



AEROSPACE MATERIAL SPECIFICATION

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Sulfuric Acid Anodizing of Aluminum and Aluminum Alloys

RATIONALE

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FOREWORD

REVISION NOTE

This Defence Standard was raised to Issue 5 to update its content and incorporate the latest MoD/DStan policy in place at the time.

HISTORICAL RECORD

This standard supersedes the following:

Def 151 dated March 1965

Def Stan 03-25 Issue 1 dated 6 September 1984

Def Stan 03-25 Issue 2 dated 14 September 1988

Def Stan 03-25 Issue 3 dated 22 August 1997

Def Stan 03-25 Issue 4 dated 16 December 2003

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INTRODUCTION

This SAE Standard specifies the properties of sulphuric acid anodizing of aluminum and aluminum alloys.

Sulphuric acid in mist form is considered a carcinogenic (see Health Protection Agency Guidance on the use of Sulphuric Acid) and care must be exercised where mist sprays are produced during processing. The latest Health and Safety Executive guidance recommends the use of "pumped flow eductor systems" to help minimize the effects of mist sprays.

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TABLE OF CONTENTS

1.	SCOPE	4
2.	WARNING	4
3.	NORMATIVE REFERENCES	4
4.	DEFINITIONS	5
5.	INFORMATION TO BE SUPPLIED TO THE PROCESSOR	5
6.	PROCESS CONTROL	6
7.	APPLICATIONS AND LIMITATIONS	6
7.1	Applications	6
7.2	Compatibility with Explosives, Propellants, and Pyrotechnics	6
7.3	Limitations	6
8.	PROCESS REQUIREMENTS	7
8.1	Surface Preparation	7
8.2	Anodizing	7
8.2.1	Process	7
8.2.2	Coating Thickness	7
8.3	Washing	7
8.4	Dyeing	7
8.5	Sealing	7
8.6	Drying	8
8.7	Re-Anodizing	9
9.	INSPECTION AND TEST	9
9.1	Processing	9
9.2	Frequency of Testing	9
9.3	Appearance	9
9.4	Thickness of Coating	9
9.5	Dyed Coatings	9
9.5.1	Resistance to Leaching	9
9.5.2	Fastness to Light	9
9.6	Sealing Assessment	10
9.7	Electrical Insulation	10
9.8	Abrasion resistance	10
9.9	Reflectivity	10
9.10	Corrosion Test	10
10.	NOTES	10
ANNEX A	SULFURIC ACID ANODIZING PROCESS FOR ALUMINIUM AND ALUMINIUM ALLOYS	11
ANNEX B	ANALYSIS OF SULFURIC ACID ELECTROLYTE	12
ANNEX C	STRIPPING SOLUTIONS FOR THE REMOVAL OF ANODIC COATINGS	14
Table 1	Sealing solutions	8

1. SCOPE

This SAE Standard specifies the properties of sulphuric acid anodizing of aluminum and aluminum alloys.

2. WARNING

This section which appeared in DEF STAN 03-25 Issue 5 has been deliberately deleted.

3. NORMATIVE REFERENCES

3.1 The publications shown below are referred to in the text of this standard. Publications are grouped and listed in alphanumeric order.

BS EN ISO 3696	Water for Analytical Laboratory Use: Specification and Test Methods
BS EN ISO 2360	Non-Conductive Coatings on Non-magnetic Basis Metals. Measurement of Coating Thickness. Amplitude Sensitive Eddy Current Method.
BS EN ISO 9227	Corrosion Tests in Artificial Atmospheres. Salt Spray Tests.
BS EN 12373-2	Aluminium and Aluminium Alloys. Anodizing. Determination of Mass Per Unit Area (Surface Density) of Anodic Oxidation Coatings. Gravimetric Method.
BS EN 12373-5	Aluminium and Aluminium Alloys. Anodizing. Assessment of Quality of Sealed Anodic Oxidation Coatings by Measurement of Admittance.
BS EN 12373-8	Aluminium and Aluminium Alloys. Anodizing. Determination of the Comparative Fastness to Ultra-Violet Light and Heat of Coloured Anodic Oxidation Coatings.
BS EN 12373-9	Aluminium and Aluminium Alloys. Anodizing. Measurement of Wear Resistance and Wear Index of Anodic Oxidation Coatings Using an Abrasive Wheel Wear Test Apparatus
BS EN 12373-10	Aluminium and Aluminium Alloys. Anodizing. Measurement of Mean Specific Abrasion Resistance of Anodic Oxidation Coatings Using an Abrasive Jet Test Apparatus
BS EN 12373-17	Aluminium and Aluminium Alloys. Anodizing. Determination of Electrical Breakdown Potential.
BS 6161 Part 18	Methods of Test for Anodic Oxidation Coatings on Aluminium and Its Alloys. Determination of Surface Abrasion Resistance
Def Stan 03-2	Cleaning and Preparation of Metal Surfaces
AMS03-2	Cleaning and Preparation of Metal Surfaces
Def Stan 03-21	Mechanical Methods for the Inducement of Compressive Surface Residual Stresses
Def Stan 03-24	Chromic Acid Anodizing of Aluminium and Aluminium Alloys
Def Stan 07-85 Pt 2	Design Requirements for Weapons and Associated Equipment. Supplementary and Design Process Requirements

3.2 This section which appeared in DEF STAN 03-25 Issue 5 has been deliberately deleted.

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4. DEFINITIONS

4.1 For the purposes of this Part of the Standard the following definitions apply:

4.2 BATCH

A batch consists of items of the same material specification processed together in the same anodizing bath.

4.3 PROCESS CONTROL SCHEDULE

The document which specifies/defines:

- a. The sequence of manufacturing operations and processes.
- b. The control parameters and their tolerances for each individual process within the total sequence.

4.4 PURIFIED WATER

Water which has been produced by distillation, deionization or reverse osmosis, having a conductivity measurement not greater than 30 $\mu\text{S}/\text{cm}$ at 20 °C and a silica content of not greater than 5 ppm w/w (as SiO_2).

4.5 SIGNIFICANT SURFACE

That area of the item covered, or to be covered by the coating, and for which the coating is essential for serviceability and/or appearance.

5. INFORMATION TO BE SUPPLIED TO THE PROCESSOR

The following information shall be given on the drawing, contract or order:

- a. The number of this AMS Material Specification or its superseded Defence Standard.
- b. The required film thickness (see 8.2.2).
- c. The specification, heat treatment, and surface condition of the aluminum alloy.
- d. The sealing requirements (see 8.5).
- e. The significant surface. This may be indicated on the drawing or by a marked sample.
- f. Any preference for position of contacts.
- g. The surface finish where there is a special requirement.
- h. The color, where articles are to be dyed. This may be by the provision of an anodized and dyed sample.
- i. Any special conditions of service, e.g., contact or close proximity to Explosives, Propellants or Pyrotechnics or Hydrogen Peroxide (HTP).
- j. Any special condition of service, e.g., Aerospace Applications requiring Mandatory Process Control Schedules (see Section 6) with HTP.

k. Any limit on the number of re-treatments (see 8.7).

l. Any special test requirements (see 9.5 to 9.10).

6. PROCESS CONTROL

6.1 A Process Control Schedule suitable for achieving the requirements of this Standard shall be prepared by the processing contractor(s) prior to the commencement of production.

6.2 Details of the coating process, including all preparatory treatments, coating treatment and processing, significant surface, tests and all other treatments shall be included in the Process Control Schedule.

6.3 All stages in the complete schedule shall follow each other without delay.

7. APPLICATIONS AND LIMITATIONS

7.1 Applications

7.1.1 The sulfuric acid anodizing process is capable of yielding a range of coating thicknesses on most aluminum alloys. It is suitable for general protective purposes and for the production of a suitable surface for subsequent painting or adhesive bonding.

7.1.2 The process produces coatings suitable for coloring with organic dyestuffs or certain inorganic compounds.

7.1.3 The process produces coatings which are electrically insulating. Where anodizing is being used to impart this property it may be necessary to specify a maximum surface roughness for the items prior to anodizing, as the initial surface finish will affect the electrical resistance of the coating.

7.1.4 It is the preferred process for anodizing those aluminum alloy castings containing more than 5% but less than 7% of copper, or containing a specified maximum total of 7% of (copper + nickel + iron).

7.1.5 The process shall be specified where anodizing is required on aluminum alloy items which are liable to be used in contact with concentrated hydrogen peroxide (HTP).

7.2 Compatibility with Explosives, Propellants, and Pyrotechnics

7.2.1 Whilst anodizing is not essential to render aluminum or aluminum alloys compatible with explosives, propellants or pyrotechnics, anodized surfaces may be expected to be compatible provided that all residues of reagents used in anodizing and sealing are removed by the final washing process.

7.2.2 Sulfuric acid anodizing of items of explosive stores will normally be permitted when the treated surfaces are not to be placed in contact or close proximity with explosives, propellants, or pyrotechnics. Otherwise chromic acid anodizing (Def Stan 03-24) shall be used.

7.2.3 Advice regarding the choice of process for applications where compatibility with explosives, propellants or pyrotechnics is a requirement, may be obtained from the responsible Technical or Supervising Authority named in the tender, contract or order. See also Def Stan 07-85 section 10.5.9.3.3.

7.3 Limitations

7.3.1 The process is unsuitable for items having riveted, lap or folded joints, tubes, or other items where there is a risk of electrolyte becoming entrapped.

7.3.2 The process is not suitable for items of less than 0.25 mm section except for applications where a coating thickness of not more than 2 μm will suffice.

7.3.3 The process shall not be used on alloys containing more than 7% copper.

7.3.4 The fatigue strength may be significantly reduced by the anodizing process. This may be restored to some extent by sealing the coating in aqueous dichromate solution at the cost of some loss of abrasion resistance or by shot peening in accordance with Def Stan 03-21 before anodizing.

7.3.5 Composite items which include non-aluminum materials, which would be attacked by the process, or would prevent satisfactory formation of the anodic coating on the aluminum alloy, or would cause attack of the aluminum alloy, shall not be anodized as assemblies unless the non-aluminum surfaces are effectively masked.

7.3.6 All heat treatment procedures shall be completed prior to anodizing.

8. PROCESS REQUIREMENTS

8.1 Surface Preparation

Surfaces shall be cleaned and free from grease, oil, oxide, scale or other foreign matter. All items shall be degreased prior to commencement of a cleaning sequence which shall be in accordance with AMS03-2/Def Stan 03-2 to produce a chemically clean surface.

8.2 Anodizing

8.2.1 Process

The standard process described in Annex A and method of analysis of the electrolyte described in Annex B shall be used.

8.2.2 Coating Thickness

Unless otherwise stated on the drawing, contract or order, the coating thickness shall be 8 to 13 μm , except that items which are to be dyed black may have a coating thickness of not greater than 25 μm .

8.3 Washing

8.3.1 Immediately after removal from the anodizing bath all items shall be washed to remove electrolyte as follows:

8.3.1.1 Items which have been anodized, other than for painting or adhesive bonding, shall be washed in clean cold running water. They shall be finally rinsed in purified water at a temperature not exceeding 60 °C and shall then be sealed in accordance with 8.5.

8.3.1.2 Items which have been anodized for painting or adhesive bonding shall be washed in clean cold running water. They shall then be finally rinsed in purified water at a temperature not exceeding 60 °C.

8.3.1.3 Water used for final rinsing shall be discarded or retreated when the conductivity exceeds 100 $\mu\text{S}/\text{cm}$ and/or when the silica content becomes greater than 5 ppm w/w (as SiO_2).

8.4 Dyeing

Items to be dyed shall be transferred to the dye bath immediately after washing and without allowing the items to dry. After dyeing, the items shall be rinsed in purified water prior to any sealing operation required.

8.5 Sealing

8.5.1 Items other than those to be dyed, or required to retain the natural anodized color, or liable to be used in contact with concentrated HTP, or to be subsequently painted or adhesive bonded, shall be sealed by immersion in one of the solutions from Table 1.

Table 1 - Sealing solutions

Solution	Component Part	Measure
A	Potassium dichromate ($K_2C_2O_7$)	70 -100 g
	or	
	Sodium dichromate ($Na_2Cr_2O_7 \cdot H_2O$)	18 g
	Sodium carbonate (Na_2CO_3)	
	or	13 g
	Sodium hydroxide (NaOH)	
	Purified Water	1 Liter
B	pH (glass electrode)	6.3 to 7.4
	or	
	pH (bromothymol blue)	40 - 60 g
	Potassium dichromate ($K_2C_2O_7$)	
	or	
	Sodium dichromate ($Na_2Cr_2O_7 \cdot H_2O$)	1 Liter
	Purified Water	
pH (glass electrode)	5.6 to 6.0	
or	5.9 to 6.4	
pH (bromothymol blue)		

8.5.1.1 The temperature of the solution shall be maintained at not less than 96 °C. Items shall be immersed for not less than the anodizing time. The pH value of the solution shall be maintained by the addition of chromic acid or sodium hydroxide as necessary. After sealing, the items shall be thoroughly washed in clean cold running water.

8.5.2 Items which are liable to be used in contact with concentrated HTP, shall be sealed by immersion in purified water at not less than 96 °C for not less than 20 minutes, at a pH of 5.5 to 7.0 (corrected to 20 °C), adjusted by the addition of one of the following: sodium hydroxide, ammonium hydroxide, or acetic acid as necessary.

8.5.3 Items which have been dyed or are required to retain the natural anodized color shall be sealed by the treatment described in 8.5.2.

8.5.4 Items anodized as a pre-treatment for painting or adhesive bonding shall not be sealed.

8.6 Drying

8.6.1 Items other than those anodized as a preparation for painting or adhesive bonding may be dried at elevated temperatures; the temperature shall not exceed 110 °C. The duration at this temperature shall not exceed 10 minutes.

8.6.2 Items to be subsequently painted or adhesive bonded shall be dried at a temperature not exceeding 60 °C. Suitable precautions shall be taken to prevent contamination.

8.6.3 Drying by means of chlorinated solvents containing surface active agents shall not be used.

8.7 Re-Anodizing

8.7.1 Items which are to be re-anodized shall have the anodic coating removed before re-treatment.

8.7.2 The use of stripping solutions for the removal of anodic coatings may result in an attack on the substrate which, in finished items, may adversely affect their fatigue strength. Suitable chemical methods are described in Annex C.

8.7.3 Attention is also drawn to the desirability of abrasive blasting (AMS03-2/Def Stan 03-2, Method D) before re-anodizing. Some measure of compressive stress is introduced into the surface by this method.

9. INSPECTION AND TEST

9.1 Processing

The processing methods employed shall comply with the Process Control Schedule where specified (see Section 6).

9.2 Frequency of Testing

9.2.1 Visual examination shall be applied to all items. The test for thickness of coating (see 9.4) and, where specified, any of the tests in 9.5 to 9.10, which may be applicable shall be carried out on not less than 2% of the items represented, with a minimum of one item per anodizing batch.

9.2.2 In exceptional circumstances, e.g., the treatment of a small number of large items or a large number of small items, the frequency of such tests may be modified. Coupon samples coated together with the items may be used, due consideration being given to their shape, size, and material. The treatment of the coupon samples shall be representative of that applied to the items being coated.

9.3 Appearance

The significant surface of all items shall be completely anodized and shall be of uniform color. All items except those which have been dyed, or are required to retain the natural anodized color, or are to be used in contact with concentrated hydrogen peroxide (HTP), or are to be subsequently painted or adhesive bonded, shall have a yellow color indicating that chromate sealing has been carried out. The color will be affected by alloying constituents. The color of dyed items shall match that of an agreed standard or sample. Minor variations in color shall not normally be cause for rejection. The presence of processing residues shall be cause for rejection.

9.4 Thickness of Coating

The average thickness shall be determined either by the gravimetric stripping method described in BS EN 12373-2 or the eddy current instrument method described in BS EN ISO 2360.

9.5 Dyed Coatings

9.5.1 Resistance to Leaching

The samples of dyed and sealed items shall be immersed for 30 minutes in a gently boiling aqueous solution of 0.1% (m/v) borax. They shall then show no perceptible loss of color when compared with an untested sample from the same batch of anodized items.

9.5.2 Fastness to Light

The fastness to light determined by the method described in BS EN 12373-8 shall be not less than the values specified on the drawing, contract or order.

9.6 Sealing Assessment

- 9.6.1 Undyed coatings on wrought alloy sealed by the method specified in 8.5.2 shall be tested by the method described in BS EN 12373-5. This method shall not be used for other sealed coatings.
- 9.6.2 The staining intensity for 1000, 3000, 5000, and 6000 series alloys shall not be greater than 1 according to the annex of BS EN 12373-5.
- 9.6.3 For 2000, 7000, and 8000 series wrought alloys, the staining intensity shall be agreed between the contractor and purchaser.

9.7 Electrical Insulation

The electrical breakdown voltage, determined by the method described in BS EN 12373-17, shall be not less than the values specified on the drawing, contract or order.

9.8 Abrasion resistance

- 9.8.1 The abrasion resistance, assessed by the method described in BS EN 12373-9 or BS EN 12373-10, shall comply with the standard of acceptance stated on the drawing, contract or order.
- 9.8.2 For production control purposes, the method described in BS 6161: Part 18 may be used for 1000, 3000, 5000, and 6000 series wrought alloys only.

9.9 Reflectivity

The total light reflectivity, specular light reflectivity, image clarity and infrared reflectivity, determined by the appropriate method described in BS EN 12373 or BS 6161 series, shall comply with the standards of acceptance stated on the drawing, contract or order.

9.10 Corrosion Test

Where the Process Control Schedule requires a corrosion test, a test panel (minimum dimensions 120 mm x 60 mm) of the same material specification as the components shall be anodized and sealed along with the components. The test panel shall be subjected to the neutral salt spray test as described in BS EN ISO 9227. Where the components are of a form other than plate or sheet, the actual component shall be subjected to the test. The duration of the test shall be 336 hours with no corrosion spots or staining evident following test.

10. NOTES

- 10.1 A change bar (I) located in the left margin is for the convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of this document. An (R) symbol to the left of the document title indicates a complete revision of the document, including technical revisions. Change bars and (R) are not used in original publications nor in documents that contain editorial changes only.

ANNEX A - SULFURIC ACID ANODIZING PROCESS FOR ALUMINIUM AND ALUMINIUM ALLOYS

A.1 ELECTROLYTE

The electrolyte shall consist of an aqueous solution of sulfuric acid at a concentration of 90 to 400 g/l which may contain additives. The concentration shall be maintained within 10% of the nominal. The chloride content, expressed as NaCl, shall be less than 0.20 g/l. The aluminum content depends on the nominal concentration of the solution. It shall be less than 25 g/l for minimum concentration and less than 12 g/l for the maximum concentration of sulfuric acid. The sulfuric acid anodizing bath shall be made up with purified water. The electrolyte shall be agitated by air or mechanical means to maintain a uniform temperature throughout the bath, and should be kept clean and free from suspended matter by filtration or other means.

NOTE: The nominal electrolyte concentration will depend on the type of finish required. The lower the concentration, the harder and less porous the coating.

A.2 CATHODE

The cathode material shall be lead or antimonial lead. Where the tank is lined with one of these materials this may be used as the cathode. The sides and bottom of the tank may, if desired, be partially covered by glass or other chemically inert insulating material.

A.3 SUSPENSION OR JIGGING

The items to be anodized shall be suspended by such means that good electrical contact is maintained throughout the treatment. Any metallic parts of a suspension device which makes contact with the electrolyte shall be of aluminum or titanium. Suspension devices with spring or screw contacts are recommended. Where possible, items shall be suspended in the bath so that air or evolved oxygen cannot become entrapped, causing incomplete coating. Where such trapping is unavoidable multiple treatment shall be acceptable. Rigid items which are too small to be held in jigs may be packed in perforated aluminum or titanium canisters which shall incorporate means of maintaining electrical contact between the items and shall permit adequate circulation of the electrolyte through their interiors. Items with flat faces, such as washers, tend to nest together and cannot normally be effectively treated in canisters unless adequately dispersed by treating them with other items. Care shall be taken that items undergoing treatment do not come into contact with the tank, stirrer, heating or cooling pipes or cathodes, as this may cause breakdown of the film and damage to the items.

A.4 ANODIZING PROCEDURE

A.4.1 The operational temperature of the electrolyte shall be $20\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. The items to be treated shall be immersed in the electrolyte and connected as the anode to a suitable electrical source for a time sufficient to obtain the required coating thickness. The treatment is preferably controlled by current density (normally 100 to 200 A/m² of anode surface) but may be controlled by maintaining a predetermined voltage.

A.4.2 The anodizing conditions (temperature, voltage, time) shall be suitable for the type of alloy being processed and bath composition and shall be defined in the PCS.