AEROSPACE MATERIAL SPECIFICATION

AMS03-2

Issued 2015-04

Cleaning and Preparation of Metal Surfaces

RATIONALE

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FOREWORD

REVISION NOTE

This Defence Standard was raised to Issue 6 to update its content and incorporate the latest MoD/DStan policy in place at the time.

HISTORICAL RECORD

This standard supersedes the following:

CS2625 dated 9 August 1951

Def 38 dated 13 May 1959

Def 39 dated November 1959

Def Stan 03-2 / Issue 1 dated 28 December 1970

QTM 5 dated February 1975

Def Stan 03-2 / Issue 2 dated 1 March 1991

Def Stan 03-2 / Issue 3 dated 24 March 1994

Def Stan 03-2 / Issue 4 dated 14 October 2005

Def Stan 03-2 / Issue 5 dated 20 November 2009

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INTRODUCTION

This Standard details processes for the cleaning of metal surfaces to remove any extraneous or undesirable material or deposits at any stage of manufacture, storage or service and for the preparation of these surfaces for further treatment. Any necessary stress-relieving treatments are also included.

This Standard is not intended to override special preparatory procedures specified in individual Defence Standards, Defence Specifications, DTD Process Specifications, DG Ships Specifications and Naval Defence Standards. It also does not cover cleaning operations associated with the maintenance of internal combustion engines, the preliminary removal of heavy deposits of oil, grease, or dirt accumulated during operational service, preparations for welding and the cleaning of electrical contacts.

The standard is constructed around a small preamble section detailing the general requirements followed by Annexes detailing the requirements for individual substrate materials and the designated cleaning and preparation processes.

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1. SCOPE

This SAE Standard specifies processes for the cleaning of metal surfaces to remove any extraneous or undesirable material or deposits at any stage of manufacture, storage or service and for the preparation of these surfaces for further treatment. Any necessary stress-relieving treatments are also included. This Standard does not override special preparatory procedures specified in individual Defence Standards, Defence Specifications, DTD Process Specifications, DG Ships Specifications and Naval Defence Standards. It also does not cover cleaning operations associated with the maintenance of internal combustion engines, the preliminary removal of heavy deposits of oil, grease, or dirt accumulated during operational service, preparations for welding and the cleaning of electrical contacts.

2. WARNING

This section which appeared in DEF STAN 03-2 Issue 6 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 alpha-numeric order.

AMS2700C	Passivation of Corrosion Resistant Steels
BS EN 2516	Passivation of Corrosion Resisting Steels and Decontamination of Nickel Base Alloys
BS EN ISO 3696	Water for Analytical Laboratory Use. Specification and Test Methods.
BS EN ISO 4527	Metallic Coatings. Autocatalytic (Electroless) Nickel Phosphorus Alloy Coatings. Specification and Test Methods.
BS EN ISO 4788	Laboratory Glassware. Graduated Cylinders
BS EN ISO 6507-1	Metallic Materials. Vickers Hardness Test. Test Method.
BS EN ISO 11124-3	Preparation of Steel Substrates before Application of Paints and Related Products. Specification for Metallic Blast Cleaning Abrasives. Part 3: High-Carbon Cast-Steel Shot and Grit.
BS EN ISO 11126-7	Preparation of Steel Substrates before Application of Paints and Related Products. Specification for Non-Metallic Blast Cleaning Abrasives. Part 7F: Fused Aluminium Oxide.
BS EN 10140	Cold Rolled Narrow Steel Strip – Tolerances on Dimensions and Shape
BS 245	Specification for Mineral Solvents (White Spirit and Related Hydrocarbon Solvents) for Paints and Other Purposes
BS 558 & 564	Specification for Nickel Anodes, Anode Nickel and Nickel Salts for Electroplating
BS 2569	Specification for Sprayed Metal Coatings
BS 2869	Specification for Fuels Oils for Agricultural, Domestic and Industrial Engines and Boilers
BS 4130	Specification for Sodium Hydroxide (Technical Grades)
BS 4921	Specification for Sherardized Coatings on Iron or Steel
BS 5599	Specification for Hard Anodic Oxidation Coatings on Aluminium and its Alloys for Engineering Purposes
BS 6338	Specification for Chromate Conversion Coatings on Electroplated Zinc and Cadmium Coatings

BS 7079	Preparation of Steel Substrates before Application of Paints and Related Products
BS 2X32	Specification for Pre-Treatment Etch Primer for Aerospace Purposes
Def Stan 03-3	Protection of Aluminium Alloys by Sprayed Metal Coatings
AMS03-3	Protection of Aluminum Alloys by Sprayed Metal Coatings
Def Stan 03-4	The Pre-Treatment and Protection of Steel Items of Specified Maximum Tensile Strength Exceeding 1450 MPa
AMS03-4	The Pre-Treatment and Protection of Steel Items of Specified Maximum Tensile Strength Exceeding 1450 MPa
Def Stan 03-11	Phosphate Treatment of Iron and Steel
AMS03-11	Phosphate Treatment of Iron and Steel
Def Stan 03-18	Chromate Conversion Coatings (Chromate Filming Treatments) for Aluminium and Aluminium Alloys
AMS03-18	Chromate Conversion Coatings (Chromate Filming Treatments) for Aluminum and Aluminum Alloys
Def Stan 03-24	Chromic Acid Anodizing of Aluminium and Aluminium Alloys
Def Stan 03-25	Sulphuric Acid Anodizing of Aluminium and Aluminium Alloys
AMS03-25	Sulphuric Acid Anodizing of Aluminum and Aluminum Alloys
Def Stan 03-27	Electro-Deposition of Nickel for Engineering Purposes
AMS03-27	Electro-Deposition of Nickel for Engineering Purposes
Def Stan 08-39	Cutting, Welding and Corrosion Protection of Aluminium Alloy Military Equipment
Int. Def Stan 68-160	Degreasing Compound, Types A and C
Def Stan 68-188	Emulsifying Agent, Cleaning Compound
Def Stan 68-189	Solvent for Grease, Emulsifiable
Def Stan 80-15	Paint, Pre-Treatment Primer (Etching Primer)
DTD 911	Protection of Magnesium-Rich Alloys Against Corrosion (Obsolescent)

3.2 This section which appeared in DEF STAN 03-2 Issue 6 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 TENSILE STRENGTH OF STEELS

4.2.1 Tensile Strength

Throughout this Standard the tensile strength figures refer to the specified maximum tensile strength. Where the steel specification specifies only the minimum tensile strength the equivalent maximum tensile strength category shall be determined from Table 1.

Table 1 - Specified minimum/maximum tensile strengths

Specified Minimum Tensile Strength MPa	**Specified Maximum Tensile Strength MPa**
Up to 1000	Up to 1100
Over 1000 up to 1400	Over 1100 up to 1450
Over 1400 up to 1800	Over 1450 up to 1800
Over 1800	Over 1800

4.2.2 Tensile Strength/Hardness Relationship

If no maximum or minimum tensile strength is specified for the steel, hardness values of 310 HV, 340 HV, 435 HV, 450 HV, and 545 HV (as measured in accordance with BS EN ISO 6507-1) shall be regarded as equivalent to 1000, 1100, 1400, 1450, and 1800 MPa respectively. Steels which have been wholly or partly surface hardened shall be considered as being in the category appropriate to the hardness of the surface layer.

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.

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 specification and metallurgical condition of the material of the item.

(c) Instructions concerning stress relieving heat treatment required including the limits of temperature and time to be employed.

(d) Instructions concerning post cleaning de-embrittlement which is required.

(e) When necessary, the cleaning method or methods given in this Standard which are to be used.

(f) Details of any particular requirements for special surfaces.

(g) Any special information regarding coatings to be removed, or coatings or inserts to be protected.

(h) Where appropriate the surface coating which is to be subsequently applied.

6. PROCESS CONTROL

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

6.2 Details of all processes and treatments, including all preparatory and post treatments, processing, significant surfaces, tests and all other processes and treatments shall be included in the Process Control Schedule.

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

7. GENERAL REQUIREMENTS

7.1 Any materials used for cleaning and preparation shall contain not more than: 5 ppm mercury (Hg). Where items can be regarded as susceptible to degradation by residual arsenic and/or antimony the following maximum limitations shall also apply: 15 ppm arsenic (As), 15 ppm antimony (Sb).

WARNING: The presence of arsenic, reduced sulphur, or reduced phosphorous compounds in an acid bath will promote absorption of hydrogen by steel and may reduce the adhesion of subsequently applied coatings.

NOTE: Contamination of steel components can occur when antimony inhibited solutions are used. The resultant deposit of antimony on exposed surfaces can adversely affect the properties of the steel and subsequent processing of the item. If processing solutions become contaminated this can lead to further items being similarly affected.

7.2 When assembled items are being processed the treatments chosen must be suitable for each constituent material and combination of materials.

7.3 Items will often require a sequence of degreasing and cleaning treatment and solvent cleaning may occasionally leave traces of soil after evaporation of the solvent, care shall therefore be taken to avoid this. Inorganic contamination may not always be removed by solvent processes therefore it may be necessary to use aqueous processes in addition after final degreasing and cleaning (see Section 6 regarding process control).

7.3.1 The surfaces shall be free from grease, oil, oxide, scale or other foreign matter and shall be in a chemically clean condition (i.e., fully receptive to any subsequent processing). To test for surface cleanliness the cleaned surface may be sprayed or immersed in cool, clean water. On clean surfaces, the water will form a uniform film, in the presence of oily soils, the water film will break away from contaminated areas. In cases of gross contamination, the entire film may break down into discrete globules. If the surface to be tested has any residue of cleaning agent containing surface active agent, a continuous film may be obtained even in the presence of grease. This false effect shall be overcome by dipping the surface in dilute acid and rinsing before carrying out the test. Subsequent specified treatments shall be applied without delay.

7.3.2 The mechanical properties shall not be impaired significantly except where they may be recovered by subsequent baking or other treatment which forms part of the normal processing sequence.

7.3.3 The dimensions and surface finish shall be such as to enable the finished (e.g., electroplated) items to be within the required drawing or specification limits.

7.4 Where solvent cleaning is used, corrosion of items, particularly those made in light alloys, may occur by subjecting them to heat before all solvent has disappeared from the surfaces, therefore, solvents which thermally degrade with the liberation of corrosive products shall not be used. Care is to be taken, therefore, to ensure that items of complicated form, or containing blind holes, have all trace of liquid removed. Bundles of items intended for immediate heat treatment are to be shaken to ensure that liquid is not retained by capillary attraction between surfaces in contact. In any event, items shall not be immersed in a molten salt bath until they are completely dry and have reached room temperature after removal from the degreaser.

NOTE: It is not good practice to allow an interval before treatment, it must be remembered that degreasing and cleaning produce a surface whose resistance to corrosive attack is low. Consequently, readily corrodible materials must be protectively coated or further processed.

7.5 Immediately after each treatment in an aqueous solution the items shall be thoroughly rinsed in clean hot and/or clean cold water, as appropriate, paying special attention to items containing crevices. Immediately after the final rinsing the metal surface shall not exhibit a water break. Water used for final rinsing after processing shall be discarded or retreated when the conductivity exceeds 10 mS/m (100 µS/mm).

7.6 After the final rinsing or preparatory treatment, items to be given a protective treatment in aqueous solutions shall be transferred immediately without drying to the treatment bath.

7.7 Items to be given further treatment by dry processes and items which are not to be subjected to further processing, shall be thoroughly dried.

7.7.1 Where drying in a draught of warm air is recommended, the air shall be free from oil or other contaminants.

7.7.2 The use of chlorinated solvents for drying purposes is not permitted.

7.8 The acid and alkaline solutions used in some of the cleaning and preparation processes can have a deleterious effect on explosives. When an item is for use with explosive materials, particular care must be taken to ensure that all such solutions are completely removed by thorough washing with clean water. Special attention should be paid to irregularly shaped items and to blind holes and crevices.

7.9 Where limits of composition of solutions are known they are quoted in this Standard. Where limits are not quoted other proportion may be used provided they have a tolerance of not more than ±10%.

7.10 Where a surface is cleaned and pretreated, at least the first coat of the paint system shall be applied in conformity with the manufacturer’s instructions as soon as possible but within 16 hours. During this period the surface shall be kept under clean, dry conditions.

8. STANDARD CLEANING METHODS

8.1 Throughout this Standard the processes and methods in brackets which follow references to cleaning treatments refer to the processes and methods in Annex J.

8.2 The efficiency of some of the methods may be increased by application of ultrasonic agitation, Process A (Method A6). But care should be taken to avoid damage to delicate items and assemblies such as electrical or electronic equipment and semi-conductor devices.

9. NOTES

9.1 A change bar (l) 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 - STEELS, NON-CORROSION RESISTING, CAST IRONS AND PURE IRONS

A.1 SELECTION OF METHOD OF CLEANING

The choice of cleaning methods for steels of specified maximum tensile strength over 1000 MPa is restricted by the requirements of Section A.2 in order to avoid embrittlement by hydrogen absorption during cathodic or acid treatments. In general the methods given are suitable for both steel and iron. Treatment of cast iron in acid solutions is, however liable to set up preferential etching around the graphite which may become detached leaving holes which may entrap treatment solution and give rise to subsequent staining or corrosion.

A.2 STRESS RELIEVING HEAT TREATMENT

A.2.1 Items made from steel of specified maximum tensile strength in the range of 1001 to 1450 MPa which have been ground or subjected to heavy machining after final tempering shall be stress relieved at a temperature between 200 and 230 °C for not less than 3 hours, or not more than 50 °C below the tempering temperature for 15 to 30 minutes. Any stress relieving treatment shall be applied after degreasing (Process A) and before any treatment liable to cause embrittlement (see Section A.1). Items which have been treated in order to introduce beneficial compressive stresses into any part of the surface, e.g., by shot peening or cold working, shall not be heated above 230 °C. Carburized surface hardened items shall be heated at 130 to 150 °C for not less than 6 hours.

A.2.2 Steels of specified maximum tensile strength over 1450 MPa are subject to the requirements of AMS03-4/Def Stan 03-4 for stress relief.

A.3 GENERAL DEGREASING AND CLEANING

A.3.1 Steels of Specified Maximum Tensile Strength up to 1000 MPa

Degrease to Process A (Method A1, A2) and Process B (Method B1, B2, B3) prior to the commencement of a cleaning sequence which shall be selected from Processes D to L as appropriate.

A.3.2 Steels of Specified Maximum Tensile Strength in the Range 1000 to 1450 MPa

Any form of cathodic cleaning treatment of these steels is prohibited. Degrease to Process A (Method A1, A2) and Process B (Method B1, B2, B3) prior to the commencement of a cleaning sequence which shall be selected from Processes D, F, H, and K1 as appropriate.

A.3.3 Steels of Specified Maximum Tensile Strength over 1450 MPa

The items shall be cleaned in accordance with the requirements of AMS03-4/Def Stan 03-4.

A.4 PREPARATION FOR ELECTROPLATING

Degrease to Process A (Method A1, A2) and Process B (Method B1, B2, B3) prior to the selection of a cleaning sequence from Section A.3 taking into account the tensile strength of the steel. If inhibited solutions are used for acid pickling (Process F) or de-rusting (Process J) it may be necessary to remove adsorbed inhibitor from the surface by acid dipping (Process G) or by a suitable alkaline solution (Process B) before chemical smoothing (Process L) and/or electroplating.

A.5 PREPARATION FOR ELECTROLESS NICKEL PLATING

A.5.1 For steels having a specified maximum tensile strength below 1100 MPa items shall be treated as follows:

A.5.1.1 Degrease to Process A (Method A1) and Process B (Method B1) prior to the commencement of a cleaning sequence which shall be in accordance with Processes D to F and K as appropriate.

A.5.1.2 Clean by periodic reverse current (finishing anodically) in an alkaline solution.

A.5.1.3 Acid dip in 10% by volume sulfuric acid (d = 1.84 g/ml).

A.5.1.4 Electroless nickel plate (BS EN ISO 4527).

NOTE 1 Some steels containing chromium and molybdenum may become passivated by anodic cleaning. For these steels, cathodic cleaning in place of cleaning by periodic reverse current may be employed.

NOTE 2 A warm rinse may be found desirable immediately before stage A.5.1.4 to pre-heat large items but care should be taken to ensure that the items do not dry off before being immersed in the electroless nickel solution.

A.5.2 For steels having a specified maximum tensile strength in the range 1100 to 1450 MPa the following procedure shall be carried out:

A.5.2.1 Degrease to Process A (Method A1) and Process B (Method B1) prior to the commencement of a cleaning sequence which shall be in accordance with Processes D, F, and K as appropriate.

A.5.2.2 Anodic etch (Process H) for between 1 and 3 minutes.

NOTE: This bath is liable to deterioration from frequent use. Therefore, care shall be exercised to monitor and maintain the bath in good condition or tarnishing will occur. Some steels containing chromium and molybdenum may become passivated by anodic cleaning.

A.5.2.3 Thoroughly rinse to remove all viscous sulfate solution from the surface.

A.5.2.4 Electroless nickel plate (BS EN ISO 4527).

A.6 PREPARATION PRIOR TO PHOSPHATING TO AMS03-11/DEF STAN 03-11

Degrease and clean to the Processes listed in Section A.3 taking into account the tensile strength of the steel. Abrasive cleaning (Process D) is preferred. If inhibited solutions are used for acid pickling (Process F) or de-rusting (Process J or K) it may be necessary to remove adsorbed inhibitors by an acid dip (Process G) or by a suitable alkaline solution (Process B) before rinsing and phosphating. Information on the effect of cleaning treatment solutions on the quality of phosphate coatings is given in AMS03-11/Def Stan 03-11.

A.7 PREPARATION PRIOR TO SHERADISING TO BS 4921

Degrease and clean to the Processes listed in Section A.3 taking into account the tensile strength of the steel.

A.8 PREPARATION PRIOR TO HOT DIP GALVANISING

A.8.1 Steel (other than castings)

Degrease by using Process A (Method A1, A2) and Process B (Method B1) prior to one of the following:

(a) Acid Pickle to Process F (Method F1 or F2). When necessary a flux coating shall be applied.

(b) Abrasive blasting with chilled iron or steel grit to Process D (Method D1) to remove welding slag or paint. After abrasive blasting, Process F (Methods F1 or F2) may be used and when necessary a flux coating shall be applied.

A.8.2 Steel Castings

Preparation shall be carried out using one of the following procedures:

(a) Abrasive blasting with chilled iron or steel grit to Process D (Method D1) to remove moulding sand and iron scale. After abrasive blasting degrease by using Process A (Method A1, A2) and Process B (Method B1) and clean by Process F (Method F1 or F2). When necessary a flux coating shall be applied.

(b) Hydrofluoric acid (30% HF) diluted 10 to 50 times by volume with water, followed by hosing with water to remove any gelatinous products. After rinsing, degrease by Process A (Method A1, A2) and Process B (Method B1). Clean by using Process F (Method F2); when necessary a flux coating shall be applied.

NOTE 1 High strength steels can be susceptible to hydrogen embrittlement during pickling.

NOTE 2 Stresses induced, e.g., by welding or hardening can result in grain boundary penetration of steels by zinc during hot dip galvanizing. Hence stress relieving prior to galvanizing should be considered.

A.9 PREPARATION PRIOR TO METAL SPRAYING

A.9.1 Spraying for Protection Corrosion

Spraying to BS 2569 for protection against corrosion and high temperature oxidation. Degrease to Process A (Method A1, A2) and Process B (Method B1) prior to abrasive cleaning Process D (Method D1) with suitable grits. The prepared surface shall be comparable in roughness with a reference surface produced in accordance with Appendix A of BS 2569 and shall provide an adequate key for the subsequent sprayed metal coating. Metal spraying shall immediately follow abrasive cleaning.

NOTE: For sheet steel of thickness up to and including 1.6 mm, some relaxation on the degree of roughness may be acceptable in order to minimize distortion during surface preparation. Distortion can be reduced by blast cleaning on both sides.

A.9.2 Spraying for Reclamation Purposes

Items shall be thoroughly degreased prior to the commencement of a cleaning sequence which shall be in accordance with the Processes and Methods in Section A.3 as appropriate to the requirements of the subsequent metal spraying process.

A.10 PREPARATION PRIOR TO DIRECT APPLICATION OF PAINT TO THE BARE METAL

Degrease to Process A (Method A4) and Process B (Method B1) prior to the selection of a cleaning sequence from Section A.3 taking into account the tensile strength of the steel. When Process D (Method D1, D2) is used for the removal of oxide, scale or rust, the entire surface shall show a blast cleaning pattern and shall be clean bare steel completely free from any contamination or discoloration.

ANNEX B - STEELS, CORROSION RESISTING

B.1 STRESS RELIEVING HEAT TREATMENT

Certain hardenable corrosion-resisting steels may need stress relieving after grinding or heavy machining. The heat treatment specified in A.2.1 shall be used, i.e., 200 to 230 °C for not less than 3 hours for steels having a maximum specified tensile strength up to 1450 MPa or not more than 50 °C below the tempering temperature for 15 to 30 minutes. Steels of specified maximum tensile strength over 1450 MPa are subject to the requirements of AMS03-4/Def Stan 03-4 for stress relief.

B.2 GENERAL DEGREASING AND CLEANING

B.2.1 Degrease prior to the selection of a cleaning sequence from Section A.3 taking into account the strength of the steel.

NOTE: Electropolishing or chemical polishing by a suitable method may be used.

B.3 PREPARATION PRIOR TO ELECTROPLATING

Degrease to Process A (Method A1) and Process B (Method B1) prior to the selection of a cleaning sequence from Section A.3 taking into account the tensile strength of the steel. The items shall then be given a nickel strike in accordance with Process V before electroplating with the metal(s) required.

NOTE: Modified methods, including the omission of the nickel strike, may be permitted provided that adequate adhesion will be obtained.

B.4 PREPARATION PRIOR TO ELECTROLESS NICKEL PLATING

B.4.1 The items shall be degreased and cleaned in accordance with Section 7 and Section A.5 taking into account the tensile strength of the steel, and then either given a nickel strike in accordance with Process V or striking electrolytically in the electroless nickel solution until the surface is covered.

NOTE: The surface may be activated by dipping for 1 to 2 minutes in 50% by volume hydrochloric acid (d = 1.16 g/ml) at a temperature of 40 to 50 °C.

B.4.2 After rinsing in water the items shall be transferred directly to the electroless nickel bath.

B.5 PREPARATION PRIOR TO METAL SPRAYING

B.5.1 Spraying to BS 2569

Cleaning and preparation prior to metal spraying shall be carried out as for steels, non-corrosion-resisting.

B.5.2 Spraying for Reclamation Purposes

Items shall be thoroughly degreased prior to the commencement of a cleaning sequence which shall be in accordance with the Processes and Methods in Section A.3 as appropriate to the requirements of the subsequent metal spraying process.

B.6 PREPARATION PRIOR TO THE DIRECT APPLICATION OF PAINT TO THE BARE METAL

Degrease to Process A (Method A1) and Process B (Method B1) prior to the selection of a cleaning sequence from Section A.3 taking into account the tensile strength of the steel. When Process D (Method D1, D2) is used for the removal of oxide, scale or rust, the entire surface shall show a blast cleaning pattern and shall be completely free from contamination or discoloration. For abrasive cleaning non-metallic grits shall be used.

B.7 PASSIVATION PRIOR TO PUTTING INTO SERVICE

B.7.1 The durability of corrosion resistant (stainless) steels is based on the presence of an extremely thin “passive layer” of a chromium-rich oxide on the component surface. This passive layer forms spontaneously in air and many aqueous environments but its quality and thickness can be improved by a final passivation treatment prior to entering service. It is generally considered that some passivation treatments will also remove light surface contamination. For effective passivation, however, the steel surface must not have depleted chromium concentration and must be free from oxide scale and many other surface defects produced during fabrication.

B.7.2 Where passivation is called for, Process M shall be used

ANNEX C - ALUMINUM AND ALUMINUM ALLOYS

WARNING: Etching or pickling processes may adversely affect fatigue strength and for certain high strength alloys may cause stress corrosion problems. In particular sodium hydroxide (Method P2) and pickling (Process N) can have a severe effect and shall only be used with discretion. The properties of certain heat treatable aluminum alloys may be adversely affected by prolonged treatment at or above 100 °C.

C.1 GENERAL DEGREASING AND CLEANING

C.1.1 Items shall be degreased and cleaned by the following methods as appropriate:

(a) Process A.

(b) Method B1 for degreasing unmachined or rough machined aluminum alloy forgings prior to final inspection.

(c) Method B2.

(d) Process D.

(e) Oxide removal treatment in chromic acid/sulfuric acid solution (Process O).

(f) Pickling to Process N.

(g) Etching in sodium hydroxide solution (Method P2).

NOTE: See warning note at head of Annex C.

(h) A chemical or electrolytic polishing treatment.

C.1.2 Drying by means of chlorinated solvents shall not be used.

C.2 REMOVAL OF CORROSION PRODUCTS

One of the following procedures shall be used.

(a) Immersion in chromic acid/phosphoric acid solution, Process U (Method U1), which results in least attack of underlying metal.

(b) Process O, Process P (Method P1) or Process D (excluding Method D1) to remove light corrosion products and soil.

(c) Process U (Method U2) or Process D (excluding Methods D1 and D4) to remove light corrosion products in situ.

NOTE: In some circumstances fine abrasive blasting may peen over pits and retain corrosion products.

C.3 PREPARATION PRIOR TO ANODIZING

Items shall be degreased and cleaned in accordance with Section C.1, to subject the requirements of Def Stan 03-24, AMS03-25/Def Stan 03-25, or BS 5599.

C.4 PREPARATION PRIOR TO PROCESS O OR FILMING TREATMENTS TO AMS03-18/DEF STAN 03-18

Items shall be degreased and cleaned in accordance with Section C.1.

C.5 PREPARATION PRIOR TO PAINTING

C.5.1 The items shall be degreased to Process A and Process B (Method B2) and then cleaned by the following Processes as appropriate.

(a) Process D.

(b) Process O.

(c) Process P.

C.5.2 Where pre-treatment primer (etching primer, e.g., Def Stan 80-15 or BS 2X32) is used, it shall be applied to the cleaned surface with the minimum delay and in any case within 16 hours of the item being degreased and cleaned. On items which have been anodized over part of the surface, an etch primer may be applied over the whole surface.

C.5.3 When etching (Processes O and P) is the final treatment before painting the paint primer shall be applied within 4 hours after drying.

C.6 PREPARATION PRIOR TO ADHESIVE BONDING OF UNANODIZED ITEMS

Items shall be prepared for bonding by degreasing, cleaning and etching (Processes O and P).

C.7 PREPARATION PRIOR TO ELECTROPLATING

As a preparation for the electrodeposition of any required finish, items shall be degreased, pickled, and given an immersion deposit of zinc followed by an electrodeposit of copper (Process W) or other metals.

C.8 PREPARATION PRIOR TO METAL SPRAYING (AMS03-3/DEF STAN 03-3)

The items shall be degreased and cleaned to Process A (Method A1, A2) and Process B (Method B2) followed by coarse abrasive blasting Process D (Method D1) using non-metallic abrasives.

NOTE: See Def Stan 08-39 for the metal spraying of aluminum armour and related materials.

ANNEX D - COPPER AND COPPER ALLOYS

D.1 GENERAL DEGREASING AND CLEANING

D.1.1 Alloys susceptible to stress corrosion cracking shall not be cleaned in acidic or ammoniacal media. De-greasing, prior to stress-relief, is permitted in stabilized chlorinated hydrocarbons, or in an aqueous alkaline solution. After stress relief, these alloys shall be degreased and cleaned in accordance with the following:

D.1.2 Items shall be degreased and cleaned by the following methods as appropriate.

(a) Degreasing in an organic to Process A.

(b) Degreasing in an aqueous solvent alkaline solution to Process B.

(c) Clean in an acid emulsion to Process C.

(d) Abrasive Cleaning to Process D (Methods D2, D3, or D4), Abrasive Cleaning Process D (Method D1) for castings only.

(e) Cleaning to Process Q.

(f) Etching or polishing by a chemical or electrolytic treatment.

D.1.3 Drying by means of chlorinated solvents shall not be used.

D.2 PREPARATION PRIOR TO ELECTROPLATING

D.2.1 The items shall be degreased and cleaned by the following Processes and Methods as appropriate:

(a) Degreasing in an organic solvent to Process A.

(b) Degreasing in an aqueous alkaline solution to Process B.

(c) Clean in an acid emulsion to Process C.

(d) Abrasive Cleaning to Process D (Methods D2, D3, or D4), Abrasive Cleaning to Process D (Method D1) for castings only.

(e) Clean to Process Q.

(f) Acid de-scale etch or smooth to Process R.

NOTE: Copper alloys containing aluminum and/or nickel may not be cleaned sufficiently using Method R and may require additional treatment (Process Q).

(g) Etching or polishing by a chemical or electrolytic treatment.

D.2.2 When electroplating in a cyanide solution is to be the next treatment, the cleaning procedure shall include acid pickling to Process Q or R (Method R1 or R2) and shall be followed by rinsing in water and immersion in a solution containing about 50 g/l of sodium or potassium cyanide.

D.2.3 Soft soldered items (other than beryllium copper alloy items) shall be degreased to Process A (Method A1, A2) and then cleaned by immersion for the minimum time necessary in a mild alkaline cleaning solution Process B (Method B2). This shall be followed by immersion in a solution containing approx. 100 ml/l fluoroboric acid (40% m/m HBF_4), rinsing thoroughly, and copper plating to a minimum thickness of 1 µm using a cyanide copper electrolyte (of the Rochelle salt-Cyanide type) having a pH 10 to 12 and operated at a temperature 60 °C ± 10 °C.

D.2.4 Beryllium copper items which are to be plated in an acid electrolyte shall be cleaned using Process Q (Method Q1) followed by rinsing in water and immersing in a solution containing 100 ml/l of sulfuric acid (d = 1.84 g/ml).

D.3 PREPARATION PRIOR TO ELECTROLESS NICKEL PLATING

Items shall be degreased and cleaned in accordance with Section D.2 and then treated as follows:

(a) Cleaned cathodically in a mild alkaline cleaner to Process B.

(b) Rinse (see 7.6).

(c) The surface catalyzed by one of four methods.

  (i) By striking in an electrolytic nickel bath (AMS03-27/Def Stan 03-27).

  (ii) By immersion in an activating dip, such as one containing palladium chloride, until the surface has darkened, showing deposition of palladium followed by rinsing and transferring to the electroless nickel bath. When proprietary solutions are used the directions of the suppliers shall be followed.

  (iii) By contacting the surface of the item with aluminum wire whilst immersed in the electroless nickel solution (or contacting with an already plated item) until the electroless nickel has spread over the entire surface.

  (iv) By applying an initial cathodic potential to the item immersed in the electroless nickel bath (BS EN ISO 4527).

D.4 PREPARATION PRIOR TO DIRECT APPLICATION OF PAINT TO BARE METAL

Where the protective treatment requires a paint to be applied to bare metal, the items shall be degreased and then cleaned by the Processes and Methods given in Section D.1 as appropriate.

ANNEX E - NICKEL ALLOYS

E.1 GENERAL DEGREASING AND CLEANING

The items shall be degreased and cleaned by the following methods as appropriate:

(a) Degrease in an organic solvent to Process A (Method A1, A2).

(b) Degrease in an aqueous alkaline solution to Process B.

(c) Clean in an acid emulsion to Process C.

(d) Abrasive cleaning to Process D.

(e) Sodium hydride de-scaling to Process E.

(f) Pickling in an acid solution to Process T.

(g) A chemical or electrolytic etching process acceptable.

E.2 PREPARATION PRIOR TO ELECTROPLATING

The items shall be degreased and cleaned by the Processes and Methods given in Section E.1 and Process X as appropriate.

E.3 PREPARATION PRIOR TO DIRECT APPLICATION OF PAINT TO BARE METAL

Where the protective treatment requires a paint to be applied to bare metal, the items shall be degreased and then cleaned by the methods given in Section E.1 as appropriate.

ANNEX F - TITANIUM AND TITANIUM ALLOYS

F.1 GENERAL DEGREASING AND CLEANING

Items shall be degreased and cleaned by one or more of the following methods as appropriate.

F.1.1 Degreasing in an Organic Solvent (Process A)

Certain chlorinated hydrocarbon solvents (including those in paint stripping agents) may cause stress corrosion cracking of some titanium alloys and the following precautions shall be observed when these solvents are used:

(a) Stressed items made of titanium and titanium alloys shall not be exposed to hot chlorinated solvents.

(b) The solvents shall be maintained clean and the accumulation of free acid shall be prevented in accordance with the supplier's operating instructions.

(c) Where hot trichloroethylene or hot perchloroethylene are the solvents used as liquid and/or vapor the time of immersion shall not exceed 30 minutes for any one decreasing operation.

(d) Care shall be taken to remove all traces of solvent from the item immediately after cleaning and before any further operation is performed.

F.1.2 Degreasing in an aqueous alkaline solution (Process B)

WARNING: Cathodic cleaning must not be used.

F.1.3 Clean in an acid emulsion to Process C.

F.1.4 Abrasive cleaning by wet methods only to Process D.

F.1.5 De-scaling by a suitable process.

F.1.6 Pickling in nitric acid/hydrofluoric acid solution to Process S.

F.2 PREPARATION PRIOR TO ELECTROPLATING

Titanium and titanium alloys are difficult to electroplate. A recommended preparatory treatment is given in Process Y.

F.3 PREPARATION PRIOR TO ELECTROLESS NICKEL PLATING

The items shall be prepared as for electroplating (see Section F.2). After the nickel strike, the items shall be thoroughly rinsed and transferred to the electroless nickel solution.

F.4 PREPARATION PRIOR TO DIRECT APPLICATION OF PAINT TO BARE METAL

Where the protective treatment requires a paint to be applied to bare metal, the items shall be degreased and then cleaned by Process D (see F.1.2(b)) or Process S, before application of the protective coating.

ANNEX G - MAGNESIUM ALLOYS

G.1 GENERAL DEGREASING, CLEANING AND PAINTING

Items shall be degreased, cleaned and painted in accordance with the requirements of DTD 911.

NOTE: Although DTD 911 has been declared obsolete the technical information within the document remains valid.

ANNEX H - ZINC BASED ALLOYS

H.1 GENERAL DEGREASING AND CLEANING

Unless otherwise required, e.g., by BS 6338 degreasing shall be carried out to Process A (Method A1) and Process B (Method B2). Zinc base alloys are normally in the form of pressure diecastings for which the final finish would be chromate passivation to BS 6338. Diecast items frequently contain sub-surface porosity and any process such as acid etching which penetrates the non-porous skin, may reveal this porosity with consequent entrapment of corrosive residues. Such processes shall therefore be avoided during all stages of manufacture.

H.2 CLEANING OF CORRODED ITEMS

Immersion in a solution containing about 100 g/l of chromic acid at 90 °C. Corrosion of pressure diecast zinc alloys during storage or service may reveal sub-surface porosity. As described in Section H.1, any cleaning process leaving corrosive residues shall be avoided.

H.3 PREPARATION PRIOR TO PAINTING

Prior to painting, degrease to Process A (Method A1) and Process B (Method B2). Clean if appropriate by Process D (excluding Method D1). Unless the surface is to be chromate passivated in accordance with BS 6338, treat with Paint, Pre-treatment Primer (Etching Primer) complying with Def Stan 80-15 or BS 2X32 prior to the application of the specified paint scheme. The pre-treatment primer shall be omitted if the surface has been chromate passivated.

H.4 PREPARATION PRIOR TO ELECTROPLATING

A preparatory treatment is given in Process Z.

ANNEX I - TUNGSTEN ALLOYS

I.1 PREPARATION PRIOR TO ELECTROPLATING

I.1.1 Items shall be degreased to Process A (Method A1) and Process B (Method B1) and cleaned by anodic treatment at a current density of 500 A/m^2 for 2 minutes in the following solution at ambient temperature:

Sodium hydroxide (NaOH) 200 g/l

Sodium cyanide (NaCN) 100 g/l

Then wash in cold running water.

I.1.2 Effective anodic treatment can also be carried out at lower current densities and for proportionally longer times, e.g., 200 A/m^2 for 5 minutes.

I.2 PREPARATION PRIOR TO ADHESIVE BONDING AND PAINTING

Prior to adhesive bonding and painting, degrease to Process A (Method A1) and Process B (Method B2). Clean to Process D. Specialist advice may be required for some alloys.

ANNEX J - CLEANING AND PREPARATION METHODS

J.1 INTRODUCTION

The processes detailed in the following pages are to be used as called for in the preceding annexes of this standard. Throughout these processes the terms ‘cold’ or ‘room temperature’, ‘warm’ and ‘hot’ are taken to mean temperatures of the order of ‘less than 25 °C’, ‘35 to 45 °C’ and ‘greater than 55 °C’, respectively.

J.2 PROCESS A - ORGANIC SOLVENT DEGREASING AND CLEANING

Method A1 - Hot Solvent, Not Water-Rinsable

(i) This method covers liquid and vapor degreasing in hot halogenated hydrocarbon solvents. The grades of solvent shall be known to be stable for this use. Subject to the restrictions laid down in Section F.1 for titanium the use of the following halogenated solvents is permitted:

Trichloroethylene ("Neutral" Type)

Perchloroethylene

NOTE 1 Other solvents used must not have an adverse effect on the metal. Attention shall be paid to the heat capacity of the component, e.g., heavily contaminated thin sheet would not degrease satisfactorily in vapor.

NOTE 2 Trichloroethylene will require a water separator.

NOTE 3 Perchloroethylene has a higher boiling point than trichloroethylene and may be more suitable for the removal of high melting point waxes.

(ii) Items of aluminum, magnesium or titanium shall be free from swarf.

(iii) Outline guidance on maintenance of organic solvent and vapor degreasing baths is given in Appendix W.

NOTE: Trichloroethylene and perchloroethylene shall not be used on assemblies containing fabric, rubber, paint or similar materials.

Method A2 - Cold Solvent, Not Water-Rinsable

(i) These cleaners are normally used by immersion, spray, or by hand application. The choice of solvent is largely governed by considerations of toxicity, volatility and flammability. Suitable cleaners include:

Trichloroethylene ("Neutral" Type)

Perchloroethylene

White Spirit (BS 245)

White Spirit/Solvent Naphthas

(ii) Items of aluminum, magnesium or titanium shall be free from swarf.

(iii) When the items are degreased by immersion in the solvent, at least three successive baths of solvent shall be used.

(iv) When hand application is used the solvent shall be applied by brushing or spraying and the solvent allowed to flood the work. When the solvent becomes contaminated with grease it shall be replaced. Outline guidance on maintenance of organic solvent baths is given in Appendix W.

Method A3 - Hot Solvent, Water-Rinsable

This method is used as both a degreasant and to remove paint and carbonaceous deposits. Material is based on a mixture of cresylic acid and O-dichlorobenzene. It is used with a water seal in a bath operated in accordance with the manufacturer's instructions.

Method A4 - Cold Solvent, Water-Rinsable

Dichloromethane based mixtures. These are mainly used for the removal of paint and carbonaceous deposits but may also act as a degreasant. Some materials contain contains cresylic acid for increased efficiency and is used with a water seal in a bath, operated at ambient temperatures. Application type (brushing) dichloromethane based mixtures are unsuitable as degreasants.

Method A5 - Petroleum Fuel Based Mixtures Containing Emulsifying Agents

These may be used for the substantial removal of heavy deposits of oil or grease. Application is normally by brush or spray with subsequent water rinsing. Petroleum fuel (BS 2869, Class Cl or C2) with an emulsifying agent (Def Stans 68-188 and 68-189) are suitable materials. For complete removal of all residual contamination these processes shall be followed by Process B.

Method A6 - Ultrasonic Cleaning

Ultrasonic cleaning can be used as a supplement to solvent cleaning to remove solid contaminants that may be particularly tenacious or which are present in deep recesses or other areas difficult to reach. The simplest process is two stage consisting of initial cleaning in a solvent with ultrasonic agitation followed by vapor immersion. A three stage process in which the initial cleaning is carried out by vapor immersion may be used where contamination is particularly severe. The level of maintenance required for organic solvent and vapor degreasing baths should also be given to ultrasonic cleaning systems and there should be similar provision for water separation where chlorinated solvents are used.

The Solvent Emissions Regulations 2004 – Note shall be made of the content of these regulations in the determination of emissions limits.

The Solvent Emissions Regulations (SER) has been introduced to further limit the emissions of volatile organic compounds due to the use of organic solvents in certain activities and installations.

The Solvent Emissions Regulations came into force in January 2004 and gave effect to European Directive 199/13/EC commonly referred to as the Solvent Emissions Directive (the SED).

There are two main compliance options:

- Meeting a VOC **emission concentration limit** and fugitive emission limits and submitting annual or continuous monitoring results depending on the size of emissions (limit values in SED Annex 11A); or

- Using a solvent reduction approach to achieve the results that would be obtained from meeting a mass emission limit **(reduction scheme)**. This approach is not to be employed where certain risk phrase compounds are used (methodology in SED Annex 11B).

In some cases (notably the coatings manufacturing and pharmaceuticals sectors) there is also the option of meeting a mass emission limit (total emission limit value).

J.3 PROCESS B – ALKALINE DEGREASING

This method may be used for complete degreasing or in addition to preliminary solvent degreasing. It has advantages over solvent degreasing in the removal of certain types of contaminants including soaps and salts. The cleaning action is based on the saponifying and emulsifying effects of aqueous alkalis, often reinforced by sequestrating, completing and surface active agents. The ingredients are usually selected from sodium hydroxide, sodium carbonate, sodium metasilicates, trisodium phosphate, sodium pyrophosphate, sodium borates, complexing agents (such as EDTA gluconates, heptonates, polyphosphates, and cyanides) and organic surfactants. They may be used hot or cold with or without applied current which may be either anodic or cathodic (but see A.3.2 and A.3.3). Very thorough washing is required to remove residual traces of alkaline cleaners from metal surfaces. The surfaces of metal items which have been alkali cleaned and which are to be painted without pre-treatment shall first be neutralized by means of an acid dip, of 2.5%(m/v) chromic acid plus 2.5%(v/v) phosphoric acid (d = 1.75 g/ml), then rinsed and dried before painting (see AMS03-4/Def Stan 03-4 for high strength steels).

Method B1 - General Purpose Alkaline Degreasinq

These may contain any of the constituents given in the preceding paragraph. Heavy duty types, e.g., Interim Def Stan 68-160, may contain a high proportion of sodium hydroxide. They are suitable for use on all metals except aluminum, lead, tin and zinc, each of which is attacked by strong alkalis. For these metals pickling in sodium hydroxide or other alkaline solutions shall only be employed with discretion and shall be strictly controlled.

Method B2 - Mild Alkaline Degreasinq

They shall be free from sodium hydroxide or other strong alkali and shall be inhibited to avoid attack on metal surfaces.

(i) Sodium metasilicate types: These shall include at least 25% by m/m of sodium metasilicate.

(ii) Silicate free types: These are based on alkaline phosphates and organic surfactants with the addition of buffers if required.

They are used hot or cold and silicate free types can be used with or without current (see A.3.2 and A.3.3). Some suitable formulations for mild alkaline cleaners are specified in Interim Def Stan 68-160.

Method B3 - Cathodic Alkaline Degreasing

Other approved aqueous degreasers not chemically inhibited may be used with cathodic current, thus avoiding attack on the metal (but see A.3.2 and A.3.3).

J.4 PROCESS C – ACID EMULSION CLEANING

For the removal of ingrained organic contamination, e.g., on cold-rolled material and in instances where organic contamination is slight, mineral acids containing 2 to 5% of non-ionic surface active agents are allowed provided the use of the mineral acid is not contrary to other sections in this Standard.

J.5 PROCESS D - ABRASIVE CLEANING

WARNING: Residual abrasive on treated items may interfere seriously with subsequent treatment and should therefore be removed.

Method D1 - Coarse Abrasive Blasting

This is carried out by blasting with chilled iron grit, alumina or other coarse abrasive of a grit size covered by, or comparable with, Grade G170 of BS EN ISO 11124, or larger. The process is unsuitable for thin material or where a fine surface profile is required. On corrosion resisting steels and on aluminum and aluminum alloys only iron-free and lead-free alumina grit (BS EN ISO 11126-7) shall be used. When used prior to metal spraying the surface shall be blasted in accordance with the requirements of BS 2569 for steel and AMS03-3/Def Stan 03-3 for aluminum alloys. This method is also useful for cleaning corroded ferrous items.

Method D2 - Fine Abrasive Blasting

This method is similar to Method D1 but employs non-metallic or metallic abrasives with or without water in such a way as to produce a finer surface profile than that produced by Method D1. Where chilled iron grit is used for non-corrosion resisting steels it shall be grade 070 or finer of BS EN ISO 11124.

Method D3 - Scourinq

This is carried out by rubbing or scrubbing with moistened pumice, alumina, or other non-metallic abrasives. Silicon carbide abrasive is not recommended for use on aluminum and aluminum alloys.

Method D4 - Tumbling

Tumbling should be carried out with a suitable abrasive in special plant. Corners and edges may become rounded or deburred in the process.

Method D5 - Wire Brushing

Brushing either by hand or mechanical methods may be used without supplementary abrasive. Compatibility between the brush material and the material being treated must be carefully considered, e.g., the use of non-stainless steel brushes must be restricted to non-corrosion resisting steels and cast-irons.

NOTE 1 When using Methods D1 or D2 on surfaces which are to be subsequently painted the resultant surface profile from the abrasive blasting must be compatible with the total dry film thickness of the specified paint system.

NOTE 2 The appropriate Parts of BS 7079 should be read in conjunction with Methods D1, D2, and D5.

J.6 PROCESS E – SODIUM HYDROXIDE DE-SCALING

This process is particularly suitable for castings, and for items in heat-resisting steel sheet stress relieved after fabrication. Unless strictly controlled, molten hydride is liable to embrittle titanium alloys and high strength steels by hydrogen absorption.

Preheat the items to 300 °C and immerse for up to 10 minutes in the following molten salt bath at 350 to 370 °C:

Sodium hydroxide	98% m/m
Sodium hydride	2% m/m

Remove from the molten salt bath and quench in cold running water and give a final rinse in hot water.

J.7 PROCESS F – ACID PICKLING WITHOUT ELECTROLYSIS

NOTE 1 Certain alloys containing sulfur or lead to promote free-machining properties may suffer coating adhesion problems unless pickled in acids containing fluorides (normally additions of ammonium bifluoride will suffice). Leaded steel items that are polished before plating are particularly at risk.

NOTE 2 When items to be subsequently chemically smoothed (process L), phosphated or electroplated following a pickling operation, it is necessary to follow the pickle with an acid dip (process G) to remove the passive surface. Immerse in one of the following aqueous solutions:

Method F1

Approx. 100 ml/l of sulfuric acid (d = 1.84 g/ml) with or without inhibitors or wetting agents. Use between 20 to 45 °C.

Method F2

100 ml/l of hydrochloric acid (d = 1.16 g/ml) with or without inhibitors or wetting agents. Use at room temperature.

Method F3

Phosphoric acid (d = 1.69 g/ml) shall be used at either the concentrations or temperatures stated below, with or without a wetting agent and a suitable inhibitor.

(1) 50% by volume; use at room temperature

(2) 30% by volume; use between 60 to 70 °C

The solution shall contain not more than:

(a) 2 g/l sulfate, calculated as sodium sulfate;

(b) 1 g/l chloride, calculated as sodium chloride;

(c) 8 ppm in total of arsenic and/or antimony (see 7.1).

Inhibitor may be added to the solution to reduce attack on the base metal. Any inhibitor shall be completely miscible in the solution and shall show no decomposition or separation on storage or dilution. The inhibitor shall pass the test for attack on mild steel given in Appendix Y. The test method is not applicable for other steels.

J.8 PROCESS G – ACID DIPPING

Immerse for not more than 2 minutes in one of the following uninhibited aqueous solutions.

Method G1

Sulfuric acid (d = 1.84 g/ml) 50 to 100 ml/l used warm or at room temperature.

Method G2

Hydrochloric acid (d = 1.16 g/ml) 50 to 100 ml/l used at room temperature.

J.9 PROCESS H – ANODIC PICKLING

Typical anodic pickling procedures are as follows:

Method H1 (preferred method suitable for all items)

Immerse the items in an aqueous solution (d = 1.36 g/ml) containing Sulfuric acid (d = 1.84 g/ml), 300 to 350 ml/l. The solution strength shall be maintained to keep a relative density greater than 1.30. Prior to the immersion in acid solution, soaking in an alkaline solution of sodium gluconate at up to boiling point assists the removal of scale.

Set the voltage so that the initial current density is not less than 1100 A/m^2 and is preferably twice this. The temperature shall not exceed 25 °C, but for removal of scale a temperature of up to 70 °C may be used. An inhibitor which is stable in the solution is allowed to reduce the formation of smut but wetting agents shall be avoided as they promote absorption of hydrogen. If the current density interruption method is used on steel of tensile strength of 1400 MPa and above, a safe procedure shall be established and incorporated in the Process Control Schedule.

Method H2

Immerse the items in an aqueous solution (d = 1.74 g/ml) containing Sulfuric acid (d = 1.84 g/ml), 750 ml/l with or without a small addition of chromic acid. The solution strength is maintained by periodic additions of sulfuric acid to keep the relative density greater than 1.70. An initial current density of at least 1000 A/m^2 (4 to 12 volts) should be applied. The temperature should not exceed 25 °C.

Method H3

An alternative aqueous solution (d = 1.22 g/ml) for use on corrosion-resisting steel prior to a nickel strike (Process V) shall contain Sulfuric acid (d = 1.84 g/ml), 200 ml/l. The solution strength is maintained by periodic additions of sulfuric acid to keep the relative density greater than 1.20. A current density of 2000 to 2500 A/m^2 (approx. 6 volts) should be applied. The temperature should not exceed 20 °C. Immersion time 1 to 3 minutes.

Notes on anodic pickling

(i) Contamination of these solutions with chloride shall be avoided.

NOTE: The essential requirement of anodic pickling of steel is to render the steel passive, a condition that will be indicated by a sharp rise in voltage between the item and the cathode, a corresponding fall in current and the onset of gas evolution from the item.

(ii) Passivity shall be maintained until the surface has acquired a light grey color, free from dark smut.

(iii) The items are allowed to be withdrawn for examination, and if necessary, replaced in the bath for further treatment.

NOTE: Anodic pickling may not be effective and may cause pitting on the inner surfaces of tubular items unless auxiliary cathodes are employed. Where it is desired to clean only the external surfaces, the item should be plugged to prevent ingress of the electrolyte.

J.10 PROCESS J – ACID DE-RUSTING

Rust may be removed by immersion in any of the solutions described in Process F.

This process shall not be applied to:

(i) steels of tensile strength above 1000 MPa (see 4.1)

(ii) springs in stress

(iii) items where rusting has rendered the components beyond repair

It is necessary to acid dip (Process G) the items before subsequent chemical smoothing (Process L), phosphating or electroplating.

J.11 PROCESS K – ALKALINE DE-RUSTING

Rust may be removed and the surface prepared for electroplating by one of the following methods:

Method K1 (For Light Rust)

Immersion in a solution based on caustic soda (BS 4130, Section 2, Type 2) and chelating agent such as sodium heptonate or gluconate.

Method K2 (For Heavy Rust)

Immersion in an electrolyte consisting essentially of sodium hydroxide in water. The solution may contain sodium cyanide or ethylenediamine tetracetic acid and wetting agents. Typical compositions are:

(i)	Sodium hydroxide (NaOH)	200 to 300 g/l
	Sodium cyanide (NaCN)	25 g/l
	Wetting agent	6 g/l
	Water 4.5 l	
(ii)	Sodium hydroxide (NaOH)	100 g/l
	Ethylenediamine tetracetic acid	100 g/l
	Non-ionic wetting agent	1.5 g/l
	Water 4.5 l	

The operating temperature is between room temperature and 60 °C. With solutions containing cyanide the operating temperature shall be below 40 °C to avoid decomposition of the cyanide. The items shall be made cathodic. In certain circumstances, periodic reverse current may also be used to enhance de-rusting. A source of direct current at approximately 10 to 12 volts is required and should be provided with a variable current control. Reasonable rates of de-rusting are obtained with current densities of the order of 2.5 to 5 A/dm^2.

The process shall not be applied to:

(i) steels of tensile strength above 1000 MPa,

(ii) assemblies containing aluminum or its alloys,

(iii) springs,

(iv) items susceptible to caustic embrittlement,

(v) items where rusting has rendered the items irreparable.

Method K3 (For Heavy Rust)

The K1 solution used electrolytically.

J.12 PROCESS L – CHEMICAL SMOOTHING PROCESS FOR STEEL

(i) Treat the items as follows:

(a) Degrease to Process A (Method A1) and/or alkaline clean to Process B (Method Bl).

(b) Acid dip to Process G (Method G1).

(c) Rinse thoroughly in cold water.

(d) Immerse in an aqueous solution containing:

Oxalic acid $((COOH)_2.2H2_2O)$	25 g/l
Hydrogen peroxide	13 g/l (e.g., 40 ml/l of ‘100 vols’ hydrogen peroxide)
Sulfuric acid (d = 1.84 g/ml)	0.053 ml/l

The solution shall be used at room temperature.

(ii) The time of treatment required varies between a few minutes or several hours depending on the application. The rate of metal dissolution, for mild steel, is approximately 10 µm per hour. The process is ineffective on corrosion-resisting steels and on low alloy steels containing more than about 1% chromium.

(iii) Hydrogen peroxide decomposes quite rapidly so additions equal to the original quantity need to be added for every 20 minutes of use.

J.13 PROCESS M – PASSIVATION OF CORROSION RESISTING STEELS

Such steels can be passivated in a number of aqueous solutions (see, for example, AMS2700C, BS EN 2516). The most commonly employed passivating solutions contain nitric acid (HNO_3), 20 to 50% used at 20 to 50 °C, with or without sodium dichromate. Increasing the nitric acid concentration and/or passivation temperature renders the solution more oxidizing and therefore should increase the passivation efficiency. Addition of sodium dichromate to the nitric acid also makes the solution more oxidizing. The most appropriate passivation solution chemistry and procedure is related to the type of the stainless steel. Method M1 (below) describes one generally suitable passivation treatment, which is based on nitric acid plus sodium dichromate. Method M2 is an alternative passivation treatment that does not utilize sodium dichromate.

Method M1

Immerse for a minimum of 20 minutes 49 to 54 °C in a solution made up to contain:

Nitric acid (specific gravity 1.42): 200 to 250 ml/l (20 to 25 vol %)

and

Sodium dichromate ($Na_2Cr_2O_7$ $2H_20$: 20 to 30 g/l

The treatment shall be followed by washing in clean water.

For ferritic, martensitic and martensitic precipitation hardening steels, the above process shall be followed by immersion for 30 to 40 minutes in a solution of 40 to 60 g/l sodium dichromate at 60 to 70 °C and final washing in cold water.

Method M2

Sodium dichromate is a hexavalent-chromium compound which poses health and safety problems and is subject to increasing regulation/restriction via International regulators such as “REACH” (Registration, Evaluation, Authorization and Restriction of Chemicals). An alternative passivation treatment that does not include this compound, and is suitable for austenitic, austenitic precipitation hardening and duplex stainless steels is as follows.

Immerse for 30 to 60 minutes at 20 to 30 °C in a solution of nitric acid (specific gravity 1.42), 200 to 500 ml/l (20 to 50 vol %)

NOTE: The article must be thoroughly clean before being passivated, i.e., free of grease and other debris (see Section A.3).

J.14 PROCESS N – ACID ETCHING OF ALUMINUM ALLOYS

(i) Aqueous solutions of the following approximate compositions are suitable for pickling degreased aluminum alloys and are approved for use prior to the final inspection of forgings:

Method N1

Sulfuric acid (d = 1.84 g/ml)	90 to 120 ml/l
Sodium fluoride (NaF)	7.5 to 15 g/l

Method N2

Sulfuric acid (d = 1.84 g/ml)	100 ml/l
Potassium fluoride (KF)	40 g/l

Method N3

Sulfuric acid (d = 1.84 g/ml)	100 ml/l
Hydrofluoric acid (40% m/m HF)	15 ml/l

Method N4

Orthophosphoric acid (d = 1.50 g/ml)	200 ml/l
Hydrofluoric acid (40% m/m HF)	7.5 ml/l

NOTE: Aluminum build up can influence the effectiveness of the bath.

(ii) Immerse the items in one of the above solutions at room temperature until uniformly clean. Rinse in cold water and transfer to a cold aqueous solution containing about 500 ml/l nitric acid (d = 1.42 g/ml) for about 1 minute and then thoroughly wash in clean water at a temperature not exceeding 50 °C.

J.15 PROCESS O – OXIDE REMOVAL TREATMENT FOR ALUMINUM AND ALUMINUM ALLOYS – BATH TREATMENT

This treatment may be used prior to painting, anodizing, filming, adhesive bonding and electroplating. It is not suitable for use on alloys containing 6% or more of copper or 2% or more of silicon and is generally unsuitable for use with composite items in which aluminum is in contact with other metals, particularly steel and copper alloys. Proceed as follows:

(i) Degreasing and Cleaning

The items shall be treated as follows:

(a) Degrease by Process A (Method A1, A2). Heavily soiled or small items may need more than one treatment.

(b) Alkaline degrease to Process B (Method B2). For items which are to be subsequently bonded, Process B (Method B2 - non-silicate type) shall be used.

(c) Rinse (see 7.5) and, where wet or dry abrasive blasting is not specified, transfer to the etching solution without drying.

(d) Where dry abrasive blasting is specified or where wet abrasive blasting is specified but does not immediately follow the rinsing operation, the items shall be dried. Care must be taken to protect the cleaned items from contamination.

(e) Items which have been dry abrasive blasted shall be treated with clean dry compressed air followed by alkaline degreasing to Process B (Method B2) (see (b)) and water rinsed (see 7.5) to remove all traces of blasting grit. They shall then be transferred to the chromic acid/sulfuric acid bath without drying.

(f) Items which have been wet abrasive blasted shall be water rinsed (see 7.5) to remove all traces of blasting grit and transferred to the chromic acid/sulfuric acid bath without drying.

NOTE: It is most important that all blasting grit be removed since its presence may impair the properties of the etched surface.

(ii) Immediately after final cleaning items shall be immersed in one of the solutions from Table J1.

(iii) Solution Maintenance

The solution shall be discarded when the specific gravity attains a value of 1.38 measured at room temperature, or when the solid materials begin to separate out on cooling the solution to room temperature or at the first signs of pitting.

NOTE: A fresh solution may be either aged with Al - Cu alloy scrap or aluminum sulphate or copper sulphate.

(iv) Rinsing

Rinse the items in running water with a temperature not exceeding 40 °C to prevent surface sealing effects (see also 7.5). Transport of items from the pickling bath to the rinsing bath shall be as rapid as possible to prevent drying spots on the surface.

(v) Drying

If no subsequent chemical treatments are carried out dry the items at a temperature not exceeding 65 °C. The surfaces shall not be touched with bare hands or be otherwise contaminated.

Table J.1 - Oxide removal solutions for aluminum and aluminum alloys

	Application	
Solution	**Adhesive Bonding or Paint Primer Application**	**As a Pre-Treatment for Anodizing and Other Chemical Treatments**
A		
Chromic acid (CrO_3)	45 to 55 g/l	30 to 55 g/l
Sulphuric acid (H_2SO_4)	220 to 300 g/l (1)	150 to 300 g/l (1)
Chloride (as NaCl)	< 200 mg/l	< 200 mg/l
Aluminum	1.5 to 14 g/l	1.5 to 14 g/l
Copper	0.06 to 1 g/l	0.06 to 1 g/l
Iron	< 1 g/l	< 1 g/l
Water (see (ii) (4))	Remainder	Remainder
Temperature range	60 to 65 °C	60 to 65 °C
Immersion time	28 to 30 minutes	20 to 30 minutes (2)
B		
Sodium bichromate ($Na_2Cr_2O_7.H_2O$)	67 to 83 g/l	45 to 83 g/l
Sulphuric acid (H_2SO_4)	220 to 300 g/l (3)	150 to 300 g/l (3)
Chloride (as NaCl)	< 200 mg/l	< 200 mg/l
Aluminum	1.5 to 14 g/l	1.5 to 14 g/l
Copper	0.06 to 1 g/l	0.06 to 1 g/l
Iron	< 1 g/l	< 1 g/l
Water (see (ii) (4))	Remainder	Remainder
Temperature range	60 to 65 °C	60 to 65 °C
Immersion time	28 to 30 minutes	20 to 30 minutes (2)

NOTE 1 H_2SO_4 : CrO_3 ratio shall be between 4.5:1 and 5.5:1 m/m.

NOTE 2 Shorter immersion times (less than 20 minutes) shall be used for items which are machined all over and which are not to be adhesive bonded.

NOTE 3 H2SO4 : Na2Cr2O7.H2O ratio shall be between 2.5:1 and 3.5:1 m/m.

NOTE 4 The solutions shall be prepared and maintained at operating level with water having a conductivity of not more than 10 mS/m and checked at regular intervals to determine conformance with the solution control limits specified.

(vi) Application of Bonding Primers or Adhesives

Adhesives or bonding primers shall preferably be applied immediately and in any case within 4 hours after drying, provided the treated items are stored under clean dry conditions.

J.16 PROCESS P – ETCHING TREATMENT OF ALUMINUM AND ALUMINUM ALLOYS

Method P1 - Brush or Spray Treatment

Where items are unsuitable for the immersion treatment, the treatment at Appendix Z shall be applied by brushing or spraying.

Method P2 - Sodium Hydroxide Treatment

(i) The etching and de-smutting method is used prior to anodizing if a polished finish is not required.

(ii) Immerse the degreased metal in the following solution:

Sodium hydroxide	25 to 50 g/l
Sodium heptonate or sodium gluconate	0.75 to 1.0 g/l

For a uniform, medium-deep etch, the concentration of free sodium hydroxide shall not fall below 25 g/l and that of aluminum shall not exceed 30 g/l. When the aluminum content of the solution approaches 55 g/l and free sodium hydroxide 40 g/l the solution shall be replaced.

NOTE: When the finish become brighter and more reflective, it indicates that the solution is nearly exhausted.

(iii) Operate the solution at 60 to 65 °C. The reaction is very vigorous and immersion times from 15 seconds to 1 minute are usually adequate.

(iv) De-smut by dipping in 30 to 50% by volume nitric acid (d = 1.42 g/ml). For high silicon-containing alloys 10% hydrofluoric acid (40% m/m HF) is added to the nitric acid de-smutting solution.

NOTE 1 A finer etch is produced at the upper end of the sodium hydroxide concentration range.

NOTE 2 If there is unavoidable delay in transferring from the de-smutting tank to the rinse, staining may occur.

NOTE 3 If a common rinse tank is used for etching and nitric acid de-smutting, corrosion of the items may occur.

J17. PROCESS O – CLEANING TREATMENT FOR COPPER AND COPPER ALLOYS

Method Q1

Immerse the items for up to 2 minutes at room temperature in an aqueous bichromate-sulfuric acid solution, a typical composition being:

Sodium dichromate ($Na_2 Cr_2 O_7 .2H_2 O$)	200 g/l
Sulfuric acid (d = 1.84 g/ml)	40 ml/l

Method Q2

Immerse the items in a solution containing:

Ferric sulfate liquor*	150 ml/l
Sulfuric acid (d = 1.84 g/ml)	50 ml/l

Use the solution at a temperature 20 to 50 °C.

*Ferric sulfate liquor containing approximately 640 g/l ferric sulfate.

J18. PROCESS R – ACID DE-SCALING, ETCHING AND SMOOTHING OF COPPER AND COPPER ALLOYS

Method R1 - 'Scale' Dip

Immerse the items in an aqueous solution containing:

Sulfuric acid (d = 1.84 g/ml)	100 ml/l

Use the solution at a temperature between 25 and 50 °C.

Method R2 - 'Bright' Dip

(i) This solution attacks copper base alloys vigorously and it is therefore not recommended for items having thin sections or close tolerances.

(ii) Immerse the items in an aqueous solution containing:

Sulfuric acid (d = 1.84 g/ml)	500 ml/l
Nitric acid (d = 1.42 g/ml)	250 ml/l
Sodium chloride or hydrochloric acid	1 g/l or 1 ml/l (may be omitted).

Use the solution at room temperature.

Method R3 - Chemical Smoothing Process for Copper and Copper Alloys

(i) Treat the items as follows:

(a) Degrease Process A (Method A1) and Process B (Method B2).

(b) Rinse thoroughly in cold water.

(c) Immerse in an aqueous solution containing:

Hydrogen peroxide	33 to 50 g/l (e.g., 100 to 150 ml/l of '100 vols' hydrogen peroxide)
Sulfuric acid (d = 1.84 g/ml)	1.3 ml/l

The solution should be at room temperature.

(d) Immerse for 20 seconds in a solution of dilute sulfuric acid Process R (Method R1).

(ii) The time of treatment required varies between 15 minutes to 1 hour depending on the application. The rate of metal dissolution depends upon the metal being treated and the solution temperature, but is in the order of 13 pm per hour. If the solution is used for continuous working the dissolved copper shall be removed by circulation through an ion exchange resin or by other suitable methods.

J.19 PROCESS S – PICKLING OF TITANIUM AND TITANIUM ALLOYS

Immerse the items in an aqueous solution having one of the following nominal compositions:

Method S1

Hydrofluoric acid (40% m/m HF)	Not more than 50 ml/l
	Not less than 40 ml/l
Nitric acid (d = 1.42)	Not less than 200 ml/l

The solution should be used at room temperature and shall not exceed 30 °C.

Method S2

Hydrofluoric acid (40% m/m HF)	Not more than 120 ml/l
Nitric acid (d = 1.42 g/ml)	Not less than 400 ml/l

The solution should be used at room temperature and shall not exceed 30 °C.

NOTE: Removal of scale may be aided by prior immersion in the alkaline permanganate solution referred to in Method T3.

J.20 PROCESS T – PICKLING OF NICKEL ALLOYS

Immerse the items in an aqueous solution having one of the following nominal compositions:

Method T1

Hydrofluoric acid (40% m/m HF)	50 ml/l
Nitric acid (d = 1.42 g/ml)	Not less than 200 ml/l

Use the solution at a temperature not exceeding 65 °C.

Method T2

Hydrofluoric Acid (40% m/m HF)	125 ml/l
Iron (111) Sulfate ($Fe_2(SO_4)_3.6H_2O$)	250 g/l
(or Iron (111) Sulfate ($Fe_2(SO_4)_3$))	200 g/l

Use the solution at a temperature of 65 to 70°C.

NOTE: Solutions in Methods T1 and T2 may promote preferential attack on nickel alloys unless the alloys are in solution heat-treated condition.

Method T3

Sodium hydroxide	200 g/l
Potassium permanganate	150 g/l

Use the solution at a temperature greater than 55 °C in a steel tank.

After rinsing, manganese oxides, etc., shall be removed by immersion in an acid solution such as a 10% sulfuric acid solution.

J.21 PROCESS U – REMOVAL OF CORROSION PRODUCTS FROM ALUMINUM ALLOYS

Method U1 (Immersion)

Immerse the items in a gently boiling aqueous solution of the following composition:

Chromic acid (CrO_3)	7.5 to 10 g/l
Phosphoric acid (d = 1.75 g/ml)	5 to 7.5 ml/l

NOTE: Twenty minutes immersion is normally adequate for sheets, and up to 1 hour for castings.

Method U2 (Swabbing)

Swab the items with a solution of 50 g/l chromic acid in water and rinse thoroughly in clean water.

J.22 PROCESS V – PREPARATION OF CORROSION-RESISTING STEELS PRIOR TO ELECTROPLATING

(i) Apply one of the following (Nickel Strike) treatments:

(a) Pickle the items anodically (Process H) and then treat cathodically for 5 minutes at 1600 to 2200 A/m^2 in an aqueous solution containing approximately:

Nickel sulfate ($NiSO_4.6H_2O$)	225 g/l
Sulfuric acid (d = 1.84 g/ml)	27 ml/l

Maintain the temperature of the electrolyte at 35 to 40 °C

Nickel or lead anodes shall be used.

(b) Make the items anodic in an aqueous solution of the following approximate composition for not more than 2 minutes, then reverse the current so that they are cathodic, for 6 minutes:

Nickel chloride ($NiCl_2.6H_2O$)	240 g/l
Hydrochloric acid (d = 1.16 or 1.18 g/ml)	85 ml/l

Use depolarized nickel electrodes to BS 558 (without bags). Maintain the solution at room temperature and apply a current density of 300 A/m^2 to the items.

(c) When current reversal is not feasible, the short anodic treatment (in (b) above) may be replaced by immersion in the solution without current flow for 15 minutes, the work then being made cathodic for 6 minutes.

NOTE: Separate tanks may be used for the anodic (or immersion) and the cathodic treatments.

(ii) After treatment (a), (b), or (c) rinse the items and transfer them to the final plating bath.