.1).
Sealing quality	1 part per batch	When tested in accordance with Clause B.2, the dye coloration shall be very light or has completely disappeared (grade 0 to 2 according Table B.2.
Paint adhesion	After agreement between manufacturer and customer	Painted specimens shall be tested in accordance with EN ISO 2409. Unless otherwise agreed with the purchaser, the adhesion grade shall not exceed at initial state and after 14 days water immersion.
		Classification 1 max. on unsealed anodizing;
		and Classification 2 max. on sealed anodizing.

Bibliography

- [1] EN 2101, Aerospace series Chromic acid anodizing of aluminium and wrought aluminium alloys
- [2] EN 12373-2, Aluminium and aluminium alloys Anodizing Part 2: Determination of mass per unit area (surface density) of anodic oxidation coatings Gravimetric method
- [3] EN ISO 15528, Paints, varnishes and raw materials for paints and varnishes Sampling (ISO 15528)
- [4] EN ISO 2376, Anodizing of aluminium and its alloys Determination of electric breakdown potential (ISO 2376)



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