

Code of practice for

Cleaning and preparation of metal surfaces

Committees responsible for this British Standard

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Agricultural Engineers' Association
 Association of Manufacturers of Domestic Electrical Appliances
 British Steel Industry
 British Steel plc
 Institute of Metal Finishing
 Institute of Vitreous Enamellers
 International Tin Research Institute
 Metal Finishing Association
 Metal Sink Manufacturers' Association
 Ministry of Defence
 Vitreous Enamel Development Council
 Welding Institute

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Foreword

This British Standard has been prepared under the direction of the Materials and Chemicals Sector Board. It supersedes CP 3012:1972 which is withdrawn.

As a code of practice, this British Standard takes the form of guidance and recommendations. It should not be quoted as if it were a specification and particular care should be taken to ensure that claims of compliance are not misleading.

The standard is divided into sections. Section 2 gives recommended practice for cleaning a variety of metals and alloys prior to the application of comprehensive range of coatings (or when no subsequent coating is required). Full details of those methods recommended in section 2 are given in section 3. The designation of the methods conforms alphabetically to that given in DEF STAN 03-2/1 [1]. It is noteworthy, however, that this standard includes a number of processes not referred to in DEF STAN 03-2/1 as they are not generally used in the defence field and vice versa.

The cleaning and preparation of iron and steel prior to painting is already covered by BS 6150:1991, BS 5493:1977 and the various Parts of BS 7079.

It should be emphasized that the methods given for the treatment of metal are indicative of those most commonly used in practice. There are, in addition, numerous proprietary processes and other solution formulations available.

CAUTION. Some of the chemicals and processes referred to in this standard are hazardous to health. If the contractor is unfamiliar with the hazards which may be associated with any material or process, he should consult the supplier.

Nothing in this standard will relieve a contractor of his responsibilities with regard to the provision of protective clothing and the safety of his operations.

Attention is drawn to the appropriate government regulations including the Health and Safety at Work Act etc., 1974 [2] and COSHH Regulations 1988 [3] regarding protective clothing, breathing equipment, clean air and safety.

WARNING. In particular, great care should be taken in handling hydrofluoric acid. It is essential that the manufacturer's instructions be followed implicitly as burns due to contact with the acid may be apparent only after several hours.

NOTE Although it is not part of this Committee's purview, inevitably a number of processes covered by this code of practice are suitable for the preparation of metal surfaces prior to painting.

Attention is drawn to the replacement of the old nomenclature "*w/w*" by "*m/m*" in this standard.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 30, an inside back cover and a back cover.

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

Section 1. General

1.1 Scope

This British Standard gives recommendations for processes for the cleaning of metal surfaces to remove any extraneous or undesirable materials or deposits at any stage of manufacture. Such cleaning processes are also suitable for cleaning prior to adhesive bonding (see also BS 5350-A1:1976), brazing and non-destructive testing. Appropriate stress relieving treatments are also included.

NOTE 1 While some of the methods given might be applicable to the preparation of surfaces before welding, the recommendations regarding stress relief in Annex A do not apply to parts which are to be welded.

NOTE 2 Specific advice should be sought from the purchaser about the treatment of individual products and materials including the need for stress relieving heat treatment, and particularly where the shape, quantity and size will influence the choice of procedures to be adopted.

NOTE 3 For practices applicable to high strength steel bolts special concern is given to minimizing or eliminating the risk of embrittlement. Relevant requirements are stated in BS 7371-1.

This British Standard also covers the processes which are necessary for preparing metals prior to the application of the following surface coatings:

- a) electrodeposited metal coatings;
- b) autocatalytic metal coatings (sometimes referred to as electroless);
- c) conversion coatings;
- d) anodic oxidation coatings;
- e) hot dipped coatings;
- f) sprayed metal coatings;
- g) diffusion coatings;
- h) vitreous and porcelain enamel coatings;
- i) ceramic coatings;
- j) vacuum deposited coatings;
- k) impacted metal coatings.

NOTE 4 This British Standard does not cover in detail the cleaning and preparation of metal surfaces prior to painting although some general recommendations are given where appropriate.

Section 3 of this standard describes methods for the following treatments:

- 1) degreasing;
- 2) descaling;
- 3) pickling;
- 4) etching;
- 5) derusting;
- 6) chemical smoothing;
- 7) chemical polishing;
- 8) electrobrightening;
- 9) electropolishing;
- 10) fluoride anodizing process for cleaning magnesium alloys.

1.2 References

1.2.1 Normative references

This British Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are made at the appropriate places in the text and the cited publications are listed on the inside back cover. For dated references, only the edition cited applies; any subsequent amendments to or revisions of the cited publication apply to this British Standard only when incorporated in the reference by amendment or revision. For undated references, the latest edition of the cited publication applies, together with any amendments.

1.2.2 Informative references

This British Standard refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

Section 2. General cleaning of metal surfaces

2.1 Information to be supplied to the processor

The following information should be given on the drawing or order, when appropriate:

- a) the specification and metallurgical condition of the material of the part;
- b) details of any particularly difficult surface condition or coating required to be removed;
- c) the minimum specified tensile strength (or hardness of the material of the part);
- d) instructions concerning any stress relieving heat treatment required before processing including the limits of temperature and time to be employed;
- e) when necessary, the cleaning method or methods given in this standard which are to be used, together with the condition of the basis metal after cleaning;
- f) the surface coating which is to be applied subsequently;
- g) instructions concerning any post-cleaning de-embrittlement which may be required.

2.2 General provisions

2.2.1 Avoidance of mercury

The mercury content of cleaning solutions (as mercury metal or mercury compounds) should not exceed 5 p.p.m. mercury by mass.

2.2.2 Rinsing

Rinsing is essential after each treatment in an aqueous solution, preferably in a multiple rinse sequence. Contamination and staining problems may be avoided or reduced by the use of demineralized water, especially for the final rinse. Parts should be given a final rinse in a pure solvent (before drying) to prevent deposition of soluble materials which may cause problems in subsequent treatments or service.

NOTE To avoid repetition, rinsing operations are not mentioned in the methods described in this standard, except where special sequences are required.

2.2.3 Drying

Parts to be given further treatment by a dry or non-aqueous process and parts which are not to be subjected to further processing, should be thoroughly dried.

If an air draught is used for this purpose it should be free from oil and other contaminants.

2.2.4 Cleaning treatments

Where necessary for efficient cleaning, more than one cleaning treatment should be given.

2.2.5 Condition of basis metal after cleaning

After final cleaning the following conditions should apply:

- a) the surface is free from foreign matter;
- b) the mechanical properties are not significantly impaired except in so far as they may be recovered by subsequent heat treatment;
- c) the dimensions and surface finish are suitable to achieve the desired coating finish.

2.2.6 Increased efficiency of cleaning

The efficiency of some of the cleaning methods may be increased by the application of ultrasonic or other physical methods, e.g. agitation, but care should be taken to avoid damage to delicate parts and assemblies.

2.2.7 Composite components

When composite components are to be processed, the treatments chosen should be suitable for each constituent material and combination of materials.

2.3 Cast irons

2.3.1 Selection of method of cleaning

It should be borne in mind that treatment of cast iron in acid solutions is liable to set up preferential etching around the graphite grains which may become detached, leaving holes which may entrap treatment solution and give rise to subsequent staining or corrosion.

NOTE Cast irons containing flake graphite are particularly liable to give this effect.

2.3.2 Cleaning prior to electroplating

The parts should be cleaned in accordance with one or more of the methods given in 3.1 to 3.11 as appropriate (methods A to L).

2.3.3 Cleaning prior to autocatalytic nickel plating

The following procedure should be carried out:

- a) cleaning in accordance with one or both of the methods given in 3.1 and 3.6 as appropriate (methods A and F);
- b) acid dip in 50 % (V/V) hydrochloric acid or other suitable acid of density 1.16 g/ml.

NOTE A warm rinse may be found desirable immediately before autocatalytic nickel plating to preheat large components but care has to be taken to ensure that the parts do not dry off before being immersed in the autocatalytic nickel solution.

2.3.4 Cleaning prior to phosphating and the application of other conversion coatings

Clean in accordance with one or more of the methods given in 3.1 to 3.11 as appropriate (methods A to L).

NOTE If inhibited solutions are used for acid pickling, see 3.6 (method F), or for derusting, see 3.9 (method J), it may be necessary to remove adsorbed inhibitor before rinsing and phosphating or the application of other conversion coatings.

2.3.5 Cleaning and preparation for hot tinning¹⁾

NOTE The special methods described in 2.3.5.1 to 2.3.5.3 have been developed for preparing cast irons for hot tinning since cast irons may possess hard siliceous casting skins and their surfaces are often rich in graphite which interferes with the tinning processes.

2.3.5.1 (ITRI) Direct chloride process

The procedure for this process should be carried out as follows.

a) Prepare the parts mechanically by grinding, machining, or coarse abrasive blast cleaning in accordance with 3.4.1 (method D.1). Cutting oils or coolants should not be used during machining. When abrasive blast cleaning is used, metallic shot or grit is preferred.

b) Degrease in accordance with 3.1.1 (method A.1);

c) Grit-blast with fine angular chilled iron grit which will pass a BS 410:1986 test sieve of 212 μm nominal aperture size, or fine abrasive liquid blast clean using alumina, unless the mechanical preparation given in item a) consists of fine grinding.

d) Immerse in an aqueous flux solution of the following composition:

zinc chloride	400 g/l
ammonium chloride	25 g/l
hydrochloric acid of density 1.16 g/ml	100 ml (maximum)

e) Tin the parts using an ebullient flux cover on the first tinning bath, consisting of zinc chloride 73 % (V/V), sodium chloride 18 % (V/V) and ammonium chloride 9 % (V/V). The initial thickness of the flux cover should be about 10 mm but before use it should be thoroughly activated by copious spraying with water, which will make it froth and the thickness may rise to 50 mm to 75 mm.

The pre-fluxed castings should be lowered slowly through the ebullient flux cover into the tin.

2.3.5.2 Proprietary fused salt processes

The proprietary processes available which prepare cast iron for tinning by oxidizing graphite exposed at the surface should normally be used electrolytically.

2.3.5.3 Electroplating with a readily tinnable metal

NOTE 1 The difficulty of tinning over a graphite-contaminated surface may be avoided by electroplating the cast iron with a readily tinnable metal such as iron, copper or nickel, iron plating being preferred.

Iron plating should be carried out as follows.

a) Tumble the parts with a suitable fine abrasive in accordance with 3.4.4 (method D.4) or shot blast with fine abrasive in accordance with 3.4.2 (method D.2).

b) Degrease in an organic solvent vapour in accordance with 3.1.1 (method A.1).

c) Pickle for approximately 2 min in hydrochloric acid in accordance with 3.6 (method F.2).

d) Electroplate with iron until the casting is completely covered.

NOTE 2 A suitable electrolyte consists of a solution of 300 g/l ferrous ammonium sulfate operated at room temperature at a current density of 1 A/dm² to 1.5 A/dm².

e) After rinsing, flux and tin the plated castings immediately.

2.3.6 Cleaning and preparation for hot dip galvanizing

Preparation should be by one of the following procedures.

a) Welding slag, iron scale and moulding sand should be removed mechanically (e.g. by abrasive blast-cleaning, see 3.4.1). After blast-cleaning, an acid dip (dilute hydrochloric acid) is advantageous to remove residual iron dust from the surface prior to fluxing where necessary.

b) A hydrofluoric acid solution (e.g. 30 % (m/m) hydrogen fluoride (HF) diluted to 2 % to 10 % (V/V) aqueous solution) is an alternative. Vigorous rinsing and brushing to remove any gelatinous products should follow prior to acid pickling (see 2.4.8) and fluxing where necessary.

WARNING. Rigorous safeguards are needed for operators using hydrofluoric acid.

2.3.7 Cleaning prior to metal spraying (for protection against corrosion and high temperature oxidation)¹⁾

The surface should be degreased in accordance with 3.1 to 3.3 (methods A to C), as appropriate and then roughened by compressed air or centrifugal blasting with a suitable abrasive grit in accordance with 3.4.1 (method D.1). Immediately before spraying it should be free from grease, scale, rust, moisture or other foreign matter. It should be comparable in roughness with a reference surface produced in accordance with Appendix A of BS 2569-2:1965 and should provide an adequate key for the subsequent sprayed metal coating, or meet the requirements of BS 7079-C2:1989, coarse grade.

¹⁾ the information given in this clause has been abstracted from the booklet *Practical hot-tinning* [4] by C.J. Thwaites (published by the International Tin Research Institute) which should be consulted for further information.

2.3.8 Cleaning prior to the application of diffusion coatings

The surface should be cleaned in accordance with one or more of the methods given in 3.1 to 3.11, as appropriate (methods A to L).

2.3.9 Cleaning and preparation for vitreous enamelling

The surface of the casting should be cleaned by blasting in accordance with 3.4.1 (method D.1) using chilled angular grit to remove the chilled surface of the casting and leave an angular finish.

NOTE 1 The correct grade of blasting media will depend upon the degree of cleaning required, but generally a mixture of grade G14 and G15 conforming to BS 2451:1963 is found to be satisfactory.

Unless the castings can be used fresh from the foundry, a burn-off process should be used before the blast-cleaning. This should be carried out at around 700 °C to 750 °C for an appropriate time depending upon the thickness and weight of the casting. Burn-off processes can also sometimes be used to refine the metallurgical structure.

NOTE 2 An annealing ground coat may be used in certain cases so that only one fusing will be necessary in place of two. This will depend upon the quality of the casting.

Castings which have stood for some time should be given heat treatment and blast-cleaning before use.

2.4 Non-corrosion-resisting steels

2.4.1 Selection of method of cleaning

The choice of cleaning methods for medium and high strength steels should be restricted by the recommendations given in 2.4.3 in order to reduce risk of embrittlement by hydrogen absorption during cathodic or acid treatments.

2.4.2 Stress-relieving heat treatment

The appropriate stress-relieving heat treatment given in Annex A should be used.

2.4.3 Cleaning methods²⁾

2.4.3.1 Steels of tensile strength less than 1 000 MPa

The steels should be cleaned in accordance with one or more of the methods as given in 3.1 to 3.11 as appropriate (methods A to L).

2.4.3.2 Steels of tensile strength in the range of 1 000 MPa to 1 400 MPa

No form of cathodic cleaning treatment of these steels should be carried out. The parts should be cleaned in accordance with one or more of the methods given in 3.1, 3.2, 3.4, 3.8 and 3.10 (methods A, B, D, H and K).

NOTE Methods D.2 and K.1 are preferred.

2.4.3.3 Steels of tensile strength in the range of 1 400 MPa to 1 800 MPa

The steels should be cleaned by one or more of the following methods:

- a) degreasing in an organic solvent in accordance with 3.1.1 or 3.1.2 (method A.1 or A.2);
 - b) degreasing in an aqueous alkaline solution in accordance with 3.2.2 (method B.1), with applied anodic direct current if desired. Neither cathodic nor alternating current treatments should be used;
 - c) alkaline derusting in accordance with 3.10.2 (method K.1);
 - d) abrasive cleaning in accordance with 3.4 (method D). This method, especially grit blasting in accordance with 3.4.1 (method D.1), leaves the surface readily corrodible, and further processing should be given without any delay;
 - e) anodic etching in a sulfuric acid solution in accordance with 3.8 (method H), subject to close control. Additionally, parts should be connected and the current switched on before the parts are immersed in the solution, and after treatment the parts should be withdrawn rapidly before the current is finally switched off, and washed immediately. The sulfuric acid should at no time contain more than 11 p.p.m. by volume of oxidizable material calculated as sulfur dioxide, determined in accordance with Annex B;
- NOTE This precaution is necessary because reduced sulfur, phosphorus and arsenic compounds in the acid promote hydrogen absorption during washing.
- f) acid pickling in an inhibited acid solution in accordance with 3.6 (method F.1) followed, if desired and subject to agreement, by a dip in plain acid in accordance with 3.7 (method G) and then by baking prior to further treatment, at not less than 190 °C for not less than 4 h.
 - g) electrolytic derusting using an alkaline electrolyte and anodic current.

2.4.3.4 Steels of tensile strength greater than 1 800 MPa

Steels should be cleaned by one or more of the following treatments:

- a) degreasing in an organic solvent in accordance with 3.1.1 or 3.1.2 (method A.1 or A.2);
- b) alkaline treatments, in accordance with items b), c), and g) of 2.4.3.3;
- c) abrasive cleaning in accordance with item d) of 2.4.3.3.

²⁾ The information in this clause has been abstracted from the booklet *Practical hot-tinning* [4] by C.J. Thwaites (published by the International Tin Research Institute) which should be consulted for further information.

2.4.4 Cleaning prior to electroplating

The parts should be cleaned in accordance with one or more of the methods given in 2.4.3 appropriate to the tensile strength of the steel but may require activation by immersion in a weak acid for up to 60 s.

NOTE If inhibited solutions are used for acid pickling, see 3.6 (Method F), or for derusting, see 3.9 (method J), it may be necessary to remove adsorbed inhibitor from the surface by acid dipping, see 3.6 (method F without inhibitors) or by suitable alkaline solution, see 3.2 (method B) before chemical smoothing, see 3.11, (method L), and/or electroplating.

2.4.5 Preparation for autocatalytic nickel plating

2.4.5.1 Steels of tensile strength less than 1 000 MPa

The following procedure should be carried out:

- a) clean in accordance with one or more of the methods given in 3.1 to 3.6 (methods A to F), as appropriate;
- b) acid dipping in 50 % (V/V) hydrochloric acid or other suitable acid of density 1.16 g/ml;
- c) autocatalytic nickel plating.

NOTE 1 An alkaline periodic reverse current cleaner (10 s each way finishing anodically) can be used as an alternative to the activating treatment for stage b). Cathodic treatments alone may cause hydrogen embrittlement and prolonged anodic treatments are undesirable because of the risk of passivating the surface.

NOTE 2 A warm rinse may be found desirable immediately before stage c) to preheat large components but care should be taken to ensure that the parts do not dry off before being immersed in the autocatalytic nickel solution.

2.4.5.2 Steels of tensile strength in the range of 1 000 MPa to 1 400 MPa

The following procedure should be carried out.

- a) cleaning in accordance with one or more of the methods given in 3.1 to 3.6 (methods A to F), as appropriate;
- b) anodically etching in accordance with 3.8 (method H), for 1 min to 3 min;
- c) rinsing very thoroughly to remove all viscous sulfate solution from the surface;
- d) autocatalytic nickel plating.

2.4.6 Cleaning prior to phosphating and the application of other conversion coatings

One or more of the methods given in 2.4.3 appropriate to the phosphating or other process and to the tensile strength of the steel should be used. Before phosphating, a conditioner or grain refiner may be used.

2.4.7 Cleaning and preparation for hot tinning³⁾

2.4.7.1 Normal mild steels

The parts should be cleaned in accordance with 3.1.1 (method A.1) or 3.2.2 (method B.1), followed by either method F.1 or method F.2 given in 3.6.

2.4.7.2 "Difficult" mild steels

NOTE 1 Certain batches of mild steel sheet and strip are more difficult to tin than others, coatings tending to de-wet even when the usual methods of cleaning and pickling are thoroughly carried out. Such steels are called "difficult" steels. They possess a non-reactive surface skin formed by the polymerization of certain rolling lubricants present on the surface during low temperature bright annealing, or during such operations as pressing and spinning.

"Difficult" steels should be prepared for tinning by one of the following methods, as appropriate to the particular component:

- a) abrasive cleaning in accordance with 3.4.1 or 3.4.2 (method D.1 or D.2);

- b) heat treatment within the temperature range 700 °C to 850 °C for sufficient time for the component to reach the operating temperature of the furnace. The temperature employed should be the lowest which will effectively oxidize the surface contaminant and should be determined by carrying out trials on test pieces from the same batch of material. Any loose scale should be brushed off the component, see 3.4.5 (method D.5), which is then treated in accordance with 2.4.7.1;

- c) etching in nitric acid. If there is any visible rust or scale on the article it should be treated in accordance with 2.4.7.1. It should then be immersed in an approximately 10 % (V/V) solution of nitric acid of density 1.42 g/ml at a temperature of approximately 25 °C. An immersion time of 1 min to 3 min is normally sufficient. The article should be rinsed, dipped for approximately 1 min in dilute hydrochloric acid in accordance with 3.6 (method F.2), and rinsed again.

NOTE 2 It is then ready for fluxing and tinning.

- d) etching in nitric-sulfuric acid mixture. The article should be immersed in a solution containing approximately 4 % (m/m) of nitric acid of density 1.5 g/ml and 20 % (m/m) of sulfuric acid of density 1.84 g/ml at 25 °C to 40 °C for about 4 min to 6 min. The removal of any rust or scale should be carried out in accordance with method F.1 or F.2 given in 3.6 before or after the etching in the nitric-sulfuric acid mixture.

³⁾ The information given in this clause has been abstracted from the booklet *Practical hot-tinning* [4] by C.J. Thwaites (published by the International Tin Research Institute which should be consulted for further information).

2.4.8 Cleaning and preparation for hot dip galvanizing⁴⁾

2.4.8.1 Steel (other than castings)

Preparation should be by the following general procedures.

- a) Degreasing, if required, is generally in a hot alkaline solution, the strength of the alkali being related to the amount of grease to be removed.
- b) Abrasive blast-cleaning may be used to remove millscale and other mechanical methods (e.g. chipping) may be used to remove slag from the unclean welds. Paint may also be removed mechanically or by chemical paint strippers. Pickling etc. follows.
- c) Pickling is generally in cold hydrochloric acid at room temperature (18 °C or above), typically made initially by diluting commercial 27.5 % (m/m) acid with an equal volume of water. Hot sulfuric acid may alternatively be used (e.g. 10 % to 14 % (m/m) sulfuric acid at 60 °C to 65 °C). Pickling inhibitors to reduce attack on the basis metal are often added.
- d) A flux coating of dry iron chloride salts (from hydrochloric acid pickling), zinc ammonium chloride or proprietary flux is then normally applied before immersion in the molten zinc.

2.4.8.2 Steel castings

Preparation should be carried out using one of the following procedures.

- a) *Abrasive blast-cleaning with chilled iron or steel grit* in accordance with 3.4.1 (method D.1) to remove moulding sand and iron scale. After blasting, methods F.1 or F.2 given in 3.6 may be used and where necessary a flux coating should be applied.
- b) *Hydrofluoric acid* [30 % (m/m)] diluted to 2 % (V/V) to 10 % (V/V) aqueous solution may be used, followed by hosing with water to remove any gelatinous products. After hosing, method F.2 given in 3.6 is used and if necessary a flux coating should be applied.

WARNING. Rigorous safeguards are needed for operators using hydrofluoric acid.

2.4.9 Cleaning for metal spraying (for protection against corrosion and high temperature oxidation)⁵⁾

The surface should be degreased in accordance with 3.1 to 3.3 (methods A to C) where necessary and as appropriate and then roughened by compressed air or centrifugal blasting with a suitable abrasive grit in accordance with 3.4.1 (method D.1).

NOTE 1 After preparation the surface should be sprayed without undue delay.

Immediately before spraying the surface should be free from grease, scale, rust, moisture, or other foreign matter. It should be comparable in roughness with a reference surface produced in accordance with appendix A of BS 2569-2:1965 and should provide an adequate key for the subsequent sprayed metal coating.

NOTE 2 Depending on the geometry of the component, normal methods of preparation may not prove satisfactory due to the risk of distortion. Material thinner than 3 mm cannot always be satisfactorily prepared for metal spraying; the metal sprayer should be consulted.

2.4.10 Cleaning prior to the application of diffusion coatings

The surface should be cleaned in accordance with one or more of the methods given in 2.4.3, according to the tensile strength of the steel.

2.4.11 Cleaning and preparation for vitreous enamelling

2.4.11.1 Conventional enamelling

The surface should be cleaned and prepared as follows.

- a) Subject the surface to vapour degreasing in accordance with 3.1.1 (method A.1) or abrasive blasting in accordance with 3.4 (method D), where appropriate.

NOTE 1 Vapour degreasing is optional but may be preferred for components with welded seams.

- b) Clean in an alkaline solution in accordance with 3.2 (method B), with or without prior vapour degreasing. Rinse in hot and cold water.
- c) Acid pickle in accordance with 3.6 (method F.1 or F.2). Wetting agents and inhibitors should be avoided.
- d) Rinse.

⁴⁾ The information given in this clause has been abstracted from *General galvanizing practice* [5] (published by the Galvanizers' Association), which should be consulted for further information.

⁵⁾ For further information see BS 2569-2:1965 and BS EN 22063:1994.

e) A useful, but non-essential, extra process is the deposition of a displacement coating of nickel by immersion in a solution

containing 10 g/l to 20 g/l nickel sulfate at a pH of 3.0 to 3.5 and a temperature of 70 °C to 80 °C. Time of immersion should be such that 0.40 g/m² to 0.60 g/m² of nickel is deposited on one side. Rinse thoroughly.

f) Immerse in a neutralizing solution of the following composition for 3 min to 5 min:

anhydrous sodium carbonate (soda ash) (Na ₂ CO ₃)	4.5 g/l
borax	1.5 g/l

The bath temperature should be 70 °C to 80 °C.

g) Dry, preferably in clean, warm air.

NOTE 2 When oil, grease or drawing compound is difficult to remove by other means, heat treatment ("grease burning") at 600 °C to 700 °C may be necessary before the operation given in item c).

2.4.11.2 Direct-on enamelling

NOTE 1 Direct-on enamelling is appropriate only for use on decarbonized steel.

The surface should be cleaned and prepared as follows.

a) Degrease in organic solvent in accordance with 3.1 (method A), either as a vapour or a liquid. If the liquid phase is used, it is essential that the ware be allowed to drain well before passing to the next operation.

b) Rinse if necessary.

NOTE 2 The operations given in items a) and b) are optional.

c) Clean in alkaline solution in accordance with 3.2 (method B).

d) Rinse in hot agitated, flowing water.

e) Rinse in cold flowing water.

f) Acid pickle in accordance with 3.6 (method F.1).

NOTE 3 The amount of etch carried out is extremely critical to the finished quality of the enamel and the required amount is a function of the steel/enamel system. This etch is normally measured as a mass loss in grams per square metre and is controlled by temperature, 60 °C to 80 °C, and concentration 50 g/l to 80 g/l. It is essential that wetting agents are not used.

g) Rinse in cold water.

h) Deposit a nickel coating by immersion in either one of the following solutions:

1) reduction solution	
nickel sulfate	10 g/l
sodium acetate	6 g/l
sodium hypophosphite	5 g/l

at a pH of 4.8 to 5.1, adjustment being made with ammonia solution. A temperature of 30 °C to 35 °C is normal.

2) displacement solution

nickel sulfate 10 g/l to 20 g/l at a pH of 3.0 to 3.5 and a temperature of 70 °C to 80 °C.

Time of immersion should be such that 0.60 g/m² to 1.00 g/m² of nickel is deposited on one side of the sheet steel surface (greater than this will reduce adhesion of enamel).

i) Water rinse. This should be at pH 3 to pH 5 for rinse after the displacement nickel process.

j) Nickel treatment should be followed by immersion in a neutralizing solution of the following composition for 3 min to 5 min:

anhydrous sodium carbonate (soda ash) (Na ₂ CO ₃)	4.5 g/l
borax	1.5 g/l

The bath temperature should be 70 °C to 80 °C.

k) Dry, preferably with clean, warm, dry air.

2.4.12 Cleaning prior to painting

Phosphate coatings types 3 or 4 conforming to BS 3189:1991 should normally be used prior to painting. When it is required to paint directly onto metal, it is essential that the surface be free from grease, oxide, scale or other foreign matter. This should be accomplished by the use of methods given in 3.1 to 3.4 and 3.7 (methods A, B, C and G) alone or in combination.

2.5 Corrosion-resisting steels (steels containing not less than 9 % (m/m) chromium)

2.5.1 General cleaning

Parts should be cleaned by one or more of the methods given in 2.4.3 appropriate to the tensile strength of the steel. After cleaning, parts not subject to further processing should either be given a passivation treatment in accordance with 3.12 (method M), or should be electropolished.

NOTE 1 Such treatment may be essential if iron or iron oxide remains embedded in the surface, e.g. from abrasive blast-cleaning or from pick-up from forming tools.

NOTE 2 High density solutions are used for electropolishing (electrochemical brightening) martensitic, ferritic and austenitic stainless steels. The exact working conditions are critical.

Electropolishing generates an ideally clean and passive condition by anodic electrolytic action in strong mineral acid solutions: it is generally regarded as a final process which includes passivation treatment.

2.5.2 Preparation for electroplating

2.5.2.1 General

Parts should be cleaned in accordance with one or more of the methods given in 2.5.2.2 to 2.5.2.4, appropriate to the tensile strength of the steel.

2.5.2.2 Steels of tensile strength less than 1 000 MPa

The parts should be cleaned in accordance with one or more of the methods as appropriate (methods A to L).

2.5.2.3 Steels of tensile strength in the range of 1 000 MPa to 1 400 MPa

No form of cathodic cleaning treatment of these steels should be carried out. The parts should be cleaned in accordance with one or more of the methods given in 3.1, 3.2, 3.4, 3.8 and 3.10 (methods A, B, D, H or K), of which methods D.2 and K.1 are preferable.

2.5.2.4 Steels of tensile strength above 1 400 MPa

The parts should be cleaned by one or more of the following methods. The steps selected in the cleaning sequence should follow one another without interruption other than any necessary washing and should be followed immediately by the protective treatment:

- a) degreasing in an organic solvent in accordance with 3.1.1 or 3.1.2 (method A.1 or A.2);
- b) degreasing in an aqueous alkaline solution in accordance with 3.2.2 (method B.1), with applied anodic direct current if desired. Neither cathodic nor alternating current treatments should be used;
- c) alkaline derusting in accordance with 3.10.2 (method K.1);
- d) abrasive cleaning in accordance with 3.4 (method D). This method, especially grit blasting in accordance with 3.4.1 (method D.1) leaves the surface very readily corrodible, and further processing should be given without any delay;
- e) anodic etching in a sulfuric acid solution in accordance with 3.8 (method H) subject to close control. Additionally, parts should be connected and the current switched on before the parts are immersed in the solution, and after treatment the parts should be withdrawn rapidly before the current is finally switched off, and washed immediately.

The sulfuric acid should at no time contain more than 11 p.p.m. by volume oxidizable material calculated as sulfur dioxide, determined in accordance with Annex B;

NOTE 1 This precaution is necessary because reduced sulfur, phosphorus and arsenic compounds in the acid promote hydrogen absorption during washing.

f) acid pickling in an inhibited acid solution in accordance with 3.6 (method F.1), followed if desired and subject to agreement, by a dip in uninhibited acid in accordance with 3.7 (method G) and then by heat treating prior to further treatment, at not less than 190 °C for not less than 4 h;

g) electrolytic derusting using an alkaline electrolyte and anodic current in accordance with 3.10 (method K).

Cleaning should normally be followed by a nickel strike in accordance with 3.20 (method U) before plating with the metal(s) required.

NOTE 2 Modified methods including the omission of the nickel strike, may be used provided that adequate adhesion is obtained.

2.5.3 Preparation for autocatalytic nickel plating

The parts should be cleaned by one or more of the methods given in 2.5.1 appropriate to the tensile strength of the steel and then given a nickel strike either in accordance with 3.20 (method U) or electrolytically in the autocatalytic nickel solution until the surface is covered.

NOTE If the shape of the component makes electrolytic treatments difficult or impossible the surface can usually be activated by dipping for 1 min to 2 min in 50 % (V/V) hydrochloric acid of density 1.16 g/ml at a temperature of 40 °C to 50 °C.

After rinsing the part should be transferred directly to the autocatalytic nickel bath.

2.5.4 Cleaning prior to metal spraying

Cleaning and preparation prior to metal spraying should be carried out as for non-corrosion-resisting steels in accordance with 2.4.9.

2.5.5 Cleaning prior to vitreous enamelling

2.5.5.1 If the design of the component is suitable, abrasive blast-cleaning is a satisfactory method of preparation, and one of the procedures given in 2.5.5.2 or 2.5.5.3 should be used.

When abrasive blast-cleaning is not possible, one of the procedures given in 2.5.5.4 or 2.5.5.5 should be used.

2.5.5.2 The following procedure should be carried out.

- a) Vapour degrease in trichloroethylene in accordance with 3.1 (method A), or treat by immersion in an alkaline solution in accordance with 3.2 (method B).
- b) Rinse in hot, agitated, flowing water.
- c) Dry in clean, warm, dry air.

d) Abrasive blast-clean in accordance with 3.4.2 (method D.2), using alumina grit.

e) Blow dry with clean, high pressure air.

2.5.5.3 The following procedure should be carried out.

a) Heat treat at approximately 800 °C to burn off any grease.

b) Abrasive blast-clean in accordance with 3.4.2 (method D.2), using alumina grit.

c) Blow dry with clean, high pressure air.

2.5.5.4 The following procedure should be carried out.

a) Degrease either by a vapour solvent in accordance with 3.1 (method A), or by immersion in an alkaline solution in accordance with 3.2 (method B).

b) Parts which have been degreased in an alkaline cleaner should be rinsed in hot, agitated, flowing water.

c) Rinse in cold running water.

d) Etch in an aqueous solution containing 10 % (V/V) nitric acid of density 1.42 g/ml and 2 % (V/V) hydrofluoric acid [40 % (m/m)]. The temperature should be in the range from room temperature to 40 °C and the time of immersion is usually from 5 min to 20 min.

WARNING. Rigorous safeguards are needed for operators using hydrofluoric acid.

e) Rinse with high pressure water to remove solids.

f) Immerse in neutralizing solution of the following composition for 3 min to 5 min:

anhydrous sodium carbonate (Na ₂ CO ₃)	4.5 g/l
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borax	1.5 g/l
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The bath temperature should be 70 °C to 80 °C.

g) Rinse in hot, agitated, flowing water.

h) Dry in clean, warm, dry air.

2.5.5.5 The following procedure should be carried out.

a) Heat treat at approximately 800 °C to burn off any grease.

b) Etch in an aqueous solution containing 10 % (V/V) nitric acid of density 1.42 g/ml and 2 % (V/V) hydrofluoric acid (40 % (m/m)). The temperature should be in the range from room temperature to 40 °C and the time of immersion is usually from 5 min to 20 min.

WARNING. Rigorous safeguards are needed for operators using hydrofluoric acid.

c) Rinse with high pressure water to remove sludge.

d) Immerse in neutralizing solution of the following composition for 3 min to 5 min:

anhydrous sodium carbonate (Na ₂ CO ₃)	4.5 g/l
--	---------

borax	1.5 g/l
-------	---------

The bath temperature should be 70 °C to 80 °C.

e) Rinse in hot, agitated, flowing water.

f) Dry in clean, warm, dry air.

2.6 Aluminium and its alloys

2.6.1 General cleaning

Parts should be cleaned by one or more of the following methods as appropriate:

a) in accordance with 3.1 (method A), 3.2.3 (method B.2) and 3.4 (method D);

NOTE 1 Aluminium alloys are usually resistant to pure halogenated hydrocarbons. However, aluminium in a finely divided form, as in a powder, should not be exposed to a halogenated hydrocarbon and surfaces should therefore be free from aluminium dust.

b) etching in chromic acid/sulfuric acid solution in accordance with 3.14 (method O);

c) pickling in accordance with 3.13 (method N);

d) an agreed chemical polishing treatment in accordance with 3.29 (method DD), or electrolytic polishing treatment in accordance with 3.28 (method CC).

NOTE 2 Certain heat treatable aluminium alloys may suffer some alteration in mechanical properties as a result of prolonged treatment at or above 100 °C.

2.6.2 Cleaning of corroded parts

Where corrosion products are to be removed, immersion in chromic acid/phosphoric acid solution in accordance with 3.13 (method N.6) is the method recommended as it results in least attack on underlying metal. Light corrosion products and soil should be removed in accordance with 3.14 (method O), by a phosphoric acid solution in accordance with 3.15 (method P.1) or by fine abrasive blast-cleaning in accordance with 3.4.2 (method D.2).

2.6.3 Preparation for adhesive bonding of unanodized parts

Parts should be prepared for bonding by cleaning and etching in accordance with 3.14 (method O) or by abrasive blast-cleaning in accordance with 3.4.2 (method D.2).

2.6.4 Preparation for electroplating

As a preparation for the electrodeposition of any desired finish, parts should normally be degreased, pickled and given an immersion deposit of zinc or tin followed by an electrodeposit of copper or brass. A recommended procedure is given in 3.21 (method V).

NOTE Other preparatory procedures may be employed, provided that the inspection criteria of the relevant plating specifications are satisfied.

2.6.5 Preparation for autocatalytic nickel deposition

Individual steps should be modified according to the alloy processed, but any process providing a good zincate or modified zincate coating suitable for electroplating will also be satisfactory for autocatalytic nickel plating.

NOTE 1 Proprietary processes are available which cover a wide range of alloys.

A recommended typical sequence, employing the double zincate method, is as follows.

- a) Degrease in accordance with 3.1 (method A), and mild alkali clean in accordance with 3.2.3 (method B.2), as required.
- b) Rinse.
- c) Etch in mixed acid etch (approximately 30 s) in accordance with 3.21.3.
- d) Rinse.
- e) Immerse in zincate solution for 1 min to 2 min in accordance with 3.21.5.
- f) Rinse.
- g) Immerse in 50 % (V/V) nitric acid for approximately 1 min.
- h) Rinse.
- i) Immerse in zincate solution for 10 s to 30 s in accordance with 3.21.5.
- j) Rinse.
- k) Autocatalytic nickel plate.

NOTE 2 When autocatalytic nickel is used as an undercoat, a minimum thickness of 25 µm is recommended. As with electroplating of aluminium, there are numerous methods for preparing aluminium successfully for plating and some of these methods may also be found to be satisfactory for autocatalytic nickel, e.g. anodic treatments on aluminium which give porous oxide films satisfactory for plating and which give good adhesion of the coating.

2.6.6 Cleaning and preparation prior to anodizing

Parts should be cleaned by one or more of the following methods:

- a) the procedures described in 3.1, 3.2.3 and 3.4 (methods A, B.2 and D);
- b) etching in chromic acid/sulfuric acid solution in accordance with 3.14 (method O);
- c) pickling in accordance with 3.13 (method N);

d) a chemical polishing treatment in accordance with 3.27 (method BB), or an electropolishing treatment in accordance with 3.28 (method CC);

e) etching in sodium hydroxide solutions in accordance with 3.15.2 (method P.2).

NOTE Certain heat-treatable aluminium alloys may be adversely affected as a result of prolonged treatment at or near 100 °C.

2.6.7 Cleaning prior to the application of conversion coatings

Parts should be cleaned by one or more of the methods given in 2.6.6.

2.6.8 Preparation for metal spraying

The parts should be cleaned in accordance with one or both of the methods given in 3.1 and 3.2 (methods A and B) followed by coarse abrasive blast-cleaning in accordance with 3.4.1 (method D.1) using a non-metallic abrasive.

2.6.9 Preparation for vitreous enamelling

2.6.9.1 Sheet, extrusions and certain casting alloys

A recommended procedure which is generally applicable to sheet and extrusions and is satisfactory for certain casting is as follows.

- a) Degrease in a proprietary mild etching cleaner or buffered alkaline cleaner in accordance with 3.2.3 (method B.2) but at pH 8.9 to pH 10.0.
- b) Rinse in cold water.
- c) Either of the following:
 - 1) immerse for 7 min to 15 min in the following deoxidizing solution:

chromic acid (CrO ₃)	30 g/l to 40 g/l
sulfuric acid	165 g/l to 185 g/l

at an operating temperature of 80 °C;

- 2) use a proprietary non-chromated deoxidizer.
- d) Rinse in cold water.
- e) Immerse for 30 s to 2 min in the following alkaline chromate solution:

potassium chromate	(200 ± 10) g/l
sodium hydroxide	(40 ± 10) g/l

at an operating temperature of 25 °C.

- f) Rinse in cold water.
- g) Rinse in hot or cold deionized water.
- h) Dry with hot air or high velocity ambient air.

2.6.9.2 Castings

A recommended procedure for treating castings generally is as follows.

- a) Degrease in a non-etching acid cleaner or buffered alkaline cleaner.
- b) Rinse in cold water.
- c) Rinse in hot or cold deionized water.
- d) Dry with hot air or high velocity air at ambient temperature.

2.6.10 Preparation of parts for painting that have not previously received an anodic conversion

All parts should be degreased in accordance with one or both of the methods given in **3.1** and **3.2.3** (methods A and B.2) and then treated in one of the following ways.

On components which have been anodized over part of the surface, a suitable pretreatment (etch) primer should be applied overall.

NOTE 1 Prior to the application of a pretreatment (etch) primer, parts may, if desired, be treated by etching in accordance with **3.14** or **3.15** (methods O or P) or by an etching process agreed between the parties.

NOTE 2 Parts for painting which have not been coated with a suitable etch primer should be treated by etching in accordance with **3.15** (method P) or by an etching or filming process agreed between the parties.

2.7 Copper and its alloys

2.7.1 Avoidance of stress corrosion cracking

It is recommended that alloys in a condition susceptible to stress corrosion cracking should be cleaned only in organic solvents in accordance with **3.1** (method A) prior to stress relief. After stress relief, these alloys should be cleaned by any of the methods given in **2.7.2**.

2.7.2 General cleaning

Parts should be cleaned by one or more of the following methods as appropriate:

- a) degreasing in an organic solvent in accordance with **3.1** (method A);
- b) degreasing in an aqueous alkaline solution in accordance with **3.2** (method B);
- c) coarse abrasive blast-cleaning in accordance with **3.4.1** (method D.1) (for castings), fine abrasive blast-cleaning in accordance with **3.4.2** (method D.2) or scouring in accordance with **3.4.3** (method D.3);
- d) cleaning etch treatment in accordance with **3.16** (method Q);
- e) etching or brightening by an agreed chemical treatment in accordance with **3.17** (methods R.2 and R.3) or electrolytic treatment in accordance with **3.29** (method DD).

2.7.3 Cleaning prior to electroplating

2.7.3.1 Parts should be freed from any grease, tarnish or scale by one or more of the following treatments, as appropriate:

- a) degreasing in organic solvent in accordance with **3.1** (method A);
- b) degreasing in an aqueous alkaline solution in accordance with **3.2** (method B);
- c) scouring in accordance with **3.4.3** (method D.3);
- d) cleaning etch treatment in accordance with **3.16** (method Q);
- e) acid pickling in accordance with **3.17** (method R);

NOTE Sulfuric acid and hydrochloric acid should be avoided with leaded brass parts which should be treated as in **2.7.3.2**.

- f) etching or polishing by a chemical or electrolytic treatment, e.g. in accordance with **3.29** (method DD), acceptable to the purchaser.

2.7.3.2 Soft soldered parts (other than beryllium copper parts) should be dipped in a solution containing approximately 100 ml/l fluoroboric acid [40 % (*m/m*)] and rinsed thoroughly in water and copper flashed in an alkaline copper electrolyte at pH 10 to pH 12 and a temperature of $(60 \pm 10) ^\circ\text{C}$.

2.7.3.3 The cleaning etch treatment in accordance with **3.16.1** (method Q.1) is suitable for beryllium copper parts and should be followed by rinsing in water and immersion in a solution containing 100 ml/l of sulfuric acid when plating in an acid electrolyte is to be the next treatment.

2.7.4 Preparation for autocatalytic nickel plating

The surface should be treated in accordance with **2.7.3.1** and then the following procedure should be carried out.

- a) Clean cathodically in a mild alkaline cleaner in accordance with **3.2.3** (method B.2), preferably containing cyanide.
- b) Rinse.
- c) Catalyse the surface by one of the following three methods.

1) Strike in an electrolytic nickel bath in accordance with **3.20** (method U), until the surface is covered. Electrolytic striking in the autocatalytic nickel solution is also satisfactory.

2) Immerse in an activating solution, e.g. one containing palladium chloride, until the surface has darkened, showing deposition of palladium, or follow the directions of suppliers of proprietary solutions.

3) Contact the surface with aluminium wire while under the solution, or contact with an already plated part. Autocatalytic nickel will gradually spread to cover the entire surface.

2.7.5 Cleaning prior to hot tinning⁶⁾

Parts should be cleaned by degreasing in an organic solvent in accordance with 3.1 (method A), or in an aqueous alkaline solution in accordance with 3.2 (method B). Oxygen-containing copper should preferably be treated cathodically at a current density of 1 A/dm² to 5 A/dm² in an alkaline solution (e.g. 50 g/l sodium hydroxide).

Subsequent pickling should be carried out by one of the following methods:

- a) scale dip in accordance with 3.17.1 (method R.1);
- b) bright dip in accordance with 3.17.2 (method R.2);
- c) nitric acid dip in accordance with 3.17.4 (method R.4).

2.7.6 Preparation for vitreous enamelling

The following procedure is recommended.

- a) Degrease using one of the following procedures.
 - 1) Particularly for small pieces, e.g. for jewellery, heat in a furnace or with a blow torch. When the metal is at a dull red heat, either plunge the pieces directly into cold water, which should be flowing, or allow them to cool in air.

NOTE 1 There is the possibility of distortion occurring when hot metal of relatively thin section is quenched in hot water.

 - 2) For large and heavy articles or panels, use alkaline immersion cleaners in accordance with 3.2 (method B). Rinse in cold, flowing, agitated water.
 - 3) Depending on the type of lubricant used in pressing, use trichlorethylene in accordance with 3.1 (method A). Rinse in cold, flowing, agitated water.

b) Pickling should be carried out by one of the following methods.

- 1) Immerse for 2 min to 10 min in an aqueous solution of nitric acid [10 % (V/V) to 15 % (V/V)] at room temperature.
- 2) Immerse for 5 min to 15 min in a 10 % (V/V) aqueous solution of hydrochloric acid.

NOTE 2 It may be desirable to remove scale with a stiff brush during pickling when grease-burning has been carried out during the procedure in item a).

c) Rinse in cold flowing water which should preferably be agitated.

d) Neutralize in a mildly alkaline solution.

e) Dry with clean, hot air.

2.7.7 Cleaning prior to painting

Parts to be painted should be cleaned by one or more of the following methods:

- a) degreasing in an organic solvent in accordance with 3.1 (method A);
- b) degreasing in an aqueous alkaline solution in accordance with 3.2 (method B);
- c) coarse abrasive blast-cleaning in accordance with 3.4.1 (method D.1) (for castings), fine abrasive blast-cleaning in accordance with 3.4.2 (method D.2), or scouring in accordance with 3.4.3 (method D.3);
- d) cleaning etch treatment in accordance with 3.16 (method Q);
- e) etching or polishing by a chemical treatment in accordance with 3.17 (method R), or electrolytic treatment in accordance with 3.30 (method EE).

2.8 Nickel and its alloys

2.8.1 General cleaning

The parts should be cleaned by one or more of the following methods as appropriate:

- a) degreasing in an organic solvent in accordance with 3.1 (method A);
- b) degreasing in an aqueous alkaline solution in accordance with 3.2 (method B);
- c) abrasive cleaning in accordance with 3.4 (method D);
- d) molten caustic alkali descaling in accordance with 3.5 (method E);
- e) pickling in an acid solution;

NOTE The supplier should always be consulted concerning the pickling of nickel alloys. The choice of solution depends on the type of alloy concerned and this should be agreed between the parties.

Solutions are usually of an aggressive nature and are generally based on nitric/hydrofluoric acids, with or without the addition of ferric sulfate.

- f) an agreed chemical polishing process in accordance with 3.31 (method FF), or electropolishing process in accordance with 3.32 (method GG).

⁶⁾ The information given in this clause has been abstracted from the booklet *Practical hot-tinning* [4] by C.J. Thwaites (published by the International tin Research Institute) which should be consulted for further information.

2.8.2 Preparation for electroplating

Parts should be treated in accordance with 3.22 (method W) preceded by one or more of the following methods as appropriate:

- a) degreasing in an organic solvent in accordance with 3.1 (method A);
- b) degreasing in an aqueous alkaline solution in accordance with 3.2 (method B);
- c) abrasive cleaning in accordance with 3.4 (method D);
- d) descaling in molten caustic alkali in accordance with 3.5.1 (method E.1);
- e) pickling in an acid solution in accordance with 3.19 (method T);
- f) a chemical polishing process in accordance with 3.31 (method FF), or electrolytic polishing process in accordance with 3.32 (method GG), acceptable to the purchaser.

2.8.3 Preparation for autocatalytic nickel plating

The parts should be prepared in the same way as for electroplating in accordance with 2.8.2. After the nickel strike, see 3.22 (method W), the parts should be thoroughly rinsed and then transferred to the autocatalytic nickel solution.

2.8.4 Preparation for vitreous enamelling

Parts should be cleaned in accordance with 2.5.5.

2.8.5 Preparation for painting

Parts to be painted should be cleaned by one or more of the methods given in 2.8.2, as appropriate.

2.9 Titanium and its alloys

2.9.1 General cleaning

2.9.1.1 Parts should be cleaned by one or more of the methods given in 2.9.1.2 to 2.9.1.6, as appropriate. Care should be taken to ensure that the particular process will not cause any significant hydrogen embrittlement or stress corrosion.

2.9.1.2 Degreasing in an organic solvent should be carried out in accordance with 3.1 (method A). Chlorinated hydrocarbon solvents may cause stress corrosion cracking of some titanium alloys and the following precautions should be observed when these solvents are used.

- a) Parts of Ti-5Al-2½Sn alloy made by cold forming or welding should not be exposed to hot chlorinated hydrocarbon solvents prior to stress relief.
- b) The solvents should be maintained clean and the accumulation of free acid should be prevented in accordance with the supplier's operating instructions.

c) The time of immersion in hot solvents and/or vapour should not exceed 30 min for any one degreasing operation.

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

2.9.1.3 Degreasing in an aqueous alkaline solution should be carried out in accordance with 3.2 (method B). Cathodic cleaning should not be used.

2.9.1.4 Abrasive cleaning by wet methods only should be carried out in accordance with 3.4 (method D).

2.9.1.5 Descaling should be carried out by a process, e.g. in accordance with 3.5 (method E), acceptable to the purchaser.

2.9.1.6 Pickling in nitric acid/hydrofluoric acid solution should be carried out in accordance with 3.18 (method S).

WARNING. Rigorous safeguards are needed for operators using hydrofluoric acid.

2.9.2 Preparation for electroplating

NOTE Titanium and its alloys are difficult to electroplate. Preparatory treatments should be in accordance with 3.2.3 (method X).

2.9.3 Preparation for autocatalytic nickel plating

The parts should be prepared as for electroplating in accordance with items a) to c) of 3.23.1 (method X.1). After the nickel strike, the parts should be thoroughly rinsed and transferred to the autocatalytic nickel solution.

2.9.4 Preparation for painting

2.9.4.1 Parts should be cleaned by one or more of the methods given in 2.9.4.2 to 2.9.4.6 as appropriate. Care should be taken to ensure that the particular process will not cause any significant hydrogen embrittlement or stress corrosion.

2.9.4.2 Degreasing in an organic solvent should be carried out in accordance with 3.1 (method A). Chlorinated hydrocarbon solvents may cause stress corrosion cracking of some titanium alloys and the following precautions should be observed when the solvents are used.

- a) Parts of Ti-5Al-2½Sn alloy made by cold forming or welding should not be exposed to hot chlorinated hydrocarbon solvents prior to stress relief.
- b) The solvents should be kept clean and the accumulation of free acid should be prevented in accordance with the supplier's operating instructions.

- c) The time of immersion in some solvents, e.g. hot trichloroethylene or perchloroethylene and/or vapour liquid should not exceed 30 min.
- d) Care should be taken to remove all traces of solvent from the part immediately after cleaning before any further operation is performed.

2.9.4.3 Degreasing in an aqueous alkaline solution should be carried out in accordance with **3.2** (method B). Cathodic cleaning should not be used.

2.9.4.4 Abrasive cleaning by wet methods only in accordance with **3.4** (method D.2, D.3 or D.4).

2.9.4.5 Descaling should be carried out by a process, acceptable to the customer, e.g. in accordance with **3.5** (method E).

2.9.4.6 Pickling in nitric acid/hydrofluoric acid solution should be carried out in accordance with **3.18** (method S).

WARNING. Rigorous safeguards are needed for operators using hydrofluoric acid.

2.10 Magnesium alloys

2.10.1 General cleaning

Cleaning of magnesium alloys, including specific methods to ensure a decontaminated surface, forms part of a complete protective scheme which should be agreed between the parties. Degreasing should be carried out in accordance with one of the methods given in **3.1** and **3.2**, as appropriate.

2.10.2 Preparation for electroplating

A recommended preparatory treatment is given in **3.25** (method Z).

2.10.3 Preparation for conversion coating and painting

2.10.3.1 General

In order that magnesium alloys should show the highest resistance to corrosion it is important that:

- the alloy itself should contain no entrapped flux and should have only a low content of impurities;
- the surfaces of parts should not be contaminated by flux, sand, or other residues from the casting process, or by metallic or other impurities which may be introduced during working processes such as die casting, rolling or forging or during abrasive blasting. Precautions should be taken during machining to avoid smearing other metals, e.g. from machining residues in cutting fluids, into the surface.

2.10.3.2 As-cast castings

If contaminated with sand, or if otherwise necessary, castings, other than certain die castings, should be rough cleaned by blasting with steel grit or non-metallic grit in accordance with **3.4.1** (method D.1), by machining or scurfing, by chemical milling or by immersion in acid pickling solutions in accordance with **3.6** (method F).

With the exception of castings which are to be machined all over, all castings should then be cleaned by fluoride anodizing in accordance with **3.30** (method EE).

Unless the subsequent chromate treatment is to be carried out in an acid bath capable of removing it, the fluoride film left by the cleaning process should then be removed by immersing the parts for up to 15 min in a boiling aqueous solution containing 100 g/l to 150 g/l of chromic acid (CrO_3) followed by immersion either for 10 min in a boiling aqueous solution containing 50 g/l of caustic soda or for 5 min in a cold aqueous solution containing 150 g/l to 200 g/l of hydrofluoric acid.

WARNING. Rigorous safeguards are needed for operators using hydrofluoric acid.

NOTE After this treatment, the parts are ready for conversion coating and the application of any agreed organic finishes.

2.10.3.3 Finish machined castings

Finished machined castings should be degreased, if necessary, in an organic solvent, e.g. trichloroethylene in accordance with **3.1.1** (method A.1) and then should be thoroughly cleaned in a suitable alkaline solution in accordance with **3.2** (method B).

NOTE After this treatment, the castings are ready for chromate treatment and the application of any agreed organic finishes.

2.10.3.4 Forgings

With the exception of parts which are to be machined all over, all forgings should be cleaned by fluoride anodizing in accordance with **3.30** (method EE). The fluoride film should be removed in accordance with **2.10.3.2** and then chromate treated and if required, coated with agreed organic finishes.

2.10.3.5 Sheet and extrusions

Sheet and extrusions should be degreased, if necessary, in an organic solvent, e.g. trichloroethylene, in accordance with **3.1.1** (method A.1), and then cleaned in a suitable alkaline solution in accordance with **3.2** (method B). The parts should then be further cleaned by fluoride anodizing in accordance with **3.30** (method EE), or by scouring with pumice powder applied with a moist rag in accordance with **3.4** (method D.3).

NOTE After this treatment the parts are ready for conversion coating and the application of any agreed organic finishes.

2.11 Zinc and its alloys

2.11.1 General cleaning

Unless otherwise required, degreasing should be carried out in accordance with **3.1.1** or **3.1.2** and/or **3.2.3** (methods A.1, A.2 and/or B.2). As die-cast components may contain subsurface porosity and any process such as acid etching which might penetrate the non-porous surface skin will reveal this porosity with consequent entrapment of corrosive residues, such processes should therefore be avoided during all stages of manufacture.

2.11.2 Cleaning of corroded parts

A recommended method of removing corrosion products is by immersion in a solution containing about 100 g/l of chromic acid at 90 °C. Corrosion of pressure die-cast zinc alloys during storage or service may reveal subsurface porosity, and any cleaning process leaving corrosive residues should be avoided (see **2.11.1**).

2.11.3 Preparation of zinc alloy die-castings for electroplating

Preparatory treatment should be in accordance with **3.25** (method Z).

2.11.4 Preparation of zinc alloy castings and zinc-coated articles for chromate passivation or phosphate treatment

The preparation of zinc alloy castings and zinc-coated articles for chromate passivation or phosphate treatment should be carried out as follows.

- a) Freshly zinc-coated components including those which have been baked for relief of hydrogen embrittlement or other reason and have not been contaminated with oil or grease (e.g. from handling) do not normally require degreasing.
- b) Zinc-coated parts which are not freshly coated and zinc alloy die-castings should be degreased in an organic solvent in accordance with **3.1** (method A), or cleaned in an alkaline solution in accordance with **3.2.3** (method B.2).

2.11.5 Preparation for painting

When parts are to be treated with a pretreatment primer (etching primer), they should be cleaned in accordance with **3.1** and/or **3.2.3** (methods A and/or B.2). When other primers are to be used, the zinc surfaces should normally be given a chemical conversion coating such as a chromate passivation or phosphate treatment.

2.12 Cadmium-coated articles

2.12.1 Preparation for chromate passivation

Cadmium-coated articles should be prepared in accordance with **2.11.4**.

2.12.2 Preparation for painting

Cadmium-coated articles should be prepared in accordance with **2.11.5**.

2.13 Tin and its alloys

2.13.1 Preparation for electroplating

Preparatory treatment should be in accordance with **3.26** (method AA).

2.13.2 Preparation for painting

Parts should be degreased in an organic solvent in accordance with **3.1** (method A).

2.14 Chromium-coated articles

Electroplated coatings of chromium should be prepared for painting by immersion in a solution of approximately 10 g/l of chromic acid (CrO_3).

The solution should be used at room temperature with an immersion time of approximately 5 min, but this is not critical. Parts should be washed in cold water, dried and painted.

Chromium-chromium oxide coatings require no pretreatment for painting and lacquering but such operations should immediately follow the production of the coating itself.

2.15 Lead and its alloys

Parts should be prepared for painting by degreasing with an organic solvent in accordance with **3.1** (method A), and then coated with a pretreatment primer (etching primer).

Section 3. Methods of cleaning and preparation

NOTE 1 The following methods for the pretreatment of metals are indicative of those most commonly used in practice. There are, in addition, numerous proprietary products.

NOTE 2 Rinsing is essential after each treatment in an aqueous solution. Rinsing is frequently carried out with hot and cold water. The use of demineralized water especially for the final dip may help to avoid contamination and staining problems. To avoid repetition, however, these rinsing operations are not mentioned in the methods described in this standard, except where special sequences are required.

NOTE 3 The expressions "ml/l" and "g/l" mean, respectively, millilitres and grams made up into 1 l of solution.

3.1 Method A. Organic solvent cleaning⁷⁾

3.1.1 Method A.1. Hot solvent (not water-rinsable)

NOTE The hot solvent (not water-rinsable) method covers liquid and vapour degreasing in hot hydrocarbon (including halogenated) solvents.

The grades of solvent should be known to be stable for this use. Halogenated hydrocarbons may decompose by hydrolysis if water is present or by other processes to yield mineral acids such as hydrochloric acid. Any solvents used need to be materials permitted by current legislation.

Attention should be paid to the heat capacity of the component, e.g. heavily contaminated thin sheet may not degrease satisfactorily with one treatment in vapour; spraying with clean condensed solvent after vapour treatment or immersion in boiling liquid is preferred in such cases.

Boiling liquid and vapour cleaning plants should be operated and maintained in accordance with the supplier's instructions and current legislation.

3.1.2 Method A.2. Cold solvent (not water-rinsable)

The cold solvent (not water-rinsable) cleaners should be used by immersion, spray, or hand application. The choice of solvent should be largely governed by considerations of toxicity, volatility, and flammability. Suitable cleaners such as the following should be used:

- a) trichloroethylene;
- b) perchloroethylene;
- c) white spirit conforming to BS 245:1976;
- d) white spirit/solvent naphthas;
- e) kerosene;
- f) terpenes;

NOTE Terpenes are currently being recommended as a possible replacement for chlorinated hydrocarbons. They are often used mixed with other hydrocarbons such as white spirit, kerosene and distillate with or without the addition of emulsifying agents.

3.1.3 Method A.3. Hot solvent (water-rinsable)

The hot solvent (water-rinsable) method should be used for paint removal or for assisting in the removal of carbonaceous deposits, and is based on an emulsifiable blend of cresylic acid and *o*-dichlorobenzene. It should be used with a water seal in a bath operated at temperatures up to 60 °C.

3.1.4 Method A.4. Cold solvent (water-rinsable)

Dichloromethane-based mixtures should be used for the removal of paint and carbonaceous deposits but they also act as effective degreasants. They should contain a minimum of 70 % (*m/m*) of dichloromethane conforming to BS 1994:1953.

Thickening and emulsifying agents should be added to assist contact, to delay evaporation and drainage, and to facilitate free rinsing.

NOTE Corrosion inhibitors may also be added for applications on close tolerance parts or where slight etching of the metal surface is undesirable. They may be formulated for brush application or for immersion. Some immersion processes also contain cresylic acid or other materials for increased efficiency and are usually used under a water seal.

3.1.5 Method A.5. Hydrocarbon-based mixtures containing emulsifying agents

The hydrocarbon-based mixtures containing emulsifying agents should be used for the substantial removal of heavy deposits of oil or grease. Application should be by brush or spray or dip with subsequent water rinsing. Hydrocarbons, e.g. white spirit conforming to BS 245:1976, kerosene or terpenes, e.g. limonene, or a mixture of any of these, with an emulsifying agent (one or more biodegradable synthetic surface active agents), are recommended.

3.2 Method B. Alkaline cleaning

3.2.1 General

Methods B.1 and B.2 (see 3.2.2 and 3.2.3) should be used for complete degreasing or in addition to preliminary solvent degreasing.

NOTE Methods B.1 and B.2 have 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 sequestering, complexing and surface active agents.

⁷⁾ For complete removal of all residual contamination these procedures may be followed by further cleansing in accordance with 3.2 (method B).

The ingredients should be selected from sodium or potassium hydroxide, silicates, carbonates, phosphates, borates, complexing agents (such as ethylene diaminetetra-acetic acid (EDTA), gluconates, heptonates, polyphosphates and cyanides) and organic surfactants. They should be used hot or cold with or without applied current which may be either anodic or cathodic. There should be very thorough washing, and possibly neutralization to remove residual traces of alkaline cleaners; this is specially important prior to painting.

3.2.2 Method B.1. Strongly alkaline cleaners

NOTE 1 These cleaners may contain any of the constituents given in 3.2.1. Heavy duty types may contain a high proportion of caustic alkali.

Strongly alkaline cleaners can be used as soak or spray cleaners or used electrolytically, preferably anodically, and are suitable for use on all metals except aluminium, lead, tin and zinc, each of which is attacked by strong alkalis. Simultaneous cleaning and etching of aluminium in caustic soda or other alkaline solutions should be employed only when agreed between the parties and should be strictly controlled.

NOTE 2 Strongly alkaline cleaners are preferred for the removal of metallic soaps.

3.2.3 Method B.2. Mild alkaline cleaners

NOTE 1 Mild alkaline cleaners are suitable for most metals. They are essentially free from strong alkali and may contain inhibitors such as silicates against metal attack.

Mild alkaline cleaners can be used by soak or spray.

NOTE 2 Applied current may be used for immersion applications but is less effective than with strongly alkaline cleaners.

3.3 Method C. Emulsion cleaning

NOTE Emulsion cleaners consist of a solvent, usually a hydrocarbon such as kerosene, emulsified in water with a solvent content of around 2 % (V/V) to 5 % (V/V).

Emulsion cleaners should be used cold or warm by spray or dip but are most effective when used by spray. Emulsion cleaners should normally be followed by a water rinse, but this may be omitted for inter-operational cleaning where a subsequent cleaning operation is employed, or to leave a temporary protective film.

3.4 Method D. Abrasive cleaning

NOTE 1 It is essential that residual abrasive is removed from parts treated by any of the methods given in 3.4.1 to 3.4.4 as it may interfere seriously with subsequent treatment.

NOTE 2 To stop the abrasive media from becoming contaminated by oil or grease, all parts should be precleaned in accordance with 3.1 to 3.3 (methods A to C).

3.4.1 Method D.1. Coarse blast-cleaning

Coarse blast-cleaning should be carried out by blasting with a suitable abrasive material, e.g. steel grit or chilled iron grit, alumina, chopped wire or other coarse abrasive of a grit size covered by, or comparable with, grade G17 of BS 2451:1963 or larger, when tested according to BS 7079-C2:1989 the profile should meet the coarse limit. Where blasting is a pretreatment for metal spraying to give good adhesion of a coating, grade G24 grit as defined in BS 2451:1963, or its equivalent is recommended.

Abrasive blast-cleaning is normally carried out by projecting particles of shot or grit at high speed onto the metal surface by compressed air or centrifugal force. Typical abrasives are either *spherical* (shot) of cast steel to produce rounded surface profiles or *angular* (grit) either of metallic particles, e.g. chilled iron grit, chopped wire or non-metallic particles, such as slag grit or alumina, to produce an angular surface profile.

Shot blast abrasives are used particularly in shot blast plants in preparation of surfaces for paints and thin film coatings. Grit blast abrasives are mainly used for metal (thermal) sprayed coatings to ensure good mechanical adhesion, however sometimes they may be used for paint primers where adhesion can be a problem.

NOTE 1 It is essential that the abrasives used should be clean, dry and free from soluble salts. The compressed air, where used, should be clean also. The adhesion of metal sprayed coatings depends upon a clean, angular profile of the surface to be coated.

NOTE 2 The process is unsuitable for thin material (generally less than 4 mm thick), small threaded fasteners or where a fine surface finish is required.

Non-metallic abrasives are recommended for metals other than non-corrosion-resisting types. If metallic grit or shot is used for stainless steels (as used for some processes), then an appropriate pickling process is essential as secondary cleaning to remove iron contamination and repassivate the surface (see 3.12.1, method M.1). On stainless steels and on aluminium and aluminium alloys, non-metallic abrasives such as alumina, glass beads, etc. should be used.

When this method is used on steel surfaces prior to metal spraying, the recommendations given in 2.3.7 should be followed. When it is used on aluminium alloys prior to metal spraying (see 2.6.8) the following should be considered.

- a) Care should be taken to avoid using abrasives which are insufficiently coarse or which have broken down with use in recirculating blast cabinets without adequate separation of fines and dust. The coarseness and angularity of the abrasive used have a great effect on the adhesion of the coating.

b) The alumina abrasive used should not have been used on any other type of materials, e.g. steel and copper-rich alloys, or have been otherwise contaminated.

c) Immediately after abrasive blast-cleaning, residual abrasive should be removed from the metal surfaces by blowing with clean, dry air or by vacuum hose unless instantaneous vacuum recovery of abrasive is employed.

3.4.2 Method D.2. Fine abrasive blast-cleaning

The fine abrasive blast-cleaning method should be as given in 3.4.1 (method D.1), but using non-metallic abrasives, e.g. alumina, glass beads, corn husks, using dry or compressed air, wet abrasive blast-cleaning to produce a surface profile in accordance with fine grade of BS 7079-C2:1989.

NOTE This method is particularly useful for cleaning lightly corroded parts.

3.4.3 Method D.3. Scouring

Scouring should be carried out by rubbing or scrubbing with moistened pumice, alumina or other suitable non-metallic abrasive.

3.4.4 Method D.4. Tumbling

Tumbling should be carried out with a suitable abrasive in special plant.

NOTE Corners and edges may become rounded or deburred in the process.

3.4.5 Method D.5. Wire brushing

Brushing by hand or mechanical methods should be used without supplementary abrasives.

Compatibility between the brush material and the material being treated should be considered carefully, e.g. the use of non-corrosion-resisting steel brushes with corrosion-resisting steels should be avoided.

3.5 Method E. Caustic alkali descaling processes

3.5.1 Method E.1. Sodium hydride descaling

Sodium hydride descaling has less tendency to induce hydrogen embrittlement in steel than pickling, but its use on high strength steel should be subject to agreement with the purchaser. This process is particularly suitable for castings, and for parts in heat resisting steel stress relieved after fabrication. Molten hydride is, however, liable to embrittle titanium alloys by hydrogen absorption.

Preheat the items to 300 °C and immerse for up to 10 min in the following solution at 350 °C to 370 °C:

sodium hydroxide	98 % <i>m/m</i> ;
sodium hydride	2 % <i>m/m</i>

Remove from the solution and quench in cold running water and give a final rinse in hot water.

3.5.2 Method E.2. Alkali permanganate descaling

Scale on iron and on non-corrosion-resisting steels should be removed by immersion in an aqueous solution containing:

sodium hydroxide	200 g/l to 250 g/l;
potassium permanganate	10 g/l to 20 g/l.

The time of immersion should depend upon the degree of scale. Following descaling there should be adequate rinsing and a short immersion in 10 % (*m/m*) hydrochloric acid (preferably inhibited) to remove any remaining loosened scale.

NOTE Potassium permanganate will decompose in a hot alkaline solution, even when not in use, and should be replaced on a regular basis; an excess is not harmful.

3.6 Method F. Acid pickling

The parts should be immersed in one of the following aqueous solutions.

Method F.1. 30 ml/l to 50 ml/l of sulfuric acid ($d = 1.84$ g/ml) with or without inhibitors or wetting agents. Use at 60 °C to 85 °C. For pickling stainless steels the concentration of sulfuric acid should be increased to 200 ml/l.

Method F.2. 200 ml/l of hydrochloric acid ($d = 1.16$ g/ml) with or without inhibitors or wetting agents. Use at room temperature to 40 °C.

Method F.3. Phosphoric acid solutions with or without inhibitors and/or wetting agents [phosphoric acid $d = 1.70$ g/ml, 15 % (V/V) to 20 % (V/V)]. Use at 60 °C to 80 °C. For the removal of brazing fluxes more dilute solutions of phosphoric acid may be used.

Method F.4. Aqueous acid solutions not given in this standard but agreed between the parties.

Method F.5. The following solution is typical of that used for removing light scale from austenitic stainless steels:

ferric sulfate liquor [40 % (<i>m/m</i>) ($d = 1.5$ g/ml)	200 ml/l to 300 ml/l;
hydrofluoric acid [40 % (<i>m/m</i>)] ^a	50 ml/l to 75 ml/l;
temperature:	60 °C to 70 °C;
immersion time:	2 min to 30 min.

^a See the warning in the foreword concerning the handling of hydrofluoric acid.

NOTE 1 Other solutions containing dichromates, mixed acids, etc. may be used by agreement.

NOTE 2 Pastes may be made from the ferric sulphate solution with barium powder and may be left in contact with localized scale overnight. Thorough rinsing and removal of residues is essential.

Method F.6. Nitric acid ($d = 1.42 \text{ g/ml}$) 100 ml/l with hydrofluoric acid [40 % (m/m)]⁸⁾ 50 ml/l.

Temperature: 60 °C to 70 °C.

Immersion time: 2 min to 30 min.

NOTE 1 If the parts are to be subsequently chemically smoothed see (3.11 (method L), phosphated or electroplated, it may be necessary to follow the use of pickling solutions containing wetting agents or inhibitors by acid dipping (see 3.6 (methods F1 or F2 without inhibitors or wetting agents) or by dipping in a suitable alkaline solution, in order to remove absorbed films.

NOTE 2 The presence of arsenic, reduced sulfur or reduced phosphorus compounds in an acid bath will promote absorption of hydrogen by steel and may reduce the adhesion of subsequently applied coatings.

3.7 Method G. Neutral cleaners

NOTE These materials, usually proprietary, contain combinations of wetting agents and inhibitors and anti-foams and are safe for use on any substrate.

Neutral cleaners should be employed by spray in purpose built power washes, and should be used for interstage cleaning prior to handling or inspection operations or for final cleaning before assembly. They should be employed at 1 % (V/V) to 2 % (V/V) in water with no subsequent rinsing but with an air blow to give a stain-free appearance.

3.8 Method H. Anodic etching

3.8.1 Method H.1

The parts should be immersed in a 300 ml/l aqueous solution of sulfuric acid of density 1.84 g/ml.

The solution strength should be maintained by periodic additions of sulfuric acid to keep the density greater than 1.30 g/ml.

The voltage should be set so that the initial current density is not less than 10 A/dm² (4 V to 8 V) and should preferably be twice this. The temperature should preferably 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 should be added, if desired, to reduce formation of smut and absorption of hydrogen by the steel, but wetting agents should be avoided as they promote absorption of hydrogen.

NOTE Removal of scale can be made easier by prior soaking in an alkaline solution of sodium gluconate at temperatures up to boiling point. The current density needed to remove scale can be reduced by interrupting the current for a few seconds from time to time. If current interruption is to be used on steels of tensile strength 1 430 MPa and stronger, a safe procedure should be established to the satisfaction of the purchaser.

3.8.2 Method H.2

The parts should be immersed in an aqueous solution ($d = 1.74 \text{ g/ml}$) containing sulfuric acid ($d = 1.84 \text{ g/ml}$), 750 ml/l with or without a small addition of chromic acid.

The solution strength should be maintained by periodic additions of sulfuric acid to keep the density not less than 1.70 g/ml.

An initial current density of at least 10 A/dm² (4 V to 12 V) should be applied. The temperature should not exceed 25 °C.

3.8.3 Method H.3

An alternative aqueous solution ($d = 1.22 \text{ g/ml}$) for use on stainless steel prior to a nickel strike [see 3.20 (method U)], should contain sulfuric acid ($d = 1.84 \text{ g/ml}$), 200 ml/l.

The solution strength should be maintained by periodic additions of sulfuric acid to keep the density not less than 1.20 g/ml.

A current density of 20 A/dm² to 25 A/dm² (approximately 6 V) should be applied. The temperature should not exceed 20 °C.

The immersion time should be 1 min to 3 min.

3.8.4 General considerations for anodic etching

The following points should be considered.

- a) Contamination of the solutions with chloride should be avoided.
- b) The essential purpose of anodic etching of steel, is to render the steel passive, a condition that will be indicated by:
 - 1) a sharp rise in voltage between the part and the cathode;
 - 2) a corresponding fall in current; and
 - 3) the onset of gas evolution from the part.
- c) Passivity should be maintained until the surface has acquired a light grey colour, free from dark smut.
- d) The parts may be withdrawn for examination, and if necessary, replaced in the bath for further treatment.
- e) Anodic etching may not be effective and may cause pitting on the inner surfaces of tubular parts unless auxiliary cathodes are employed. Where it is desired to clean only the external surfaces, the part should be plugged to prevent ingress of the electrolyte.

⁸⁾ See the warning in the foreword concerning the handling of hydrofluoric acid.

3.9 Method J. Acid derusting

Rust should be removed by any of the acid pickling procedures described in 3.6 (methods F.1 to F.4).

3.10 Method K. Alkaline derusting

3.10.1 General

Rust should be removed and the surface should be prepared for electroplating by either of the methods given in 3.10.2 and 3.10.3 (methods K.1 and K.2), as appropriate.

3.10.2 Method K.1. Light rust

Immerse in a solution based on caustic soda and a chelating agent such as sodium heptonate or gluconate.

3.10.3 Method K.2. Heavy rust

Subject the part to electrolytic treatment in an alkaline solution consisting essentially of sodium hydroxide in water, the concentration being adequate to ensure good electrical conductivity.

NOTE 1 The solution may contain substances which assist in the removal of the rust, e.g. sodium cyanide and ethylenediamine tetracetic acid. Typical compositions which should be used are as follows:

a)	sodium hydroxide	200 g/l to 300 g/l
	sodium cyanide	20 g/l to 30 g/l;
b)	sodium hydroxide	100 g/l to 200 g/l
	ethylenediamine	100 g/l to 150 g/l;
	tetracetic acid	
c)	sodium hydroxide	200 g/l to 300 g/l
	sodium gluconate	50 g/l to 100 g/l

NOTE 2 Suitable stable wetting agents may be added to the above formulations with advantage.

The solutions may be operated from room temperature up to 100 °C, but it is recommended that the solution is given in item a) of note 1 is not taken above 40 °C to prevent rapid decomposition of the cyanide.

The parts should be treated anodically or cathodically or with periodic reversal of the current at current densities of at least 2.5 A/dm².

After derusting, parts should be washed thoroughly in running water, special attention being paid to any crevices.

When residual alkali on the surface is undesirable, e.g. on surfaces to be phosphated or painted, the cold water wash should be followed by a rinse in phosphoric acid solution (normally a maximum of 0.5 % (V/V) is recommended).

3.11 Method L. Treatment of corrosion-resisting steel parts for electropolishing

For electropolishing corrosion-resisting steel (martensitic, ferritic or austenitic) parts should be treated using either of the following mixtures:

- | | | |
|----|----------------------------------|---|
| a) | phosphoric acid | (<i>d</i> = 1.70 g/ml) 500 ml/l; |
| | sulfuric acid | (<i>d</i> = 1.84 g/m) 450 ml/l; |
| | temperature: | 70 °C to 90 °C; current density; 20 A/dm ² to 30 A/dm ² (with the parts anodic) |
| b) | phosphoric acid | 600 ml/l; |
| | (<i>d</i> = 1.70 g/m) | |
| | chromic acid (CrO ₃) | 150 g/l; |
| | temperature: | 30 °C to 80 °C; current density: 10 A/dm ² to 100 A/dm ² (with the parts anodic). |

3.12 Method M. Passivation of corrosion-resisting steels

3.12.1 Method M.1. Passivation

Parts should be immersed for 10 min to 30 min at 20 °C to 50 °C in a solution made up to contain:

- nitric acid (*d* = 1.42 g/ml) 200 ml/l to 300 ml/l;
- sodium dichromate (Na₂Cr₂O₇·2H₂O) 25 g/l (which may be omitted when passivating austenitic stainless steels).

This treatment should be followed by rinsing in water and, in the case of ferritic and martensitic steels, by immersion for approximately 30 min in a solution containing 50 g/l of sodium dichromate at approximately 65 °C and final rinsing in water. The components should be tested for surface effectiveness of passivation in accordance with 3.12.2 or 3.12.3.

3.12.2 Method M.2. Test for surface effectiveness of passivation

Swab the component for a few minutes with an aqueous solution containing:

- copper sulfate 4 g/l (approximately); (CuSO₄·5H₂O)
- sulfuric acid 1.5 ml/l (approximately). (*d* = 1.84 g/ml)

After drying, the presence of any areas of deposited copper will confirm that the surface is unsatisfactory and that it should be re-passivated before being submitted for reapproval.

3.12.3 Method M.3. Alternative test for surface effectiveness of passivation

The alternative test should be carried out on a component, if suitable, or on a test piece with a flat smooth surface made from material of similar composition to the parts being processed.

Within 30 min of processing, place one spot of the following solution on a flat passivated surface and allow it to remain for 3 min.

palladium chloride	0.5 g;
hydrochloric acid ($d = 1.16$ g/ml)	20 ml;
water	98 ml.

Wash off the spot with cold running water. Do not swab.

No trace or only a slight trace of a dark deposit should be evident on the area tested to show that passivation has been effected.

3.13 Method N. Acid pickling of aluminium and aluminium alloy parts

Aqueous solutions of the following approximate compositions should be used for pickling degreased aluminium alloys and should be used prior to the final inspection of forgings.

<i>Method N.1.</i>	Sulfuric acid ($d = 1.84$ g/ml)	100 ml/l
	Sodium fluoride (NaF)	10 g/l
<i>Method N.2.</i>	Sulfuric acid ($d = 1.84$ g/ml)	100 ml/l
	Potassium fluoride (KF)	40 g/l
<i>Method N.3.</i>	Sulfuric acid ($d = 1.84$ g/ml)	100 ml/l
	Hydrofluoric acid [40 % (m/m)] ^b	15 ml/l
<i>Method N.4.</i>	Orthophosphoric acid ($d = 1.50$ g/ml) ^b	200 ml/l
	Hydrofluoric acid [40 % (m/m)] ^a	7.5 ml/l

^a For complete removal of all residual contamination these procedures may be followed by further cleansing in accordance with 3.2 (method B).

^b See the warning in the foreword concerning the handling of hydrofluoric acid.

Parts should be immersed in one of the solutions given in methods N.1 to N.4 at room temperature or a temperature not exceeding 50 °C until uniformly clean. They should be rinsed in cold water and transferred to a desmutting solution such as a cold aqueous solution containing approximately 500 ml/l nitric acid ($d = 1.42$ g/ml) for approximately 1 min and then thoroughly washed in clean water at a temperature not exceeding 50 °C.

Method N.5 Immerse the parts in the following solution at 90 °C to 98 °C until uniformly clean:

sulfuric acid ($d = 1.84$ g/ml)	100 ml/l;
<i>o</i> -toluidine	10 ml/l;

Method N.6. The parts should be immersed in a gently boiling aqueous solution of the following composition:

chromium(VI) oxide acid (CrO_3)	7.5 to 10 g/l;
phosphoric acid ($d = 1.75$ g/ml)	5 to 7.5 ml/l.

An immersion time of 20 min should normally be used for sheets, and up to 1 h for castings.

If the solution is contained in a metal tank, the parts should not be in electrical contact with the tank.

NOTE Suitable acid stable wetting agents may be added to the formulations given in this clause with advantage.

3.14 Method O. Etching treatment for aluminium and aluminium alloys (bath treatment)

3.14.1 The parts should be treated in accordance with items a) to e).

NOTE 1 This treatment may cause pitting on alloys containing 6 % (m/m) or more of copper and is generally unsuitable for use with composite parts in which aluminium is in contact with other metals, particularly steel and copper alloys. It is not suitable for castings containing more than 2 % (m/m) silicon.

NOTE 2 7XXX series aluminium alloys in the over aged temper can suffer significant intergranular attack in this solution, particularly at elevated temperatures and prolonged immersion times. Fatigue critical components should therefore be processed to minimize any intergranular attack.

a) The parts should be given preliminary solvent cleaning in accordance with 3.1 (method A), and should then be immersed in the following etching solution under the appropriate conditions given in item c).

b) The etching solution should consist of demineralized water containing: sulfuric acid ($d = 1.84$ g/ml) 150 ml/l (approximately) and either chromium(VI) oxide acid (CrO_3) 50 g/l (approximately) or sodium dichromate 80 g/l (approximately).

c) The temperature of the etching solution and the time of immersion of the parts should be appropriate to the subsequent treatment of the parts and should conform to the values given in Table 1.

Table 1 — Etching treatment conditions

Subsequent treatment	Temperature °C	Approximate time min
Painting, anodizing, filming	50 to 65	20
Adhesive bonding, electroplating	60 to 65	30

d) If the etching solution is contained in a metal (e.g. lead or stainless steel) tank, the parts should not be in electrical contact with the tank.

e) The etched parts should be washed thoroughly and as rapidly as possible in cold water. Parts for subsequent painting or bonding should then be washed in water at a temperature not exceeding 65 °C and dried. The final rinse should preferably be in soft or demineralized or distilled water, particularly before painting or bonding. Parts to be bonded should be dried for the minimum time necessary in warm air at a temperature not exceeding 65 °C. The subsequent treatment should then be applied with the least possible delay. The etched surfaces should not be touched with bare hands or be otherwise contaminated. The etched surfaces should be of uniform appearance and free from any accretions, deposits, stains or pitting.

3.14.2 The etching solution should be controlled as follows.

a) The volume of the solution should be kept constant by additions of distilled or demineralized water.

b) The chromium (VI) oxide content of the solution should not be allowed to fall below 30 g/l. The contents of chromic acid and sulfuric acid in the solution should be determined by chemical methods at intervals in relation to the rate of use of the solution. The solution should be regenerated by the appropriate additions of sulfuric and chromic acids.

c) The solution should be discarded when the density attains a value of 1.38 g/ml, when solid material begins to separate out on cooling the solution to room temperature, or at the first signs of pitting, whichever occurs first.

d) Contamination of the solution with chloride, copper or iron may cause pitting of the metal. The chloride content should not exceed the equivalent of 0.2 g/l sodium chloride and the copper or iron contents should not exceed 1 g/l each.

3.15 Method P. Etching treatment for aluminium and aluminium alloys

3.15.1 Method P.1. Etching by brush or spray treatment

Where parts are unsuitable for the immersion treatment, a pretreatment solution should be applied by brushing or spraying. This should consist of phosphoric acid in aqueous alcohols in which are suspended kaolin and a green pigment. The free acidity should be equivalent to 6.0 % (m/m) to 6.5 % (m/m) of phosphoric acid.

3.15.2 Method P.2. Etching and desmutting treatment

The etching and desmutting method should be used prior to anodizing if a polished finish is not required.

The degreased metal should be immersed in the following solution:

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

The solution should be operated at 60 °C to 65 °C.

NOTE 1 The reaction is very vigorous and immersion times from 15 s to 1 min are usually adequate.

A smut from alloying metals in the aluminium is left on the surface and this should be removed by dipping in 30 % (V/V) to 50 % (V/V) nitric acid of density = 1.42 g/ml.

NOTE 2 A finer and brighter etch is produced at the upper end of the sodium hydroxide concentration range and even higher concentrations can be used if there is adequate fume extraction.

NOTE 3 For high silicon-containing alloys 10 % (m/m) hydrofluoric acid [40 % (m/m)] is added to the nitric acid desmutting solution. HF High silicon castings [10 % (m/m) to 14 % (m/m)] cannot be satisfactorily desmuted and the casting should merely be vapour degreased prior to anodizing.

NOTE 4 Many proprietary materials are available which may be used instead of the solution recommended in this clause.

NOTE 5 Proprietary desmuts are available which may be safer in use than nitric acid.

3.16 Method Q. Cleaning etch for copper and its alloys

3.16.1 Method Q.1. Cleaning by immersion in dichromate-sulfuric acid solution

Parts should be immersed for up to 2 min at room temperature in an aqueous dichromate-sulfuric acid solution, a typical composition being:

sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$)	200 g/l;
sulfuric acid ($d = 1.84 \text{ g/ml}$)	40 ml/l.

3.16.2 Method Q.2. Cleaning by immersion in ferric sulfate-sulfuric acid solution

The parts should be immersed in the following solution:

ferric sulfate	125 g/l;
sulfuric acid ($d = 1.84 \text{ g/ml}$)	50 ml/l

Use the solution at a temperature between 20 °C and 50 °C.

3.17 Method R. Acid pickling of copper and its alloys

3.17.1 Method R.1. Scale dip

The parts should be immersed in an aqueous solution containing approximately 100 ml of sulfuric acid ($d = 1.84 \text{ g/ml}$).

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

3.17.2 Method R.2 Bright dip

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

The parts should be immersed in an aqueous solution containing approximately:

sulfuric acid ($d = 1.84 \text{ g/ml}$)	500 ml/l;
nitric acid ($d = 1.42 \text{ g/ml}$)	250 ml/l;
sodium chloride or hydrochloric acid	1 g/l or 1 ml/l respectively (this may be omitted).

The solution should be at room temperature and when it is heavily worked, cooling may be necessary. Efficient fume extraction is essential as the process results in the evolution of highly toxic fumes. After treatment the work should be adequately rinsed with a minimum of delay.

3.17.3 Method R.3. Chemical smoothing of copper and copper alloys

The parts should be treated as follows.

- Degrease in accordance with 3.1 (method A) and/or alkaline clean in accordance with 3.2 (method B).

b) Rinse thoroughly in cold water.

c) Immerse in an aqueous solution containing: hydrogen peroxide, 33 g/l to 50 g/l (e.g. 100 ml/l to 150 ml/l of "100 vols" hydrogen peroxide); sulfuric acid ($d = 1.84 \text{ g/ml}$), 1.3 ml/l.

Use at room temperature.

d) Immerse for 20 s in a solution of dilute sulfuric acid in accordance with 3.17.1 (method R.1) to remove the visible oxide film.

The time of treatment should be 15 min to 1 h depending on the application.

NOTE The rate of metal dissolution depends upon the metal being treated and the solution temperature, but is in the order of 13 $\mu\text{m/h}$. If the solution is used for continuous working the dissolved copper may be removed by circulation through a suitable ion exchange system.

3.17.4 Method R.4. Nitric acid pickle

The parts should be immersed in an aqueous solution containing 15 % (V/V) to 20 % (V/V) of commercial concentrated nitric acid ($d = 1.42 \text{ g/ml}$) at room temperature. Time of immersion should be 2 min to 5 min. Oxygen-containing copper which has been given a cathodic alkaline cleaning treatment should be given the minimum immersion time.

3.18 Method S. Acid pickling of titanium and its alloys

The parts should be immersed in an aqueous solution having one of the following nominal compositions.

<i>Method S.1.</i>	Hydrofluoric acid (40 % (m/m) ^a)	50 ml/l
	Nitric acid ($d = 1.42 \text{ g/ml}$)	not less than 200 ml/l

^a See the warning in the foreword concerning the handling of hydrofluoric acid.

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

<i>Method S.2.</i>	Hydrofluoric acid [40 % (m/m)] ^a	120 ml/l
	Nitric acid ($d = 1.42 \text{ g/ml}$)	400 ml/l

^a See the warning in the foreword concerning the handling of hydrofluoric acid.

Use at room temperature.

NOTE These solutions have to be handled with care. Efficient fume extraction is essential. The amounts of hydrofluoric acid quoted should not be exceeded.

3.19 Method T. Acid pickling of nickel and its alloys

Immerse the parts in one of the following solutions.

<i>Method T.1.</i>	Hydrofluoric acid [40 % (m/m)] ^a	50 ml/l
	Nitric acid ($d = 1.42$ g/ml)	not less than 200 ml/l

Use at a temperature not exceeding 65 °C.

<i>Method T.2.</i>	Hydrofluoric acid [40 % (m/m)] ^a	125 ml/l
	Ferric sulfate Fe ₂ (SO ₄) ₃ ·6H ₂ O	250 g/l

^a See the warning in the foreword concerning the handling of hydrofluoric acid.

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

NOTE Methods T.1 and T.2 may promote preferential attack on nickel alloys unless the alloys are in the solution heat-treated condition.

<i>Method T.3</i>	Sodium hydroxide	200 g/l
	Potassium permanganate	150 g/l

Use as hot as possible. After rinsing, loosened material should be removed in an acid solution such as 10 % (V/V) sulfuric acid solution.

3.20 Method U. Preparation of corrosion resisting steels for electroplating

One of the following treatments should be applied.

- a) Parts should preferably be etched anodically in accordance with 3.8 (method H) for not more than 2 min and then treated cathodically for 5 min at approximately 15 A/dm² to 20 A/dm² in an aqueous solution containing approximately:

nickel sulfate (NiSO ₄ ·6H ₂ O)	225 g/l;
sulfuric acid ($d = 1.84$ g/ml)	27 ml/l.

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

Insoluble anodes, e.g. lead, should normally be used; the use of nickel anodes or a proportion thereof is also permissible.

- b) The parts should be made anodic in an aqueous solution of the following approximate composition for not more than 2 min, where possible, and the current then reversed, so that they are cathodic, for about 5 min:

nickel chloride (NiCl ₂ ·6H ₂ O)	250 g/l;
hydrochloric acid ($d = 1.16$ g/ml)	100 ml/l

Nickel electrodes conforming to BS 558:1970 BS 564:1970 should be used. The solution should be maintained at room temperature and a current density of about 3 A/dm² should be employed.

- c) When current reversal is not feasible, the short anodic treatment described in item b) should be replaced by immersion in the solution without current flow for 15 min, the work then being made cathodic for about 5 min.

Separate tanks should preferably be used for the anodic (or immersion) and the cathodic treatments. After treatment in accordance with items a), b), or c), rinse the parts and transfer them to the final plating bath.

3.21 Method V. Preparation of aluminium alloys for electroplating

3.21.1 General

Parts to be electroplated should be treated either in a suitable proprietary process or in accordance with 3.21.2 to 3.21.8.

3.21.2 Degreasing

The parts should be degreased in an organic solvent in accordance with 3.1 (method A), and/or in an alkaline solution in accordance with 3.2.3 (method B.2).

3.21.3 Acid etching

The parts should be treated by one of the following methods.

- a) Pickle in an acid fluoride solution. The following treatment is recommended: immerse for 1 min at a temperature not exceeding 40 °C in an aqueous solution containing approximately 100 ml/l hydrofluoric acid [40 % (m/m)]⁹⁾ and 100 ml/l nitric acid ($d = 1.42$ g/ml).

Renew the solution when its action becomes sluggish.

Efficient fume extractions should be provided as this solution evolves toxic fumes.

⁹⁾ See the warning in the foreword concerning the handling of hydrofluoric acid.

b) Alternatively, etch in chromic acid/sulfuric acid solution, see 3.14 (method O). This treatment has been found to be only slightly less satisfactory than the acid fluoride treatment described in item a) for promoting adhesion, and, because of its less vigorous attack on the material, provides a smoother cleaned surface.

c) Carry out the alkaline etching and desmutting treatment in accordance with 3.15.2 (method P.2).

3.21.4 Rinsing

Rinse thoroughly and proceed immediately to the zincate treatment.

3.21.5 Zincate treatment

The following treatment should be carried out.

a) Immerse for 1 min in an aqueous solution containing approximately 500 ml/l nitric acid ($d = 1.42$ g/ml).

b) Rinse thoroughly.

c) Immerse for about 3 min at room temperature in an aqueous solution containing approximately:

zinc oxide (ZnO)	100 g/l;
sodium hydroxide (NaOH)	540 g/l.

d) Rinse thoroughly.

e) Repeat the operations in items a) to d).

3.21.6 Plating

Copper plating, in accordance with 3.21.7, should preferably then follow but electrodeposition of other metals may be carried out directly onto the zincate film.

3.21.7 Copper plating

Transfer the parts to a Rochelle copper plating bath and plate for 10 min at 1 A/dm^2 .

The following bath composition is recommended.

		Range (g/l)
sodium cyanide (NaCN)	38 g/l	30 to 45
copper cyanide (CuCN)	30 g/l	25 to 35
Rochelle salt ($\text{KNa}_4\text{C}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$)	50 g/l	40 to 60
sodium carbonate (Na_2CO_3)	38 g/l	25 to 50

The pH of this bath should be maintained between 9.5 and 10.5 (electrometric) and the temperature should be maintained at $(40 \pm 5) ^\circ\text{C}$.

It is important that the current connection should be made before the parts are immersed in this bath.

3.21.8 Rinsing

After copper plating, the articles should be rinsed thoroughly and transferred immediately, without drying, to the final plating bath. An intermediate dip in 5 % (m/m) sulfuric acid followed by a water rinse should be used prior to an acid plating bath.

3.22 Method W. Preparation of nickel alloys for electroplating

Parts should be degreased and should be given the following treatment.

a) Etch for a maximum of 1 min at room temperature in an aqueous solution containing:

either ferric chloride (FeCl_3)	150 g/l to 200 g/l;
or ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$)	250 g/l to 330 g/l;
hydrochloric acid ($d = 1.16$ g/ml)	150 ml/l to 200 ml/l.

b) The parts should be subjected to a nickel strike, as follows.

NOTE 1 This process is not usually required prior to chromium plating.

The parts should be made cathodic in an aqueous solution of the following approximate composition:

nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$)	250 g/l to 400 g/l;
hydrochloric acid ($d = 1.16$ g/ml)	100 ml/l.

The bath should be maintained at room temperature and nickel anodes should be used. A cathode current density of approximately 15 A/dm^2 should be maintained for not more than 2 min.

c) After the nickel strike rinse the parts thoroughly and transfer to the final plating bath. Surfaces previously nickel plated should be treated as the anode in a fresh 30 % (m/m) sulfuric acid solution at room temperature and a current density of approximately 20 A/dm^2 for 5 min. At the conclusion of this high current density treatment make the part cathodic for about 2 s by current reversal, remove from the solution, rinse thoroughly and transfer to the plating bath.

NOTE 2 An effective alternative to the procedure given in items a) and b) is given in 3.22 (method U).

3.23 Method X. Preparation of titanium and its alloys for electroplating

3.23.1 Method X.1¹⁰⁾

For plating some titanium alloys (e.g. Ti-2 % Cu; Ti-4 % Al, 4 % Mn; Ti-5 % Al, 2.5 % Sn; Ti-2.5 % Al, 11 % Sn, 4 % Mo and Ti-3 % Al, 11 % Cr, 13 % V), the following method is recommended.

a) After any preliminary vapour degreasing and/or descaling or pickling it is advisable to scour with pumice powder moistened with a solution of 50 g/l caustic soda (NaOH) and 50 g/l sodium carbonate (Na₂CO₃) and then to rinse and drain off excess water.

b) Etch in analytical reagent grade quality concentrated hydrochloric acid ($d = 1.18$ g/ml) at approximately 30 °C. For some titanium alloys an addition of 0.05 g/l chloroplatinic acid may improve the adhesion of the plated coatings. The optimum time of immersion depends on the composition of the alloy and may range from 10 min to 2 h. If the solution is maintained at 90 °C to 110 °C, an immersion time of approximately 5 min is adequate.

c) Transfer rapidly to a rinsing solution containing 50 g/l Rochelle salt (sodium potassium tartrate) at room temperature and agitate the work thoroughly in this solution.

d) Transfer without delay to a copper plating bath of the following approximate composition:

copper sulfate (CuSO ₄ ·5H ₂ O)	60 g/l;
Rochelle salt (KNaC ₄ H ₄ O ₆ ·4H ₂ O)	160 g/l;
sodium hydroxide (NaOH)	50 g/l.

Use at room temperature and plate at 0.4 A/dm² for 5 min, electrical contact being made before immersion of parts in the plating bath.

e) Rinse in either of the following:

- 1) dilute sulfuric acid [approximately 1 % (V/V)] if subsequent plating is to be from an acid plating bath (e.g. nickel or chromium);
- 2) approximately 5 % (m/m) sodium or potassium cyanide solution if subsequent plating is to be from a cyanide electroplating bath (e.g. silver).

NOTE Further electrodeposition by conventional methods may be applied to the thin copper coating. For maximum adhesion the parts should be heat treated at 450 °C for 1 h after final plating.

3.23.2 Method X.2

NOTE This method is suitable for plain titanium and a few of its alloys. It is not considered suitable for Ti-4 % Al, 4 % Mo, 2 – 4 % Sn, 0.5 % Si alloys.

The following procedure should be carried out.

- a) After any required degreasing and/or preliminary pickling or descaling, etch in hydrochloric acid ($d = 1.18$ g/ml) for approximately 1 h at room temperature or approximately 5 min at 90 °C to 110 °C.
- b) Rinse in cold water.
- c) Transfer to a nickel strike solution of the following composition used at room temperature:

		Range (g/l)
nickel chloride (NiCl ₂ ·6H ₂ O)	200 g/l	150 to 250
hydrochloric acid ($d = 1.16$ g/ml or $d = 1.18$ g/ml)	100 g/l	75 to 125

Use nickel anodes conforming to BS 558:1970 & BS 564:1970.

The cathode current density should be approximately 3 A/dm².

- d) Rinse in cold water.
- e) Electroplate with any desired metal.

3.24 Method Y. Preparation of magnesium for electroplating

The following procedure is recommended.

a) Clean cathodically for 1 min at room temperature in the following solution:

sodium hydroxide	30 g/l (approximately);
sodium cyanide	30 g/l (approximately);
sodium carbonate	15 g/l (approximately).

- b) Rinse.
- c) Pickle in phosphoric acid ($d = 1.75$ g/ml) at room temperature for 30 s.
- d) Rinse.

¹⁰⁾ Thicker copper plating is not advised since brittle intermetallic alloys may be formed between copper and titanium on any subsequent heat treatments.

e) Activate by dipping in the following solution at room temperature. The parts should remain in the solution for about 30 s after the initial gassing has ceased.

		Range
Phosphoric acid ($d = 1.75$ g/ml)	135 ml/l	130 ml/l to 150 ml/l.
Potassium fluoride	70 g/l	60 g/l to 80 g/l

f) Rinse.

g) Immerse in the following zinc solution at a temperature of 90 °C to 95 °C for 5 min:

		Range g/l
zinc sulfate $ZnSO_4 \cdot 7H_2O$	50 g/l	45 to 55
sodium pyrophosphate $Na_4P_2O_7$	180 g/l	150 to 200
potassium fluoride KF	6 g/l	4 to 8
sodium carbonate Na_2CO_3	4 g/l	3 to 10

The pH should be 10.6.

h) Rinse.

i) Plate in a cyanide copper solution to produce a deposit thickness of at least 5 μ m.

NOTE Electrical connections should preferably be made before the parts are immersed in the copper bath and they should enter the solution live.

The copper deposit should be followed by any other required finish.

3.25 Method Z. Preparation of zinc alloy for electroplating

The following procedure is recommended.

a) Degrease with hot organic solvent in accordance with 3.1.1 (method A.1), and/or an alkaline cleaner in accordance with 3.2 (method B).

It is strongly recommended that buffing compound is removed as soon as possible after polishing, as such materials become considerably more difficult to remove after ageing for several days, and may be hardened by hot solvents.

b) Clean, preferably anodically, in a mild alkaline cleaner in accordance with 3.2.3 (method B.2).

c) Rinse thoroughly in cold, running water.

d) Dip in dilute sulfuric acid [e.g. 0.5 % (V/V)] or hydrofluoric acid 1 % (m/m) to 1.5 % (m/m).

e) Rinse thoroughly in cold, running water.

f) Deposit at least 5 μ m of copper from a cyanide solution.

g) Rinse thoroughly in cold, running water.

h) Plate with other metals as required.

3.26 Method AA. Preparation of tin alloy for electroplating

The following procedure is recommended.

a) Degrease with hot organic solvent in accordance with 3.1.1 (method A.1).

b) Clean, either by immersion or electrolytically, in a mild alkaline cleaner in accordance with 3.2.3 (method B.2).

c) Rinse thoroughly in cold, running water.

d) Dip for approximately 2 min in a solution containing 10 % (V/V) of hydrochloric acid ($d = 1.18$ g/ml).

NOTE For tin alloys containing lead approximately 100 m/l fluoroboric acid [40 % (m/m)] may be substituted.

e) Rinse thoroughly in cold, running water.

f) Deposit approximately 5 μ m of copper from a cyanide solution.

g) Rinse thoroughly in cold, running water.

h) Plate with other metals as required.

3.27 Method BB. Chemical polishing of aluminium and its alloys

A recommended procedure is immersion in the following solution:

phosphoric acid ($d = 1.75$ g/ml)	75 % (V/V);
sulfuric acid ($d = 1.84$ g/ml)	20 % (V/V);
nitric acid ($d = 1.42$ g/ml)	5 % (V/V).

This solution is operated at 95 °C to 105 °C with an immersion time of up to 2 min. Nitric acid is lost from the solution by decomposition and should be maintained by analysis in the range 5 % (V/V) to 10 % (V/V). Other constituents are lost mainly by drag-out and the volume should be restored by additions of the original mixture.

NOTE 1 This process gives rise to the emission of nitrous fumes which have to be removed by adequate exhaust equipment. The solution is strongly acid and appropriate precautions should be taken to protect the operator.

After chemical polishing the work should be rinsed and further cleaned by immersion in a 30 % (V/V) solution of nitric acid ($d = 1.42$ g/ml) or 10 g/l chromic acid (CrO_3), followed by further rinsing.

NOTE 2 There are a number of proprietary solutions available, the compositions of which vary, that can be used to chemically polish aluminium alloys. Lower purity alloys (below 99.9 % (m/m) Al basis) are best treated with one of the proprietary solutions.

3.28 Method CC. Electrobrightening and electropolishing of aluminium and its alloys

3.28.1 Method CC.1. Electrobrightening

This method is suitable for super purity aluminium [99.99 % (*m/m*)] and alloys based on 99.99 % (*m/m*) aluminium.

The metal is electrolytically brightened by anodic treatment in the following aqueous solution:

		Range (g/l)
sodium carbonate (anhydrous)	150 g/l	120 to 200
trisodium phosphate (anhydrous)	50 g/l	25 to 75

temperature: 75 °C to 80 °C

d.c. voltage: approximately 20 V

NOTE The time taken in processing will depend upon the degree of brightening required and may be as long as 40 min. An average time of processing is 20 min.

After electrobrightening the metal should be rinsed and the thin oxide film formed during electrobrightening should be removed by immersion for approximately 2 min in the following solution:

phosphoric acid (<i>d</i> = 1.70 g/ml)	35 ml/l;
chromium(VI) oxide (CrO ₃)	20 g/l

temperature: approximately 95 °C.

3.28.2 Method CC.2. Electropolishing

NOTE 1 This type of process is applicable to a wide range of aluminium alloys with the exception of those with a silicon content exceeding 2 % (*m/m*).

The work should be treated anodically in an aqueous solution of the following composition:

		Range (g/l)
phosphoric acid (as H ₃ PO ₄)	650 g/l	400 to 800
sulfuric acid (<i>d</i> = 1.84 g/ml)	150 g/l	100 to 200
chromium(VI) oxide (CrO ₃)	60 g/l	40 to 100

temperature: 70 °C to 80 °C

current density: approximately 2 A/dm²

dc. voltage: 12 V to 15 V

NOTE 2 The processing time varies with the amount of metal that has to be removed to achieve the desired brightness but usually lies between 2 min and 5 min.

3.29 Method DD. Electropolishing of copper and its alloys

NOTE 1 Various solutions mainly based on phosphoric acid can be used for electropolishing copper and its alloys. Those which are most commonly used contain various aliphatic alcohols as additives. A recommended composition for electropolishing copper and its alloys is a 70 % (*m/m*) solution of orthophosphoric acid of density 1.75 g/ml in aliphatic alcohol and water.

Copper cathodes should be used, with a current density of 2 A/dm² to 500 A/m² at a temperature of 20 °C to 25 °C.

NOTE 2 The solution is very viscous and it is normally followed by a dilute drag-out solution made up with extra alcohol. The drag-out is used to top up the processing solution.

NOTE 3 There are various proprietary solutions available which vary in composition that can be used to electropolish copper alloys. The choice of proprietary solution used may depend upon the composition of the alloy to be electropolished.

3.30 Method EE. Fluoride anodizing process for cleaning magnesium alloys

NOTE The fluoride anodizing process is intended to supersede abrasive blasting as a method of cleaning magnesium alloy castings, or as a method of substantially restoring the corrosion resistance lost as a result of abrasive blasting. The process can be applied to all magnesium alloys in all forms.

3.30.1 Precleaning

Loosely held particles of foundry sand should be removed by tapping or brushing. On a part which is being reconditioned, grease or paint should be removed by solvent degreasing in accordance with 3.1 (method A), and/or immersion in a suitable alkaline metal cleaning solution in accordance with 3.2 (method B).

3.30.2 Composition of solution

The bath should consist of a solution of 150 g/l to 250 g/l ammonium bifluoride (NH₄HF₂) in water.

The solution should be contained in a vessel lined with hard rubber or suitable plastics material resistant to acid fluoride solution. It is essential that the inner wall of the vessel does not conduct electricity.

3.30.3 Operation and control

The following procedure should be carried out.

- The bath should be maintained at a composition of between 15 % (*m/m*) and 25 % (*m/m*) ammonium bifluoride by the addition of fresh quantities of this salt. The bath should be discarded if it has become contaminated with foreign metals, acid radicles other than fluoride, or organic matter.
- The temperature of the bath should not exceed 30 °C. The bath should be stirred with a pole of wood, ebonite or magnesium alloy.

c) Parts should normally be treated in pairs of approximately equal surface area using alternating current. The parts should be fixed in good electrical contact and suspended not less than 225 mm below the surface of the solution. All parts of the fixing clamps which extend into the solution should be made of magnesium alloy other than magnesium-manganese alloy. Apply alternating current and raise the voltage to 90 V to 120 V. Current flow is usually heavy at first, but diminishes rapidly as surface impurities are removed and an unbroken magnesium fluoride film is formed.

The treatment should be continued for 10 min to 15 min or until the current falls to below 0.5 A/dm^2 of the smaller electrode. The current should then be switched off and the parts removed from the bath, washed and examined.

3.30.4 Appearance after cleaning

The parts should have a uniform clean white or pearly grey appearance and be free from foundry sand.

NOTE Dark areas in hollows may indicate entrapment of gas during treatment. Very thin semi-transparent films indicate treatment at too low a voltage; only on machined surfaces or on wrought surfaces in good condition is such a film acceptable. An etched appearance indicates a bath too low in ammonium fluoride, too hot, or operated at too high a voltage. A thick film may indicate the presence of acid radicles other than fluoride. Surface pitting may be caused by chloride in the bath.

3.30.5 Safety precautions

Because of the high voltage used, access to the bath has to be prevented while the current is switched on. Spray from the bath is poisonous, and suitable ventilation should be provided.

3.31 Method FF. Chemical polishing of nickel and its alloys

A recommended solution is as follows:

glacial acetic acid	50 % (V/V)
nitric acid ($d = 1.42 \text{ g/ml}$)	30 % (V/V)
phosphoric acid ($d = 1.75 \text{ g/ml}$)	10 % (V/V)
sulfuric acid ($d = 1.84 \text{ g/ml}$)	10 % (V/V)

The solution should be used at 90°C and is therefore somewhat unpleasant to use owing to the rapid evolution of the acetic acid.

NOTE This evolution and consequential change of solution composition can be retarded by the use of glass croffles. The nitric acid content permits the safe use of stainless steel containers, and the preferred technique of using this solution is to have it in a stainless steel vat, and to treat the parts in a stainless steel basket, preferably with a basket top to prevent the pick-up of the croffles. Rapid agitation of the basket is helpful, and after a treatment time of 1 min to 3 min, rapid rinsing should be used. The nickel taken up in the solution precipitates as nickel sulfate, and the sulfuric acid content should be maintained by periodic additions of sulfuric acid.

3.32 Method GG. Electropolishing of nickel and its alloys

The parts should be treated anodically in an aqueous solution containing 70 % (V/V) sulfuric acid ($d = 1.84 \text{ g/ml}$) at room temperature. A current density of 2.5 A/dm^2 should be employed with the parts anodic to the lead lining of the tank.

Great care should be taken in making up this solution and the sulfuric acid has to be added slowly to the water with thorough mixing. The solution becomes hot during mixing and should be cooled to room temperature before use.

Annex A (informative) Stress relief of steel

A.1 General

NOTE 1 The relief of internal and deleterious surface stresses by a suitable heat treatment aids resistance to sustained load and fatigue failure.

As a rule, internal stresses developed during quenching should be relieved at temperatures of 400 °C and above, and surface stresses developed during mechanical finishing operations should be reduced by heating at temperatures of 200 °C and above. The relief of beneficial surface compressive stresses introduced during production, e.g. by shot-peening or surface rolling, should however be avoided. Stress relief should be applied before cleaning, although parts may if necessary be degreased before heat treatment.

NOTE 2 The stress relief treatment can be omitted for maraging steels which have been aged after final machining.

NOTE 3 These recommendations do not apply to parts which are to be welded.

A.2 Procedure for steels of tensile strength equal to and above 1 000 MPa

With the exception of surface hardened parts the heat treatment conditions should be selected on the basis of actual tensile strength. Parts made of steel with actual tensile strength greater than or equal to 1 000 MPa (corresponding to hardness values 310 HV, 31 HRC or 300 HB) and surface hardened parts should be heat treated. When only the minimum tensile strength is specified or the tensile strength is not known, the heat treatment condition should be selected by relating known or measured hardness values to equivalent actual strengths. Steels which have been wholly or partly surface hardened should be considered as being in the category appropriate to the hardness of the surface hardened layer.

The duration of heat treatment should commence from the time at which the whole of each part attains the specified temperature. Preparations involving cathodic treatments in alkaline or acid solutions should be avoided.

The stress relief treatment given in Table A.1 is applicable to those steels whose properties are not adversely affected by heat treatment at 200 °C or higher. The heat treatment should be carried out before commencement of any preparation or cleaning treatment using aqueous solutions, or before any treatment liable to cause embrittlement.

Table A.1 — Stress relief treatment applicable to steel whose properties are not adversely affected by heat treatment at 200 °C or higher

Actual tensile strength MPa	Temperature (°C)	Time (h)
1 000 to 1 400	200 to 230	minimum 3
1 401 to 1 800	200 to 230	minimum 18
Over 1 800	200 to 230	minimum 24

Suitable combinations of shorter time at appropriate higher temperature may be used if the treatment is not shown to be detrimental. For tempered steels, items should not be heat treated above a temperature which should be at least 50 °C below the tempering temperature.

If stress relief is given after shot peening or other cold working processes to introduce beneficial compressive stresses, the temperature should not exceed 230 °C. Items having surface hardened areas which would suffer an unacceptable reduction in hardness by treatment in accordance with Table 1 should be heat treated at a lower temperature but not less than 130 °C for a minimum period of 8 h. This treatment is applicable for parts made of steel with actual tensile strength below 1 400 MPa.

Annex B (normative) Method for determination of oxidizable material in sulfuric acid

Add 15 ml of concentrated sulfuric acid to 60 ml of water, cool, and to the mixture add 0.10 ml of a potassium permanganate solution containing 3.3 g/l potassium permanganate and stir. The pink colour should persist for 5 min indicating a presence of not more than 11 p.p.m. by volume oxidizable substances calculated as sulfur dioxide.

List of references (see clause 2)

Normative references

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BS EN 22063:1994, *Metallic and other inorganic coatings — Thermal spraying — Zinc, aluminium and their alloys.*

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[1] DEF STAN 03-2 *Cleaning and preparation of metal surfaces. Issue 3*¹¹⁾.

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[3] GREAT BRITAIN. Control of Substances Hazardous to Health Regulations, 1988. London: HMSO.

[4] THWAITES, C.J. *Practical hot-tinning*. Publication No 575. International Tin Research Institute¹²⁾.

[5] GALVANIZERS' ASSOCIATION *General galvanizing practice*¹³⁾.

¹¹⁾ In preparation. Available from Ministry of Defence, Directorate of Standardization, Kentigern House, 65 Brown St, Glasgow G2 8EX.

¹²⁾ Available from ITRA Ltd., c/o International Tin Research Institute, Kingston Lane, Uxbridge, Middlesex, UB8 3PJ.

¹³⁾ Available from the The Galvanizers' Association, Wren's Court, 56 Victoria Road, Sutton Coldfield, West Midlands B72 15Y.

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