
**Metallic and other inorganic coatings —
Cleaning and preparation of metal
surfaces —**

**Part 2:
Non-ferrous metals and alloys**

*Revêtements métalliques et autres revêtements inorganiques —
Nettoyage et préparation des surfaces métalliques —*

Partie 2: Métaux non ferreux et alliages



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.



COPYRIGHT PROTECTED DOCUMENT

© ISO 2008

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

| | |
|---|------|
| Foreword..... | vii |
| Introduction | viii |
| 1 Scope | 1 |
| 2 Normative references | 2 |
| 3 Terms and definitions..... | 3 |
| 4 Essential information to be supplied by the purchaser to the processor | 3 |
| 5 Requirements | 4 |
| 6 Standard cleaning methods..... | 5 |
| 7 Non-corrosion-resisting steels, cast irons and pure irons | 5 |
| 8 Corrosion-resisting and heat-resisting steels | 6 |
| 9 Pretreatment of metals prior to powder coating | 6 |
| 10 Cleaning and preparation processes..... | 6 |
| 11 Aluminium and aluminium alloys..... | 6 |
| 11.1 General..... | 6 |
| 11.2 General degreasing and cleaning | 6 |
| 11.3 Removal of corrosion products | 6 |
| 11.4 Preparation prior to anodizing | 7 |
| 11.5 Preparation prior to oxide removal treatment (process O) or filming treatment | 7 |
| 11.6 Preparation prior to adhesive bonding of unanodized items | 7 |
| 11.7 Preparation prior to electroplating..... | 7 |
| 11.8 Preparation prior to autocatalytic nickel plating | 7 |
| 11.9 Preparation prior to conversion coating | 7 |
| 11.10 Preparation prior to metal spraying..... | 7 |
| 11.11 Preparation prior to vitreous enamelling | 8 |
| 11.11.1 Sheet, extrusions and items made from casting alloys..... | 8 |
| 11.11.2 Other castings..... | 8 |
| 11.12 Preparation prior to supplementary treatments on physically vapour deposited aluminium..... | 8 |
| 12 Copper and copper alloys..... | 8 |
| 12.1 General degreasing and cleaning | 8 |
| 12.2 Preparation prior to electroplating..... | 9 |
| 12.3 Preparation prior to autocatalytic nickel plating | 9 |
| 12.4 Preparation prior to hot tinning (TRI process) | 10 |
| 12.5 Preparation prior to vitreous tinning | 10 |
| 13 Nickel alloys | 10 |
| 13.1 General degreasing and cleaning | 10 |
| 13.2 Preparation prior to electroplating..... | 10 |
| 13.3 Preparation prior to autocatalytic deposition | 11 |
| 13.4 Preparation prior to vitreous enamelling | 11 |
| 14 Titanium and titanium alloys | 11 |
| 14.1 General degreasing and cleaning | 11 |
| 14.2 Preparation prior to electroplating..... | 12 |
| 14.3 Preparation prior to autocatalytic nickel plating | 12 |
| 15 Magnesium alloys | 12 |
| 15.1 General degreasing and cleaning prior to electroplating and conversion coating | 12 |

| | | |
|--------|--|----|
| 15.2 | Preparation prior to electroplating | 12 |
| 15.3 | Preparation prior to conversion coating | 13 |
| 15.3.1 | General | 13 |
| 15.3.2 | As-cast castings | 13 |
| 15.3.3 | Machined castings | 14 |
| 15.3.4 | Forgings | 14 |
| 15.3.5 | Sheets and extrusions | 14 |
| 16 | Zinc-based alloys | 14 |
| 16.1 | General degreasing and cleaning | 14 |
| 16.2 | Cleaning of corroded items | 14 |
| 16.3 | Preparation of zinc alloy die castings prior to electroplating | 14 |
| 16.4 | Preparation of zinc alloy castings prior to chromate conversion coating or phosphate treatment | 14 |
| 17 | Tin and its alloys | 14 |
| 18 | Tungsten alloys | 15 |
| 18.1 | Preparation prior to electroplating | 15 |
| 18.2 | Preparation prior to adhesive bonding | 15 |
| 19 | Lead and its alloys | 15 |
| 20 | Zinc-, cadmium-, chromium- and gold-coated items | 15 |
| 20.1 | Preparation of zinc-coated items prior to chromate conversion coating | 15 |
| 20.2 | Preparation of cadmium-coated items prior to chromate conversion coating | 15 |
| 20.3 | Preparation of physically vapour deposited cadmium prior to supplementary treatments | 15 |
| 20.4 | Preparation for chromium-coated parts prior to supplementary treatments | 16 |
| 20.5 | Removal of surface contamination from gold coatings | 16 |
| 21 | Pretreatment of metals prior to powder coating | 16 |
| 21.1 | Preparation of aluminium prior to powder coating | 16 |
| 21.2 | Preparation of zinc and hot-dip-galvanized steel prior to powder coating | 16 |
| 21.3 | Preparation of copper and brass prior to powder coating | 16 |
| 22 | Cleaning and preparation processes | 16 |
| 22.1 | Process A — Organic-solvent degreasing and cleaning | 17 |
| 22.1.1 | General | 17 |
| 22.1.2 | Method A1 — Hot solvent, not water-rinsable | 17 |
| 22.1.3 | Method A2 — Cold solvent, not water-rinsable | 17 |
| 22.1.4 | Method A3 — Hot solvent, water-rinsable | 17 |
| 22.1.5 | Method A4 — Cold solvent, water-rinsable | 18 |
| 22.1.6 | Method A5 — Petroleum fuel based mixtures containing emulsifying agents | 18 |
| 22.1.7 | Method A6 — Ultrasonic cleaning | 18 |
| 22.2 | Process B — Alkaline degreasing | 18 |
| 22.2.1 | General | 18 |
| 22.2.2 | Method B1 — General-purpose alkaline degreasing | 18 |
| 22.2.3 | Method B2 — Mild alkaline degreasing | 18 |
| 22.2.4 | Method B3 — Cathodic alkaline degreasing | 19 |
| 22.3 | Process C — Acid emulsion cleaning | 19 |
| 22.4 | Process D — Abrasive cleaning | 19 |
| 22.4.1 | General | 19 |
| 22.4.2 | Method D1 — Coarse-abrasive blasting | 19 |
| 22.4.3 | Method D2 — Fine-abrasive blasting | 19 |
| 22.4.4 | Method D3 — Scouring | 19 |
| 22.4.5 | Method D4 — Tumbling | 20 |
| 22.4.6 | Method D5 — Wire brushing | 20 |
| 22.5 | Process E — Caustic-alkali descaling | 20 |
| 22.6 | Process F — Acid pickling without electrolysis | 20 |
| 22.7 | Process G — Acid dipping | 20 |
| 22.8 | Process H — Anodic pickling | 20 |
| 22.8.1 | General | 20 |
| 22.8.2 | Method H1 | 20 |

| | | |
|-----------|---|----|
| 22.9 | Process J — Acid de-rusting | 20 |
| 22.10 | Process K — Alkaline de-rusting | 21 |
| 22.11 | Process L — Chemical smoothing and electropolishing of steels | 21 |
| 22.12 | Process M — Passivation and removal of surface contamination from corrosion-resisting steels and surface preparation prior to electroplating | 21 |
| 22.13 | Process N — Acid pickling of aluminium alloys | 21 |
| 22.13.1 | General | 21 |
| 22.13.2 | Method N1 | 21 |
| 22.13.3 | Method N2 | 21 |
| 22.13.4 | Method N3 | 21 |
| 22.13.5 | Method N4 | 22 |
| 22.13.6 | Method N5 | 22 |
| 22.13.7 | Method N6 | 22 |
| 22.14 | Process O — Oxide removal, i.e. “etching”, treatment of aluminium and aluminium alloys | 22 |
| 22.14.1 | General | 22 |
| 22.14.2 | Degreasing and cleaning | 22 |
| 22.14.3 | Preparation prior to anodizing and adhesive bonding | 23 |
| 22.15 | Process P — Aluminium and aluminium alloys — Removal of corrosion products, etching treatment, chemical polishing, electrobrightening and electropolishing | 24 |
| 22.15.1 | Removal of corrosion products | 24 |
| 22.15.1.1 | Method P1 — Immersion | 24 |
| 22.15.1.2 | Method P2 — Swabbing | 24 |
| 22.15.2 | Etching treatment | 24 |
| 22.15.2.1 | Method P3 — Etching by brushing or spray treatment | 24 |
| 22.15.2.2 | Method P4 — Etching and de-smutting treatment using sodium hydroxide | 24 |
| 22.15.3 | Method P5 — Chemical polishing | 25 |
| 22.15.4 | Method P6 — Electrobrightening | 25 |
| 22.15.5 | Method P7 — Electropolishing | 26 |
| 22.16 | Process Q — Cleaning of copper and copper alloys | 26 |
| 22.16.1 | Method Q1 — Cleaning in dichromate/sulfuric acid solution | 26 |
| 22.16.2 | Method Q2 — Cleaning in ferric sulfate/sulfuric acid solution | 26 |
| 22.17 | Process R — Acid descaling, etching, chemical smoothing and electropolishing of copper and copper alloys | 26 |
| 22.17.1 | Method R1 — Descaling dip | 26 |
| 22.17.2 | Method R2 — Bright dip | 26 |
| 22.17.3 | Method R3 — Chemical smoothing | 27 |
| 22.17.4 | Method R4 — Pickling in nitric acid | 27 |
| 22.17.5 | Method R5 — Electropolishing | 27 |
| 22.18 | Process S — Pickling of titanium and titanium alloys | 27 |
| 22.18.1 | General | 27 |
| 22.18.2 | Method S1 | 27 |
| 22.18.3 | Method S2 | 27 |
| 22.19 | Process T — Pickling, chemical polishing and electropolishing of nickel and its alloys | 28 |
| 22.19.1 | General | 28 |
| 22.19.2 | Method T1 | 28 |
| 22.19.3 | Method T2 | 28 |
| 22.19.4 | Method T3 | 28 |
| 22.19.5 | Method T4 — Chemical polishing | 28 |
| 22.19.6 | Method T5 — Electropolishing | 29 |
| 22.20 | Process U — Preparation of tin alloy for electroplating | 29 |
| 22.21 | Process V — Removal of surface contamination from gold coatings | 29 |
| 22.22 | Process W — Preparation of aluminium alloys prior to electroplating | 29 |
| 22.23 | Process X — Preparation of nickel alloys prior to electroplating | 30 |
| 22.24 | Process Y — Preparation of titanium alloys prior to electroplating | 31 |
| 22.24.1 | Method Y1 — General method for titanium alloys | 31 |
| 22.24.2 | Method Y2 — Method for titanium and selected titanium alloys | 32 |
| 22.24.3 | Method Y3 — Chemical-etching method | 32 |
| 22.25 | Process Z — Preparation of zinc and zinc alloys prior to electroplating | 33 |
| 22.25.1 | Method Z1 | 33 |
| 22.25.2 | Method Z2 | 33 |

| | |
|--|-----------|
| Annex A (informative) Maintenance of organic and vapour degreasing baths | 34 |
| Annex B (informative) The control of acidity in vapour cleaning baths | 35 |
| Annex C (normative) Method for the determination of oxidizable material in sulfuric acid | 36 |
| Annex D (informative) Method for determining free acidity | 37 |
| Annex E (normative) Selection of method of cleaning according to the tensile strength of the steel in zinc-coated steel items prior to chromate conversion coating | 38 |
| Annex F (normative) Method M4 | 40 |
| Bibliography | 41 |

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 27831-2 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

ISO 27831 consists of the following parts, under the general title *Metallic and other inorganic coatings — Cleaning and preparation of metal surfaces*:

- *Part 1: Ferrous metals and alloys*
- *Part 2: Non-ferrous metals and alloys*

Introduction

The adhesion of deposited coatings depends upon the efficiency of degreasing and cleaning processes used on the substrate surface. The removal of grease, oil and dirt and all other forms of contamination left from fabrication processes or due to corrosion/erosion of surfaces in storage or in service is essential for successful production of a pristine, chemically clean active surface for deposition of coatings.

The surface contaminants can be

- corrosion products, mould sand or mould release agents on ferrous and non-ferrous castings;
- corrosion products and mill scales on hot-rolled steel sheet, girders, etc. (broken mill scale is cathodic to the underlying substrate);
- oil and rolling lubricant on cold-rolled steel sheet;
- rolling lubricant on e.g. cold-rolled aluminium sheet;
- corrosion products on delivery or during storage on hot-dip-galvanized steel sheet or angle bars/brackets;
- other undesirable materials from storage and handling.

The difficulty of coating these surfaces is well known.

Cleaning processes for removing surface contaminants are varied, depending, among other things, on the identification and classification of the soil as well as the size and shape of the substrate, the degree of cleanliness required, the availability of the facilities needed, the cost of the processes used and their impact on the environment, and the nature of subsequent processes to which the substrate is going to be subjected.

ISO 27831 includes cleaning processes ranging from mechanical methods (which give the least degree of cleanliness) to chemical and ultrasonic methods (which give a higher degree of surface preparation) and substrate conditioning by glow discharge plasma (sputter cleaning) for vapour deposition.

ISO 27831 describes practices for cleaning a variety of metals and metal alloys prior to the application of a range of coatings or without any coating requirements.

The cleaning processes described in ISO 27831-1 and ISO 27831-2 are indicative only of those most commonly used in practice. However, there are numerous formulations of solutions and numerous proprietary processes available which are not included in ISO 27831-1 or ISO 27831-2. Wherever possible, references to particular processes have been made. For references concerning all other processes included in either part of ISO 27831, the publications given in the Bibliography in the respective part should be consulted.

ISO 27831-1 covers ferrous metals and their alloys, whilst ISO 27831-2 covers non-ferrous metals and their alloys. For ISO 27831-2 to be usable, as far as possible, as a “stand-alone” document, Clauses 1 to 6 of ISO 27831-1 have been included in it as Clauses 1 to 6 and the numbering of the other clauses follows the same sequence as in ISO 27831-1. Additionally, since some of the cleaning and preparation processes specified for ferrous materials in ISO 27831-1 can also be used for non-ferrous metals, the cleaning and preparation processes follow the same sequence, using the same designations, as in ISO 27831-1.

Metallic and other inorganic coatings — Cleaning and preparation of metal surfaces —

Part 2: Non-ferrous metals and alloys

WARNING — This part of ISO 27831 may not be compliant with some countries' health, safety and environmental legislation. It calls for the use of substances and/or procedures that may be injurious to health if adequate safety measures are not taken. This part of ISO 27831 does not address any health hazards, safety or environmental matters, or legislation associated with its use. It is the responsibility of the user of this part of ISO 27831 to establish appropriate health, safety and environmentally acceptable practices and take appropriate action to comply with any national, regional and/or international regulations. Compliance with this part of ISO 27831 does not, of itself, confer immunity from legal obligations.

1 Scope

This part of ISO 27831 specifies processes for the cleaning of the surfaces of non-ferrous metals and their alloys to remove any irrelevant or unwanted deposits or other material at any stage of manufacture, storage or service and for the preparation of these surfaces for further treatment. It does not cover cleaning operations associated with the preliminary removal of heavy deposits of oil, grease or dirt accumulated during operational service, preparations for welding or the cleaning of electrical contacts. However, many of the processes included in this part of ISO 27831 may be used for these operations at the discretion of the users of this part of ISO 27831.

This part of ISO 27831 covers processes which are needed for the preparation of metal surfaces prior to the application of the following surface coatings:

- electrodeposited metal coatings;
- autocatalytic metal coatings (autocatalytic and displacement types);
- conversion coatings;
- anodic oxidation coatings;
- hot-dipped coatings;
- sprayed metal coatings;
- coatings produced by vitreous enamelling;
- coatings produced by physical vapour deposition of aluminium and cadmium;
- powder coatings.

This part of ISO 27831 describes processes for carrying out the following treatments:

- degreasing;
- descaling;
- pickling;
- etching;
- de-rusting;
- chemical smoothing;
- chemical polishing;
- electrobrightening;
- electropolishing;
- fluoride anodizing (for cleaning magnesium alloys).

This part of ISO 27831 relates the processes described above to the following metals:

- aluminium and aluminium alloys;
- copper and copper alloys;
- nickel alloys;
- titanium and titanium alloys;
- magnesium alloys;
- zinc-based alloys;
- tin and tin alloys;
- tungsten alloys;
- lead and lead alloys;
- zinc-, cadmium-, chromium- and gold-coated items.

2 Normative references

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

ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

ISO 2080, *Metallic and other inorganic coatings — Surface treatment, metallic and other inorganic coatings — Vocabulary*

ISO 4527, *Metallic coatings — Autocatalytic (electroless) nickel-phosphorus alloy coatings — Specification and test methods*

ISO 8078, *Aerospace process — Anodic treatment of aluminium alloys — Sulfuric acid process, undyed coating*

ISO 9587, *Metallic and other inorganic coatings — Pretreatment of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 9588, *Metallic and other inorganic coatings — Post-coating treatments of iron or steel to reduce the risk of hydrogen embrittlement*

ISO 10074, *Specification for hard anodic oxidation coatings on aluminium and its alloys*

ISO 22778, *Metallic coatings — Physical vapour-deposited coatings of cadmium on iron and steel — Specification and test methods*

ISO 22779, *Metallic coatings — Physical vapour-deposited coatings of aluminium — Specification and test methods*

ISO 27831-1:2008, *Metallic and other inorganic coatings — Cleaning and preparation of metal surfaces — Part 1: Ferrous metals and alloys*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064, ISO 2080, ISO 9587, ISO 9588, ISO 22778 and ISO 22779 apply.

4 Essential information to be supplied by the purchaser to the processor

When ordering articles to be processed in accordance with this part of ISO 27831, the purchaser shall provide the following information in writing, e.g. in the contract, in the purchase order or on the engineering drawing:

- a) the number of this part of ISO 27831 (ISO 27831-2);
- b) the specification and metallurgical condition of the material of which the item is made;
- c) the tensile strength of any steel components and the requirements for stress relief and post-cleaning embrittlement relief heat treatments of the steel to reduce the risk of hydrogen embrittlement (see Annex E);
- d) the cleaning method or methods given in this part of ISO 27831 which are to be used, and any deviation from these methods (e.g. the use of a proprietary method for health or safety reasons);
- e) details of any particular requirements for special surfaces;
- f) details of any special procedures (e.g. process H, method H1);
- g) details of any particularly difficult surface condition and any special information regarding coatings to be removed or coatings or inserts to be protected;
- h) where appropriate, details of the surface coating which is to be subsequently applied.

5 Requirements

5.1 Any materials used for cleaning and preparation shall contain not more than a mass fraction of 5×10^{-6} of mercury (Hg). Where items can be regarded as susceptible to degradation by residual arsenic and/or antimony, the following maximum limitations shall also apply:

for arsenic (As): a mass fraction of 15×10^{-6} ;

for antimony (Sb): a mass fraction of 15×10^{-6} .

NOTE 1 The presence of arsenic, reduced sulfur or reduced phosphorous compounds in an acid bath will promote absorption of hydrogen by steel and may reduce the adhesion of subsequently applied coatings.

NOTE 2 Contamination of steel components can occur when antimony-inhibited solutions are used. The resultant deposit of antimony on exposed surfaces can adversely affect the properties of the steel and subsequent processing of the item. If processing solutions become contaminated, this can lead to further items being similarly affected.

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

5.3 Items will often require a sequence of several degreasing and cleaning treatments. Solvent cleaning may occasionally leave traces of soil after evaporation of the solvent and care shall be taken to remove such contamination. Inorganic contamination may not always be removed by organic solvents. It may therefore be necessary to use additional aqueous processes after final degreasing and cleaning.

5.3.1 The surfaces shall be free from grease, oil, oxide, scale and other foreign matter and shall be in a chemically clean condition, i.e. fully receptive to any subsequent processing.

To test for surface cleanliness, the cleaned surface may be sprayed with, or immersed in, cool, clean water. On clean surfaces, the water will form a uniform film whereas, in the presence of oily soils, the water film will break away from contaminated areas. In cases of gross contamination, the entire film may break down into discrete globules. If the surface to be tested has any residue of cleaning agent containing surface-active agent, a continuous film may be obtained even in the presence of grease. This false effect can be overcome by dipping the surface in dilute acid and rinsing before carrying out the test. Subsequent specified treatments shall be applied without delay.

5.3.2 Abrasives used on any metal or alloy shall not also be used on any dissimilar metal or alloy. Separate abrasive cleaning media shall be used for different alloy families, for example iron and its alloys, aluminium and its alloys. Thus alumina, for example, used for abrasive cleaning of aluminium surfaces, shall not have been used for any other type of material, such as steel or copper-rich alloys, or been otherwise externally contaminated.

Shot peening of the basis metal or substrate, if required, shall be performed before final cleaning and the application of the coating.

5.3.3 The mechanical properties shall not be impaired except where they may be recovered by subsequent heat, or other, treatments forming part of the normal processing sequence.

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

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

It is not good practice to allow an interval between treatments. It shall be borne in mind that degreasing and cleaning procedures produce a surface whose resistance to corrosive attack is low. Consequently, readily corrodible materials need to be protectively coated or further processed immediately.

5.5 Immediately after each treatment in an aqueous solution, the items shall be thoroughly rinsed in clean hot and/or clean cold water, as appropriate, paying special attention to items containing crevices. Immediately after the final rinsing, the metal surface shall not exhibit a water break. Water used for final rinsing after processing shall be discarded or retreated when the conductivity exceeds 10 $\mu\text{S}/\text{cm}$.

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

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

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

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

5.8 All stages of the degreasing and cleaning procedures shall follow each other without delay.

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

5.10 Where limits of composition of solutions are known, they are quoted in this part of ISO 27831. Where limits are not quoted, other proportions may be used provided they are within a tolerance of $\pm 10\%$.

6 Standard cleaning methods

6.1 The processes and methods described in this part of ISO 27831 are indicative only of those most commonly used in practice. However, there are numerous formulations of solutions and proprietary processes available which are not included in this part of ISO 27831. Throughout this part of ISO 27831, the process and method designations in brackets which follow references to cleaning treatments refer to the processes and methods in Clause 22.

6.2 All porous castings shall be degreased, cleaned and dried prior to their impregnation.

6.3 The efficiency of some of the methods may be increased by the use of ultrasonic agitation (process A, method A6), but care shall be taken to avoid damage to delicate items and assemblies such as electrical or electronic equipment and semi-conductor devices.

6.4 To avoid packing or nesting of large volumes of small items in soak cleaners, a barrel cleaning method may be used. This method is not suitable for thin or delicate items.

6.5 Vibratory cleaning methods may be used for the deburring and polishing of fragile components in a moving bed of abrasive medium (process D, method D3) containing a detergent compound.

7 Non-corrosion-resisting steels, cast irons and pure irons

See ISO 27831-1.

8 Corrosion-resisting and heat-resisting steels

See ISO 27831-1.

9 Pretreatment of metals prior to powder coating

See ISO 27831-1.

10 Cleaning and preparation processes

See ISO 27831-1.

11 Aluminium and aluminium alloys

11.1 General

Etching and pickling processes may adversely affect fatigue strength and, for certain high-strength alloys, may cause stress corrosion problems. In particular, sodium hydroxide (method P2) and pickling (process N) can have a severe effect and agreement between supplier and purchaser is necessary for its use. Some authors do not recommend the use of sulfuric acid on aluminium.

The properties of certain heat-treatable aluminium alloys may be adversely affected by prolonged treatment at or above 100 °C. Some aluminium alloys are also susceptible to hydrogen embrittlement.

11.2 General degreasing and cleaning

Degrease and clean by one or more of the following processes and methods, as appropriate:

- a) process A;
- b) process B (method B1) for degreasing unmachined or rough-machined aluminium alloy forgings prior to final inspection;
- c) process B (method B2);
- d) process D;
- e) oxide removal in chromic acid/sulfuric acid solution (process O);
- f) pickling in accordance with process N;
- g) etching in sodium hydroxide solution (method P2);
- h) a chemical or electrolytic polishing treatment (process P).

Drying by chlorinated solvents is not permitted.

11.3 Removal of corrosion products

Use one of the following procedures:

- a) immersion in chromic acid/phosphoric acid solution in accordance with process P (method P1) which results in least attack of underlying metal;

- b) process O, process P (method P3) or process D (excluding method D1) to remove light corrosion products and soil;
- c) process P (method P2) or process D (excluding methods D1 and D4) to remove light corrosion products *in situ*.

NOTE In certain circumstances, fine-abrasive blasting maypeen over pits and retain corrosion products.

11.4 Preparation prior to anodizing

Degrease and clean in accordance with 11.2. Carry out the anodizing itself in accordance with ISO 8078, ISO 10074 or another appropriate International Standard.

11.5 Preparation prior to oxide removal treatment (process O) or filming treatment

Degrease and clean in accordance with 11.2.

11.6 Preparation prior to adhesive bonding of unanodized items

Items shall be prepared for bonding by degreasing and cleaning in accordance with 11.2 and then by etching in accordance with process O or P, as appropriate.

11.7 Preparation prior to electroplating

As preparation for the electrodeposition of any required finish, items shall be degreased, pickled in accordance with 11.2, and given an immersion deposit of zinc followed by an electrodeposit of copper (process W) or another metal.

11.8 Preparation prior to autocatalytic nickel plating

Any process providing a good zincate or modified-zincate coating suitable for electroplating is satisfactory for autocatalytic nickel plating.

A typical (recommended) sequence is to apply a double zincate coating prior to application of the autocatalytic coating, as described below:

- a) process W a) followed by process W b) 2) to W d);
- b) immerse in nitric acid (volume fraction 50 %) for approximately 1 min followed by rinsing;
- c) repeat process W d).

The items are now ready for autocatalytic nickel plating in accordance with ISO 4527 or another appropriate International Standard.

11.9 Preparation prior to conversion coating

Degrease and clean in accordance with 11.2.

11.10 Preparation prior to metal spraying

Degrease and clean in accordance with process A (method A1 or A2) and process B (method B2) followed by coarse-abrasive blasting in accordance with process D (method D1) using a non-metallic abrasive.

11.11 Preparation prior to vitreous enamelling

11.11.1 Sheet, extrusions and items made from casting alloys

A typical (recommended) procedure for sheet, extrusions and items made from casting alloys is as follows:

- a) degrease in accordance with process B (method B2) using a solution buffered at pH 8,9 to 10;
- b) immerse for 5 min to 15 min at 80 °C in the following solution:

| | |
|--|---------------------|
| chromic acid (CrO_3) | 30 g/l to 40 g/l; |
| sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) | 165 g/l to 185 g/l; |

(or use a proprietary non-chromate deoxidizer);

- c) immerse for 30 s to 2 min at 25 °C in the following solution:

| | |
|---|---------------------|
| potassium chromate ($\text{Na}_2\text{CrO}_4 \cdot 10\text{H}_2\text{O}$) | 190 g/l to 210 g/l; |
| sodium hydroxide (NaOH) | 30 g/l to 50 g/l. |

11.11.2 Other castings

Degrease in accordance with process B (method B2) using a solution buffered at pH 8,9 to 10.

11.12 Preparation prior to supplementary treatments on physically vapour deposited aluminium

Degrease in accordance with process A (method A2) and clean in accordance with the requirements of ISO 22779.

12 Copper and copper alloys

12.1 General degreasing and cleaning

Alloys susceptible to stress corrosion cracking shall not be cleaned in acidic or ammoniacal media. Drying using chlorinated solvents is also unacceptable. Degreasing prior to stress relief is permitted in stabilized chlorinated hydrocarbons or in an alkaline aqueous solution. After stress relief, these alloys shall be degreased and cleaned by the following methods, as appropriate:

- a) degreasing in an organic solvent in accordance with process A;
- b) degreasing in an alkaline aqueous solution in accordance with process B;
- c) cleaning in an acid emulsion in accordance with process C;
- d) abrasive cleaning in accordance with process D (method D2, D3 or D4 or, for castings only, method D1);
- e) cleaning in accordance with process Q;
- f) etching or polishing in accordance with process R using a chemical treatment (method R2 or R3) or an electrolytic treatment (method R5).

12.2 Preparation prior to electroplating

12.2.1 Degrease and clean by one or more of following processes and methods, as appropriate:

- a) degreasing in an organic solvent in accordance with process A;
- b) degreasing in an alkaline aqueous solution in accordance with process B;
- c) cleaning in an acid emulsion in accordance with process C;
- d) abrasive cleaning in accordance with process D (method D2, D3 or D4 or, for castings only, method D1);
- e) cleaning in accordance with process Q;
- f) acid descaling, etching or smoothing in accordance with process R (note that copper alloys containing aluminium and/or nickel may not be cleaned sufficiently using process R and may require additional treatment by process Q);
- g) etching or polishing in accordance with process R using a chemical treatment (method R2 or R3) or an electrolytic treatment (method R5).

12.2.2 When plating in a cyanide solution is to be the next treatment, the cleaning procedure shall include acid pickling in accordance with process Q or process R (method R1 or R2) and shall be followed by rinsing in water and immersion in a solution containing about 50 g/l of sodium or potassium cyanide.

12.2.3 Soft-soldered items (other than beryllium/copper alloy items) shall be degreased in accordance with process A (method A1 or A2) and then cleaned by immersion for the minimum time necessary in a mild alkaline cleaning solution in accordance with process B (method B2). This shall be followed by immersion in a solution containing approximately 100 ml of 40 % mass fraction fluoroboric acid (HBF_4) per litre, rinsing thoroughly and copper plating to a minimum thickness of 1 μm using a cyanide copper electrolyte (of the Rochelle salt/cyanide type) having a pH of 10 to 12 and operated at a temperature $60\text{ }^\circ\text{C} \pm 10\text{ }^\circ\text{C}$.

12.2.4 Beryllium/copper items which are to be plated in an acid electrolyte shall be cleaned in accordance with process Q (method Q1) followed by rinsing in water and immersing in a solution containing 100 ml/l of sulfuric acid ($\rho = 1,84\text{ g/cm}^3$).

12.3 Preparation prior to autocatalytic nickel plating

Degrease and clean in accordance with 12.2 and then treat as follows:

- a) clean cathodically in a mild alkaline cleaner in accordance with process B;
- b) rinse (see 5.5);
- c) catalyse the surface by one of the following four methods:
 - by striking in an electrolytic nickel bath in accordance with process M (method M4) (see Annex F),
 - by immersion in an activating dip, containing e.g. palladium chloride, until the surface has darkened, indicating deposition of palladium, followed by rinsing and transfer to the autocatalytic nickel bath (when proprietary solutions are used, the directions of the suppliers shall be followed),
 - by contacting the surface of the item with aluminium wire whilst immersed in the autocatalytic nickel solution (or contacting with an already plated item) until the autocatalytic nickel has spread over the entire surface,
 - by applying an initial cathodic potential to the item immersed in the autocatalytic nickel bath.

Carry out the autocatalytic nickel plating itself in accordance with ISO 4527 or another appropriate International Standard.

12.4 Preparation prior to hot tinning (TRI process) [2]

Degrease in accordance with process A or B. Oxygen-containing copper shall 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).

Following degreasing, pickle in accordance with process R (method R1, R2 or R4).

12.5 Preparation prior to vitreous tinning

Degrease as follows:

- for small items, e.g. jewellery, heat in a furnace or with a blow torch until the items are at dull-red heat and plunge into cold, running water or allow to cool in air (note that relatively thin sections may be distorted due to the quenching effect);
- for large or heavy items, degrease either in accordance with process B or, if made necessary by the type of lubricant used in pressing, in accordance with process A (method A1) using trichloroethylene as solvent, in each case followed by rinsing in cold, flowing, agitated water.

Following degreasing, clean by pickling, as follows:

- immerse for 2 min to 10 min at room temperature in an aqueous solution containing a 10 % to 15 % volume fraction of nitric acid ($\rho = 1,42 \text{ g/cm}^3$);
- immerse for 5 min to 15 min in an aqueous solution containing a 10 % volume fraction of hydrochloric acid ($\rho = 1,16 \text{ g/cm}^3$);
- neutralize in a mildly alkaline solution;
- dry with hot, clean air.

13 Nickel alloys

13.1 General degreasing and cleaning

Degrease and clean by one or more of following processes and methods, as appropriate:

- a) degreasing in an organic solvent in accordance with process A (method A1 or A2);
- b) degreasing in an aqueous alkaline solution in accordance with process B;
- c) cleaning in an acid emulsion in accordance with process C;
- d) abrasive cleaning in accordance with process D;
- e) sodium hydride descaling in accordance with process E;
- f) pickling in an acid solution in accordance with process T;
- g) chemical or electrolytic etching in accordance with process X.

13.2 Preparation prior to electroplating

Degrease and clean by the processes and methods given in 13.1, as appropriate, including process X.

13.3 Preparation prior to autocatalytic deposition

Degrease in accordance with 13.1, followed by a nickel strike [process X b)].

13.4 Preparation prior to vitreous enamelling

13.4.1 Degreasing and cleaning is normally carried out in accordance with the following processes and methods:

- a) process A;
- b) process B;
- c) process D (method D2).

13.4.2 The following procedure may also apply:

- a) degrease and clean by one or more of the processes and methods in 13.4.1, as appropriate;
- b) etch by immersion for 5 min to 20 min in an aqueous solution containing a 10 % volume fraction of nitric acid ($\rho = 1,42 \text{ g/cm}^3$) and a 2 % volume fraction of hydrofluoric acid (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$) at a temperature between room temperature and 40 °C;
- c) immerse for 3 min to 5 min in the following neutralizing solution at a temperature of 70 °C to 80 °C:

| | |
|--|----------|
| anhydrous sodium carbonate (Na_2CO_3) | 4,5 g/l; |
| borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) | 1,5 g/l. |

14 Titanium and titanium alloys

14.1 General degreasing and cleaning

14.1.1 Degrease and clean by one or more of the processes and methods given in 14.1.2 to 14.1.7, as appropriate.

14.1.2 Degrease in an organic solvent in accordance with process A. Certain chlorinated-hydrocarbon solvents (including those in paint-stripping agents) may cause hydrogen embrittlement or stress corrosion cracking of some titanium alloys and the following precautions shall be observed when such solvents are used:

- a) stressed items made of titanium or titanium alloys (such as Ti-5Al-2,5Sn) shall not be exposed to hot chlorinated solvents;
- b) the solvents shall be kept clean and the accumulation of free acid shall be prevented in accordance with the supplier's instructions for use;
- c) where hot solvents (such as trichloroethylene or hot perchloroethylene) are used as liquid and/or vapour, the time of immersion shall not exceed 30 min for any one degreasing operation;
- d) care shall be taken to remove all traces of solvent from the item immediately after cleaning and before any further operation is performed.

14.1.3 Degrease in an alkaline aqueous solution in accordance with process B. Cathodic cleaning shall not be used.

14.1.4 Clean in an acid emulsion in accordance with process C.

14.1.5 Carry out abrasive cleaning, by wet methods only, in accordance with process D.

14.1.6 Descale by a suitable method.

14.1.7 Pickle in nitric acid/hydrofluoric acid solution in accordance with process S.

14.2 Preparation prior to electroplating

Titanium and titanium alloys are difficult to electroplate. A preparatory treatment is suggested in process Y.

14.3 Preparation prior to autocatalytic nickel plating

Prepare as for electroplating (see 14.2). After the nickel strike (method Y2 c), the items shall be thoroughly rinsed (see 5.5) before being transferred to the autocatalytic nickel-plating solution for plating in accordance with ISO 4527 or another appropriate International Standard.

15 Magnesium alloys

15.1 General degreasing and cleaning prior to electroplating and conversion coating

Degrease in accordance with process A or B, using the appropriate methods.

15.2 Preparation prior to electroplating

Use the following procedure:

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

| | |
|---|---------|
| sodium hydroxide (NaOH) | 30 g/l, |
| sodium cyanide (NaCN) | 30 g/l, |
| sodium carbonate (Na ₂ CO ₃) | 15 g/l; |

b) rinse (see 5.5);

c) pickle in phosphoric acid ($\rho = 1,75 \text{ g/cm}^3$) at room temperature for 30 s;

d) rinse (see 5.5);

e) activate by dipping, at room temperature, in the following solution:

| | |
|--|--|
| phosphoric acid (H ₃ PO ₄) ($\rho = 1,75 \text{ g/cm}^3$) | 130 ml/l to 150 ml/l, preferably 135 ml/l, |
| potassium fluoride (KF) | 60 g/l to 80 g/l, preferably 70 g/l; |

f) rinse (see 5.5);

g) immerse at 90 °C to 95 °C for 5 min in the following solution (the preferred values will give a pH of 10,6):

| | |
|---|---|
| zinc sulfate (ZnSO ₄ ·7H ₂ O) | 45 g/l to 55 g/l, preferably 50 g/l, |
| sodium pyrophosphate (Na ₄ P ₂ O ₇) | 150 g/l to 200 g/l, preferably 180 g/l, |
| potassium fluoride (KF) | 4 g/l to 8 g/l, preferably 6 g/l, |
| sodium carbonate (Na ₂ CO ₃) | 3 g/l to 10 g/l, preferably 4 g/l; |

- h) rinse (see 5.5);
- i) plate in a cyanide copper solution to produce a deposit thickness of at least 5 µm, using “live” entry, i.e. making the electrical connections before the items are immersed in the copper bath, followed by thorough rinsing;
- j) deposit with the required type of coating.

15.3 Preparation prior to conversion coating

15.3.1 General

To ensure high resistance to corrosion, it is important that the alloy does not contain any entrapped flux and has a low impurity content, and that the surfaces of the parts are not contaminated by flux, sand or other residues from the casting process or other impurities from fabrication processes, such as die casting, rolling or forging, or left after abrasive blasting. Smearing of other metals, e.g. machining residues in cutting fluids, into the surfaces shall also be avoided.

15.3.2 As-cast castings

15.3.2.1 Castings, other than certain die castings, shall first be blast cleaned in accordance with process D (method D1), followed by pickling in accordance with process F.

15.3.2.2 Subsequently, with the exception of castings which have been machined all over, carry out a fluoride anodizing treatment in an aqueous solution containing 150 g/l to 250 g/l of ammonium bifluoride (NH_4HF_2) in a vessel lined with hard rubber or suitable plastics material resistant to acid fluoride solution.

Maintain the concentration of the ammonium bifluoride in the bath at a mass fraction between 15 % and 25 % by the addition of fresh quantities of NH_4HF_2 . Discard the bath if it becomes contaminated with foreign metals, acid radicals other than fluoride, or organic matter.

The temperature of the bath shall not exceed 30 °C. Stir the bath with a pole made of wood, ebonite or magnesium alloy.

Treat items in pairs of approximately equal surface area using alternating current at a voltage of 90 V to 120 V. Suspend the items not less than 225 mm below the surface of the solution. All parts of the mounting clamps which extend into the solution shall be made of a magnesium alloy other than magnesium-manganese alloy. The current is usually high at first, but diminishes rapidly as surface impurities are removed and an unbroken magnesium fluoride film is formed.

Continue the treatment for 10 min to 15 min or until the current falls below 0,5 A/dm² at the smaller electrode. Then switch the current off, remove the items from the bath, and wash and examine them.

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

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

WARNING — Spray from the bath is poisonous and the application of high voltage requires appropriate safety precautions.

15.3.2.3 Unless the subsequent chromate treatment is to be carried out in an acid bath capable of removing the fluoride film left by the cleaning process, this film shall be removed by immersion 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 sodium hydroxide or for 5 min in a cold aqueous solution containing 150 g/l to 200 g/l of hydrofluoric acid ($\rho = 1,128 \text{ g/cm}^3$).

The items are now ready for conversion coating and the application of any organic finishes.

15.3.3 Machined castings

Degrease in accordance with process A (method A1) and process B.

The items are now ready for conversion coating and the application of any organic finishes.

15.3.4 Forgings

With the exception of items which are to be machined all over, all forgings shall be treated in accordance with 15.3.2.

15.3.5 Sheets and extrusions

Degrease in accordance with process A (method A1) and process B.

Then treat the items in accordance with 15.3.2.2 or by scouring with pumice powder applied with a moist rag in accordance with process D (method D3).

The items are now ready for conversion coating and the application of any organic finishes.

16 Zinc-based alloys

16.1 General degreasing and cleaning

Unless otherwise required, e.g. for chromate conversion coating of zinc surfaces, degrease in accordance with process A (method A1) and process B (method B2).

Zinc-based alloys are normally in the form of pressure die castings with chromate conversion coating as the final finish. Die-cast items frequently contain sub-surface porosity, and any process such as acid etching which penetrates the non-porous skin may expose this porosity, allowing the consequent entrapment of corrosive residues and giving rise to corrosion problems in storage and/or in service. Such processes shall be avoided.

16.2 Cleaning of corroded items

Immerse in a solution containing about 100 g/l of chromic acid (CrO_3) at 90 °C.

Corrosion of pressure die cast zinc alloys during storage or service may expose sub-surface porosity. As stated in 16.1, any cleaning process leaving corrosive residues shall be avoided.

16.3 Preparation of zinc alloy die castings prior to electroplating

Preparatory treatments are given in process Z.

16.4 Preparation of zinc alloy castings prior to chromate conversion coating or phosphate treatment

Degrease in accordance with process A and process B (method B2).

17 Tin and its alloys

Prior to electroplating, prepare in accordance with process U.

18 Tungsten alloys

18.1 Preparation prior to electroplating

Degrease in accordance with process A (method A1) and process B (method B1) and clean by anodic treatment at a current density of 5 A/dm² for 2 min at ambient temperature in the following solution:

sodium hydroxide (NaOH) 200 g/l;

sodium cyanide (NaCN) 100 g/l.

Then rinse in cold, running water (see 5.5).

Effective anodic treatment can also be carried out using lower current densities but proportionally longer times, e.g. 2 A/dm² for 5 min.

18.2 Preparation prior to adhesive bonding

Degrease in accordance with process A (method A1) and process B (method B2). Then clean in accordance with process D. Specialist advice may be required for some alloys.

19 Lead and its alloys

NOTE Lead is active chemically and forms oxide films in air, films of insoluble salts with most acids during acid pickling (sulfuric, hydrochloric and hydrofluoric acids cannot be used, but sulfamic and fluoroboric acids can), and diffuses into other non-ferrous metals when it is contact with them.

First degrease in accordance with process A.

Then clean by immersion in a hot solution containing a 5 % mass fraction of ammonium acetate to remove PbO and PbSO₄.

20 Zinc-, cadmium-, chromium- and gold-coated items

20.1 Preparation of zinc-coated items prior to chromate conversion coating

Freshly zinc-coated steel items, including those which have been given hydrogen embrittlement relief heat treatment in accordance with ISO 9588, which have not been contaminated with oil, grease (e.g. from handling) or dirt or been exposed to any outdoor environment may not need any further degreasing.

Zinc-coated items which are not freshly coated, including those which have been given hydrogen embrittlement relief heat treatment in accordance with ISO 9588, shall be degreased in accordance with process A and process B (method B2) (see also 16.1 and Annex E).

20.2 Preparation of cadmium-coated items prior to chromate conversion coating

Degrease in accordance with 20.1 (see also 16.1).

20.3 Preparation of physically vapour deposited cadmium prior to supplementary treatments

Degrease in accordance with process A (method A2) (see also ISO 22778).

20.4 Preparation for chromium-coated parts prior to supplementary treatments

Degrease in accordance with process A (method A2) and then treat by immersion in a solution containing a 1 % mass fraction of chromic acid (CrO_3) at room temperature for a few minutes.

20.5 Removal of surface contamination from gold coatings [4]

Process V is suggested as a suitable procedure.

21 Pretreatment of metals prior to powder coating

21.1 Preparation of aluminium prior to powder coating

Degrease in accordance with 11.2.

21.2 Preparation of zinc and hot-dip-galvanized steel prior to powder coating

For zinc: degrease in accordance with 16.1.

For hot-dip-galvanized steel: degrease in accordance with process A (method A1 or A2), followed by process D (method D2).

NOTE Sulfuric acid pickling (process F) is unsuitable for galvanized steel.

21.3 Preparation of copper and brass prior to powder coating

Degrease and clean in accordance with 12.1.

22 Cleaning and preparation processes (sequence follows that in ISO 27831-1:2008, Clause 10)

WARNING — Many of the chemicals and processes referred in this part of ISO 27831 are hazardous to health and safety. Appropriate precautions, especially in dealing with hydrofluoric and chromic acids, shall be taken. This part of ISO 27831 only refers to technical suitability and in no way absolves either the designer, the producer, the supplier and/or the user from statutory and all other legal obligations relating to health, safety and environmental considerations at any stage of manufacture or use. It is the responsibility of the user of this part of ISO 27831 to establish appropriate health, safety and environmentally acceptable practices and take suitable actions to comply with any national and international regulations.

When mixing concentrated sulfuric acid with water or an aqueous solution, always add the sulfuric acid slowly to the water or aqueous solution, never *vice versa*, and stir thoroughly during addition. Allow the mixture to cool to room temperature before use.

NOTE 1 The following processes and methods for the pretreatment of metals are only indicative of those most commonly used in practice. However, there are numerous formulations of solutions which are not included in this part of ISO 27831. In addition, there are various proprietary products available for processes in accordance with the suppliers' instructions.

NOTE 2 The terms "cold", "room temperature", "warm" and "hot" are considered to represent mean temperatures of the order of "less than 25 °C", "around 25 °C", "from 25 °C to 45 °C" and "greater than 45 °C", respectively. All acid solutions are technical grade unless specified otherwise.

NOTE 3 Since some of the cleaning and preparation processes specified for ferrous materials in ISO 27831-1 can also be used for non-ferrous materials, the cleaning and preparation processes in this part of ISO 27831 follow the same sequence, using the same designations, as in Part 1.

22.1 Process A — Organic-solvent degreasing and cleaning

22.1.1 General

Solvents which have an adverse effect on the metal are detrimental. The heat capacity of the component is of great significance. For instance, heavily contaminated thin sheet may degrease unsatisfactorily with a particular vapour treatment. In such cases, spraying with clean condensed solvent after vapour treatment, or immersion in boiling liquor, is preferred. For most applications, liquid and vapour degreasing as well as alkaline degreasing may be required for comprehensive degreasing of the metal surfaces. Boiling-liquid and vapour cleaning plants shall be operated and maintained in accordance with supplier's instructions and appropriate national and international regulations.

22.1.2 Method A1 — Hot solvent, not water-rinsable

This method covers liquid and vapour degreasing in hot halogenated hydrocarbon solvents. The grade of solvent shall be known to be stable for this use. Subject to the restrictions for titanium (see 14.1), as well as national and international regulations, the use of the following halogenated solvents is permitted:

trichloroethylene;

perchloroethylene.

For this method, items made of aluminium, magnesium or titanium shall be free from swarf. Some guidance on the maintenance of organic solvent and vapour degreasing baths is given in Annex A. Trichloroethylene will require a water separator. Perchloroethylene has a higher boiling point than trichloroethylene and may be more suitable for the removal of high melting point waxes. Trichloroethylene and perchloroethylene shall not be used on assemblies containing fabric, rubber, paint or similar materials.

22.1.3 Method A2 — Cold solvent, not water-rinsable

These cleaning agents are normally used by immersion or by hand application (spraying or brushing). The choice of solvent is largely governed by considerations of toxicity, volatility and flammability. Suitable cleaning agents include the following:

trichloroethylene;

perchloroethylene;

white spirit;

white spirit/solvent naphthas.

Items made of aluminium, magnesium or titanium shall be free from swarf. When the items are degreased by immersion in the solvent, at least three successive baths of solvent shall be used. When hand application is used, the solvent shall be applied by brushing or spraying and the solvent allowed to flood the work surface. When the solvent becomes contaminated with grease, it shall be replaced. Some guidance on maintenance of organic-solvent baths is given in Annex A.

22.1.4 Method A3 — Hot solvent, water-rinsable

This method is used both for degreasing and to remove paint and carbonaceous deposits by means of a solvent based on an emulsifiable blend of cresylic acid and o-dichlorobenzene. It is used with a water seal in a bath operated at up to 60 °C or in accordance with the manufacturer's instructions.

22.1.5 Method A4 — Cold solvent, water-rinsable

Dichloromethane-based mixtures (dichloromethane mass fraction approximately 70 %) are mainly used for the removal of paint and carbonaceous deposits, but may also act as a degreasant. Dichloromethane-based mixtures intended for application by brushing are unsuitable for use as degreasants.

Thickening and emulsifying agents may be added to assist contact and to facilitate rinsing. Corrosion inhibitors may also be added for application to close-tolerance items or where the slight etching of the metal surface which may take place is undesirable.

22.1.6 Method A5 — Petroleum fuel based mixtures containing emulsifying agents

These mixtures may be used for the substantial removal of heavy deposits of oil or grease. Application is normally by brushing or spraying, with subsequent water rinsing. Hydrocarbons, e.g. white spirit or kerosene, with an emulsifying agent are suitable. For complete removal of all residual contamination, this procedure shall be followed by process B.

22.1.7 Method A6 — Ultrasonic cleaning

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

22.2 Process B — Alkaline degreasing

22.2.1 General

This method may be used for complete degreasing or in addition to preliminary solvent degreasing. It has advantages over solvent degreasing in the removal of certain types of contaminants, including soaps and salts. The cleaning action is based on the saponifying and emulsifying effects of aqueous alkalis, often reinforced by sequestering, complexing and surface-active agents. The ingredients are usually selected from sodium hydroxide, sodium carbonate, sodium metasilicates, trisodium phosphate, sodium pyrophosphate, sodium borates, complexing agents (such as EDTA, gluconates, heptonates, polyphosphates and cyanides) and organic surfactants. They may be used hot or cold and with or without applied current which may be either anodic or cathodic (but see Annex E). Very thorough washing is required to remove residual traces of alkaline cleaners from the metal surfaces. The surfaces of metal items which have been alkali-cleaned and which are to be processed for the next stage of coating without pretreatment shall first be neutralized by means of an acid dip containing a 2,5 % mass fraction of chromic acid (CrO_3) plus a 2,5 % volume fraction of phosphoric acid (H_3PO_4) ($\rho = 1,75 \text{ g/cm}^3$), then rinsed (see 5.5) and dried before the application of the coating.

22.2.2 Method B1 — General-purpose alkaline degreasing

The cleaning agent may contain any of the constituents given in 22.2.1. Heavy-duty types may contain a high proportion of sodium hydroxide. They are suitable for use on all metals except aluminium, lead, tin and zinc, each of which is attacked by strong alkalis. For these metals, pickling in sodium hydroxide or other alkaline solutions shall only be employed with discretion, with the agreement of the purchaser, and shall be strictly controlled.

22.2.3 Method B2 — Mild alkaline degreasing

The cleaning agent shall be free from sodium hydroxide and any other strong alkali and shall be inhibited to avoid attack on the metal surfaces. It can be used by soaking or spraying.

Sodium metasilicate types shall include a mass fraction of at least 25 % of sodium metasilicate.

Silicate-free types are based on alkaline phosphates and organic surfactants with the addition of buffers, if required. They are used hot or cold and can be used with or without current (but see Annex E).

22.2.4 Method B3 — Cathodic alkaline degreasing

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

22.3 Process C — Acid emulsion cleaning

For the removal of ingrained organic contamination, e.g. on cold-rolled material, as well as in instances where organic contamination is slight, mineral acids containing a 2 % to 5 % volume fraction of non-ionic surface-active agents are used cold or warm by spraying or dipping, provided the use of the mineral acid is not contrary to other clauses and subclauses in this part of ISO 27831.

22.4 Process D — Abrasive cleaning

22.4.1 General

Residual abrasive material left on items treated by abrasive cleaning may interfere seriously with subsequent treatment and shall therefore be removed from the metal surface by a jet of clean, dry air which shall be free from oil and dust. Abraded items shall be handled with clean rubber, cotton or smooth plastic gloves, and any contamination with dust, dirt, oil, grease or water shall be avoided.

When using alumina for Method D1, it shall not have been used on any other type of material, e.g. steel or copper-rich alloys, or have been otherwise externally contaminated.

Care shall be taken to avoid using abrasives which are insufficiently coarse or which have lost angularity through use in recirculating blast cabinets. Both the coarseness and the angularity of the abrasive used have a great effect on the adhesion of the subsequently applied coating. Direct pressure blasting methods, which are not suitable for thin sections, is preferable to suction blasting because of the slowness and poor roughening characteristics of the latter.

22.4.2 Method D1 — Coarse-abrasive blasting

This is carried out by blasting with chilled-iron grit, alumina or another coarse abrasive. The method is unsuitable for thin items or where a fine surface profile is required. With corrosion-resisting steels and with aluminium and aluminium alloys, only lead-free alumina grit shall be used. When being cleaned prior to metal spraying, the surface shall be roughened, to provide an adequate key, by blasting with alumina. The abraded surface shall present an even, matt appearance. It shall be borne in mind that the surface will be in a very active state and any deterioration will very readily affect the performance of the coating.

This method is also useful for cleaning corroded items.

22.4.3 Method D2 — Fine-abrasive blasting

This method is similar to method D1 but employs either non-metallic abrasives (e.g. alumina, glass beads, corn husks) or metallic abrasives with or without water in such a way as to produce a finer surface profile than that produced by method D1.

22.4.4 Method D3 — Scouring

This method is carried out by rubbing or scrubbing with moistened pumice, alumina or another non-metallic abrasive. Silicon carbide abrasive is not suitable for use on aluminium and aluminium alloys.

22.4.5 Method D4 — Tumbling

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

22.4.6 Method D5 — Wire brushing

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

22.5 Process E — Caustic-alkali descaling

Not normally used for the metals and metal alloys covered by this part of ISO 27831 (see method E1 in ISO 27831-1:2008, if needed).

22.6 Process F — Acid pickling without electrolysis

Not normally used for the metals and metal alloys covered by this part of ISO 27831.

22.7 Process G — Acid dipping

Not normally used for the metals and metal alloys covered by this part of ISO 27831.

22.8 Process H — Anodic pickling

22.8.1 General

Of the anodic-pickling methods specified in ISO 27831-1, only method H1 is normally used for the metals and metal alloys covered by this part of ISO 27831.

22.8.2 Method H1

Immerse the items in an aqueous solution (ρ at least 1,30 g/cm³) containing 300 ml/l to 350 ml/l of sulfuric acid ($\rho = 1,84$ g/cm³). The solution strength shall be maintained by periodic additions of sulfuric acid to keep the relative density greater than 1,30 g/cm³. Soaking the items in an alkaline solution of sodium gluconate at up to the boiling point prior to immersing them in the acid solution assists in the removal of scale.

Set the voltage so that the initial current density is not less than 11 A/dm² (4 V to 8 V) and is preferably twice this. The temperature shall not normally exceed 25 °C although, for removal of scale, a temperature of up to 70 °C may be used. An inhibitor which is stable in the solution is allowed in order to reduce the formation of smut, but wetting agents shall be avoided as they promote the absorption of hydrogen.

In anodic pickling, contamination of the pickling solution with chloride shall be avoided. The essential aim of anodic pickling is to render the metal passive, a condition that will be indicated by a sharp rise in voltage between the item and the cathode, a corresponding fall in current and the onset of gas evolution from the item. Passivity shall be maintained until the surface has acquired a light grey colour, free from dark smut. Items can be withdrawn from the solution for examination and, if necessary, put back in the bath for further treatment.

22.9 Process J — Acid de-rusting

Not normally used for the metals and metal alloys covered by this part of ISO 27831.

22.10 Process K — Alkaline de-rusting

Not normally used for the metals and metal alloys covered by this part of ISO 27831.

22.11 Process L — Chemical smoothing and electropolishing of steels

Not normally used for the metals and metal alloys covered by this part of ISO 27831.

22.12 Process M — Passivation and removal of surface contamination from corrosion-resisting steels and surface preparation prior to electroplating

Not normally used for the metals and metal alloys covered by this part of ISO 27831.

22.13 Process N — Acid pickling of aluminium alloys**22.13.1 General**

Solutions of the approximate compositions given in methods N1 to N6 are suitable for pickling degreased aluminium alloys and are usually accepted for use prior to the final inspection of forgings.

An immersion time of up to 20 min may be used for sheets and up to 1 h for castings. If the solution is contained in a metal tank, the parts shall not be in electrical contact with the tank.

22.13.2 Method N1

Sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) 90 ml/l to 120 ml/l;

sodium fluoride (NaF) 7,5 g/l to 15 g/l.

Immerse the items at room temperature until uniformly clean. Rinse in cold water (see 5.5) and transfer to a cold aqueous de-smutting solution containing approximately 500 ml/l of nitric acid ($\rho = 1,42 \text{ g/cm}^3$) for about 1 min and then thoroughly wash in clean water at a temperature not exceeding 50 °C.

NOTE Aluminium build-up in the bath can influence the effectiveness of the bath.

22.13.3 Method N2

Sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) 100 ml/l;

potassium fluoride (KF) 40 g/l.

Immerse the items at room temperature until uniformly clean. Rinse in cold water (see 5.5) and transfer to a cold aqueous de-smutting solution containing approximately 500 ml/l of nitric acid ($\rho = 1,42 \text{ g/cm}^3$) for about 1 min and then thoroughly wash in clean water at a temperature not exceeding 50 °C.

NOTE Aluminium build-up in the bath can influence the effectiveness of the bath.

22.13.4 Method N3

Sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) 100 ml/l;

hydrofluoric acid (HF) (40 % mass fraction solution; $\rho = 1,128 \text{ g/cm}^3$) 15 ml/l.

Immerse the items at room temperature until uniformly clean. Rinse in cold water (see 5.5) and transfer to a cold aqueous de-smutting solution containing approximately 500 ml/l of nitric acid ($\rho = 1,42 \text{ g/cm}^3$) for about 1 min and then thoroughly wash in clean water at a temperature not exceeding 50 °C.

NOTE Aluminium build-up in the bath can influence the effectiveness of the bath.

22.13.5 Method N4

Phosphoric acid (H_3PO_4) ($\rho = 1,75 \text{ g/cm}^3$) 200 ml/l;

hydrofluoric acid (HF) (40 % mass fraction solution; $\rho = 1,128 \text{ g/cm}^3$) 7,5 ml/l.

Immerse the items at room temperature until uniformly clean. Rinse in cold water (see 5.5) and transfer to a cold aqueous de-smutting solution containing approximately 500 ml/l of nitric acid ($\rho = 1,42 \text{ g/cm}^3$) for about 1 min and then thoroughly wash in clean water at a temperature not exceeding 50 °C.

NOTE Aluminium build-up in the bath can influence the effectiveness of the bath.

22.13.6 Method N5

Sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) 100 ml;

o-toluidine ($\text{H}_3\text{C}-\text{C}_6\text{H}_4-\text{NH}_2$) 10 ml.

Immerse the items at 90 °C to 95 °C until uniformly clean.

22.13.7 Method N6

A gently boiling aqueous solution containing:

chromic acid (CrO_3) 7,5 g/l to 10 g/l;

phosphoric acid (H_3PO_4) ($\rho = 1,75 \text{ g/cm}^3$) 5 ml/l to 7,5 ml/l.

22.14 Process O — Oxide removal, i.e. “etching”, treatment of aluminium and aluminium alloys

22.14.1 General

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

The procedure is as follows:

22.14.2 Degreasing and cleaning

The items shall be treated as follows:

- Degrease by process A (method A1 or A2). Heavily soiled or small items may need more than one treatment.
- Alkaline degrease in accordance with process B (method B2). For items which are to be subsequently bonded, use a silicate-free type of cleaning agent (see 22.2.3).
- Rinse (see 5.5) and, where wet or dry abrasive blasting is not specified, transfer to the etching solution without drying.
- Where dry abrasive blasting is specified or where wet abrasive blasting is specified but does not immediately follow the rinsing operation, the items shall be dried. Care shall be taken to protect the cleaned items from contamination.

- e) Items which have been dry abrasive blasted shall be treated with clean, dry compressed air followed by alkaline degreasing in accordance with process B (method B2) and rinsed (see 5.5) to remove all traces of blasting grit. They shall then be transferred to the chromic acid/sulfuric acid bath without drying.
- f) Items which have been wet abrasive blasted shall be rinsed (see 5.5) to remove all traces of blasting grit and transferred to the chromic acid/sulfuric acid bath without drying.

All abrasive residues shall be removed since their presence may impair the properties of the etched surface.

22.14.3 Preparation prior to anodizing and adhesive bonding

22.14.3.1 Immediately after cleaning, immerse the items in one of the aqueous solutions specified in 22.14.3.2 to 22.14.3.5. In all cases, the temperature range shall be 60 °C to 65 °C. The immersion time shall be 25 min to 30 min for adhesive-bonding and organic-primer applications and 20 min to 30 min for pretreatments prior to anodizing and other chemical treatments. Shorter immersion times (less than 20 min) shall be used for items which are machined all over and which are not to be adhesive bonded.

22.14.3.2 For adhesive-bonding applications:

| | |
|--|---------------------|
| chromic acid (CrO_3) | 45 g/l to 55 g/l; |
| sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) | 220 g/l to 300 g/l. |

22.14.3.3 As a pre-treatment for anodizing and other chemical treatments:

| | |
|--|---------------------|
| chromic acid (CrO_3) | 30 g/l to 55 g/l; |
| sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) | 150 g/l to 300 g/l. |

22.14.3.4 For adhesive-bonding applications:

| | |
|--|---------------------|
| sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$) | 65 g/l to 85 g/l; |
| sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) | 220 g/l to 300 g/l. |

22.14.3.5 As a pre-treatment prior to anodizing and other chemical treatments:

| | |
|--|---------------------|
| sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$) | 45 g/l to 85 g/l; |
| sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) | 150 g/l to 300 g/l. |

22.14.3.6 The following limits shall not be exceeded for any of the solutions specified in 22.14.3.2 to 22.14.3.5:

| | |
|--------------------|-----------|
| chloride (as NaCl) | 200 mg/l; |
| aluminium | 14 g/l; |
| copper | 1,0 g/l; |
| iron | 1 g/l. |

The $\text{H}_2\text{SO}_4:\text{CrO}_3$ ratio shall be between 4,5:1 and 5,5:1 (calculated in terms of mass fractions). The $\text{H}_2\text{SO}_4:\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ ratio shall be between 2,5:1 and 3,5:1 (calculated in terms of mass fractions).

The solutions shall be prepared and maintained at the correct operating level with water having a conductivity of not more than 10 $\mu\text{S/cm}$ and shall be checked at regular intervals to determine conformance with the solution control limits.

A solution shall be discarded when the specific gravity attains a value of $\rho = 1,38 \text{ g/cm}^3$, measured at room temperature, or when solid materials separate out on cooling the solution to room temperature or at the first signs of pitting, whichever occurs first.

A fresh solution shall be aged either with Al-Cu alloy scrap or with aluminium sulfate or copper sulfate.

If the etching solution is contained in a metal tank, the items shall not be in electrical contact with the tank.

Rinse the items in running water with a temperature not exceeding 40 °C to prevent surface sealing effects (see also 5.5 for rinsing). The transfer of items from the pickling bath to the rinsing bath shall be as rapid as possible to prevent dry spots forming on the surface.

If no subsequent chemical treatments are to be carried out, dry the items at a temperature not exceeding 65 °C, particularly before bonding. With items which are to be bonded using adhesive or coated with a primer, the adhesive or primer shall be applied immediately after drying. The etched surfaces shall not be touched with the bare hands or be otherwise contaminated. The etched surfaces shall be of uniform appearance and free from any stains, deposits or pits.

22.15 Process P — Aluminium and aluminium alloys — Removal of corrosion products, etching treatment, chemical polishing, electrobrightening and electropolishing

22.15.1 Removal of corrosion products

22.15.1.1 Method P1 — Immersion

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

chromic acid (CrO_3) 7,5 g/l to 10 g/l;

phosphoric acid (H_3PO_4) ($\rho = 1,75 \text{ g/cm}^3$) 5 g/l to 7,5 ml/l.

An immersion time of 20 min is normally adequate for sheets and up to 1 h for castings.

22.15.1.2 Method P2 — Swabbing

Swab the items with a solution of 50 g/l of chromic acid in water and rinse thoroughly in clean water (see 5.5).

22.15.2 Etching treatment

22.15.2.1 Method P3 — Etching by brushing or spray treatment

Where items are unsuitable for immersion treatment, they can be treated by brushing or spraying with a solution of phosphoric acid in which kaolin and a suitable pigment are suspended. The free acidity of the solution, measured as described in Annex D, shall be equivalent to that of a solution containing a 6,0 % to 6,5 % mass fraction of phosphoric acid. In the case of an aluminium panel, the panel is held vertically and a film of material is applied by brush (or spray) at a rate of 140 g/m² to the surfaces of the panel. The panel is maintained at a temperature of 15 °C to 20 °C in a draught-free environment for about 20 min for the film to dry. The dry film can be easily removed by a jet of water or by the use of a stiff brush, leaving the aluminium surface lightly etched.

22.15.2.2 Method P4 — Etching and de-smutting treatment using sodium hydroxide

This etching and de-smutting method is used prior to anodizing if a polished finish is not required.

Immerse the degreased metal in the following solution:

sodium hydroxide (NaOH) 25 g/l to 50 g/l;

sodium heptonate or sodium gluconate 0,75 g/l to 1,0 g/l.

For a uniform etch of medium depth, the concentration of free sodium hydroxide in the solution shall not be allowed to fall below 25 g/l and that of aluminium shall not be allowed to exceed 30 g/l. When the sodium hydroxide and aluminium concentrations approach these values, the solution shall be replaced.

Operate the solution at 60 °C to 65 °C. The reaction is very vigorous and immersion times from 15 s to 1 min are usually adequate.

When the finish produced becomes brighter and more reflective, this indicates that the solution is nearly exhausted.

De-smut by dipping in a solution containing a 30 % to 50 % volume fraction of nitric acid ($\rho = 1,42 \text{ g/cm}^3$).

For high-silicon-content alloys, add 10 % of hydrofluoric acid (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$) to the de-smutting solution. If high-silicon castings are not de-smutted satisfactorily, vapour-degrease them prior to anodizing.

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

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

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

22.15.3 Method P5 — Chemical polishing

Immerse the items in the following solution at around 95 °C for up to 2 min:

| | |
|--|----------|
| phosphoric acid (H_3PO_4) ($\rho = 1,75 \text{ g/cm}^3$) | 75 ml/l; |
| sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) | 20 ml/l; |
| nitric acid (HNO_3) ($\rho = 1,42 \text{ g/cm}^3$) | 5 ml/l. |

Following immersion, rinse the items (see 5.5) and then further clean them in a solution containing a 30 % volume fraction of nitric acid ($\rho = 1,42 \text{ g/cm}^3$) or 10 g/l of chromic acid (CrO_3) and, finally, rinse them again (see 5.5).

The amount of nitric acid lost by decomposition shall be limited to a volume fraction of 5 % to 10 %. Other constituents lost by drag-out may be replaced by additions of the original ingredient.

WARNING — Adequate exhaust fume extraction is needed due to the emission of nitrous fumes.

22.15.4 Method P6 — Electrobrightening

This method is suitable for super-purity aluminium (mass fraction 99,99 %) and alloys based on 99,99 %-purity aluminium.

Carry out anodic treatment at 75 °C to 80 °C and a potential of 20 V DC for about 20 min in the following solution:

| | |
|--|---------------------|
| sodium carbonate (Na_2CO_3) | 120 g/l to 200 g/l; |
| trisodium phosphate (anhydrous) (Na_3PO_4) | 25 g/l to 75 g/l. |

Then rinse the items (see 5.5) and remove the thin film formed during electrobrightening by immersion at 95 °C for approximately 2 min in the following solution:

| | |
|--|----------|
| phosphoric acid (H_3PO_4) ($\rho = 1,75 \text{ g/cm}^3$) | 35 ml/l; |
| chromic acid (CrO_3) | 20 g/l. |

22.15.5 Method P7 — Electropolishing

This method is unsuitable for silicon contents exceeding a mass fraction of 2 %.

Carry out anodic treatment at 70 °C to 80 °C, at a current density of approximately 2 A/dm² and a potential of 12 V to 15 V DC, for approximately 2 min to 5 min in the following solution:

| | |
|--|---------------------|
| phosphoric acid (H ₃ PO ₄) ($\rho = 1,75 \text{ g/cm}^3$) | 400 g/l to 800 g/l; |
| sulfuric acid (H ₂ SO ₄) ($\rho = 1,84 \text{ g/cm}^3$) | 100 g/l to 200 g/l; |
| chromic acid (CrO ₃) | 40 g/l to 100 g/l. |

22.16 Process Q — Cleaning of copper and copper alloys

22.16.1 Method Q1 — Cleaning in dichromate/sulfuric acid solution

Immerse the items at room temperature for up to 2 min in an aqueous dichromate/sulfuric acid solution, a typical composition being:

| | |
|---|----------|
| sodium dichromate (Na ₂ Cr ₂ O ₇ ·2H ₂ O) | 200 g/l; |
| sulfuric acid (H ₂ SO ₄) ($\rho = 1,84 \text{ g/cm}^3$) | 40 ml/l. |

22.16.2 Method Q2 — Cleaning in ferric sulfate/sulfuric acid solution

Immerse the items at 20 °C to 50 °C in a solution containing:

| | |
|---|-----------|
| ferric sulfate [Fe ₂ (SO ₄) ₃] liquor (containing approximately 640 g/l of ferric sulfate) | 150 ml/l; |
| sulfuric acid (H ₂ SO ₄) ($\rho = 1,84 \text{ g/cm}^3$) | 50 ml/l. |

22.17 Process R — Acid descaling, etching, chemical smoothing and electropolishing of copper and copper alloys

22.17.1 Method R1 — Descaling dip

Immerse the items at 25 °C to 50 °C in an aqueous solution containing 100 ml/l of sulfuric acid (H₂SO₄) ($\rho = 1,84 \text{ g/cm}^3$).

22.17.2 Method R2 — Bright dip

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

Immerse the items at room temperature in an aqueous solution containing:

| | |
|--|--|
| sulfuric acid (H ₂ SO ₄) ($\rho = 1,84 \text{ g/cm}^3$) | 500 ml/l; |
| nitric acid (HNO ₃) ($\rho = 1,42 \text{ g/cm}^3$) | 250 ml/l; |
| sodium chloride or hydrochloric acid | 1 g/l or 1 ml/l, respectively (this may be omitted). |

Rinse the treated items (see 5.5) with a minimum of delay.

WARNING — Efficient fume extraction is essential as the process results in the evolution of highly toxic fumes.

22.17.3 Method R3 — Chemical smoothing

Treat the items at room temperature as follows:

- a) degrease in accordance with process A (method A1) and process B (method B2);
- b) rinse thoroughly in cold water (see 5.5);
- c) immerse in an aqueous solution containing:

| | |
|--|---|
| hydrogen peroxide (H_2O_2) | 33 g/l to 50 g/l (e.g. 100 ml/l to 150 ml/l of 30 % mass fraction H_2O_2 solution); |
|--|---|

| | |
|--|-----------|
| sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$) | 1,3 ml/l. |
|--|-----------|

- d) immerse for 20 s in a solution of dilute sulfuric acid as specified in 22.17.1 (method R1).

The time of treatment required ranges from 15 min to 1 h, depending on the application.

The rate of metal dissolution depends upon the metal being treated and the temperature of the solution, but is of the order of 13 μm per hour. If the solution is used continuously, the dissolved copper shall be removed by circulation through an ion-exchange resin or by another suitable method.

22.17.4 Method R4 — Pickling in nitric acid

Immerse the items at room temperature for 2 min to 5 min in an aqueous solution containing a 15 % to 20 % volume fraction of nitric acid ($\rho = 1,42 \text{ g/cm}^3$). Oxygen-containing copper which has been given a cathodic alkaline cleaning treatment shall be given the minimum immersion time.

22.17.5 Method R5 — Electropolishing

Various solutions based on phosphoric acid may be used for the electropolishing of copper and its alloys. A recommended procedure is to use a solution containing a 70 % mass fraction of orthophosphoric acid ($\rho = 1,75 \text{ g/cm}^3$) in an aliphatic-alcohol/water mixture, with copper cathodes at a current density of 2 A/dm^2 to 5 A/dm^2 , at 20 °C to 25 °C.

22.18 Process S — Pickling of titanium and titanium alloys**22.18.1 General**

Immerse the items in an aqueous solution having the nominal composition given in method S1 or S2.

22.18.2 Method S1

| | |
|--|--------------------------|
| Hydrofluoric acid (HF) (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$) | > 40 ml/l but < 50 ml/l; |
|--|--------------------------|

| | |
|---|-------------------------|
| nitric acid (HNO_3) ($\rho = 1,42 \text{ g/cm}^3$) | not less than 200 ml/l. |
|---|-------------------------|

The solution shall be used at room temperature, but its temperature shall not be allowed to exceed 30 °C.

22.18.3 Method S2

| | |
|--|-------------------------|
| Hydrofluoric acid (HF) (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$) | not more than 120 ml/l; |
|--|-------------------------|

| | |
|---|-------------------------|
| nitric acid (HNO_3) ($\rho = 1,42 \text{ g/cm}^3$) | not less than 400 ml/l. |
|---|-------------------------|

The solution shall be used at room temperature, but its temperature shall not be allowed to exceed 30 °C.

The removal of scale may be improved by prior immersion in the alkaline permanganate solution specified in 22.19.4 (method T3).

22.19 Process T — Pickling, chemical polishing and electropolishing of nickel and its alloys

22.19.1 General

Immerse the items in an aqueous solution having the nominal composition given in one of methods T1 to T5.

NOTE The solutions used in methods T1 and T2 may promote preferential attack on nickel alloys unless the alloy is in a solution heat-treated condition.

22.19.2 Method T1

| | |
|--|-------------------------|
| Hydrofluoric acid (HF) (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$) | 50 ml/l; |
| nitric acid (HNO ₃) ($\rho = 1,42 \text{ g/cm}^3$) | not less than 200 ml/l. |

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

22.19.3 Method T2

| | |
|--|-----------|
| Hydrofluoric acid (HF) (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$) | 125 ml/l; |
| ferric sulfate [Fe ₂ (SO ₄) ₃ ·6H ₂ O] | 250 g/l. |

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

22.19.4 Method T3

| | |
|---|----------|
| Sodium hydroxide (NaOH) | 200 g/l; |
| potassium permanganate (KMnO ₄) | 150 g/l. |

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

After rinsing, remove loosened oxides, etc., by immersion in an acid solution such as one containing a 10 % volume fraction of sulfuric acid.

22.19.5 Method T4 — Chemical polishing

| | |
|--|-----------------------|
| Glacial acetic acid (CH ₃ COOH) | volume fraction 50 %; |
| nitric acid (HNO ₃) ($\rho = 1,42 \text{ g/cm}^3$) | volume fraction 30 %; |
| phosphoric acid (H ₃ PO ₄) ($\rho = 1,75 \text{ g/cm}^3$) | volume fraction 10 %; |
| sulfuric acid (H ₂ SO ₄) ($\rho = 1,84 \text{ g/cm}^3$) | volume fraction 10 %. |

Use the solution at 90 °C.

The preferred way of using this solution is to place it in a stainless-steel vat, covered with glass croffles (hollow spheres) and to immerse the items in stainless-steel baskets. After an immersion time of 1 min to 3 min, the items are rapidly rinsed (see 5.5). The nickel taken up in the solution precipitates as nickel sulfate. The sulfuric acid content needs to be maintained by periodic addition of concentrated sulfuric acid.

22.19.6 Method T5 — Electropolishing

Treat the items anodically at room temperature in an aqueous solution containing a 70 % volume fraction of sulfuric acid ($\rho = 1,84 \text{ g/cm}^3$) at a current density of $2,5 \text{ A/dm}^2$ and with the items anodic with respect to the tank.

22.20 Process U — Preparation of tin alloy for electroplating

- a) degrease with hot organic solvent in accordance with process A (method A1);
- b) clean, either by immersion or electrolytically, in a mild alkaline cleaner in accordance with process B (method B2);
- c) rinse in cold, running water (see 5.5);
- d) immerse in a solution containing a 10 % volume fraction of hydrochloric acid ($\rho = 1,16 \text{ g/cm}^3$) for approximately 2 min (for tin alloys containing lead, substitute this solution by a solution containing approximately 100 ml of 40 % mass fraction fluoroboric acid solution per litre);
- e) rinse in cold, running water (see 5.5);
- f) deposit approximately $5 \mu\text{m}$ of copper from a cyanide solution;
- g) rinse in cold, running water (see 5.5);
- h) electroplate with the desired metal as required.

22.21 Process V — Removal of surface contamination from gold coatings

A recommended method ^[4] is immersion in a solution containing a 1 % to 10 % mass fraction of triammonium citrate $[(\text{NH}_4)_3\text{C}_6\text{H}_5\text{O}_7]$ at pH 6,5 and 40°C to 80°C for 5 min to 10 min.

This method also improves the solderability of gold-plated items.

22.22 Process W — Preparation of aluminium alloys prior to electroplating

Treat the items in the following sequence of operations:

- a) Degrease in accordance with process A (method A1) and clean in accordance with process B (method B2).
- b) Acid pickle in accordance with either procedure 1) or 2):
 - 1) Immerse for 1 min at a temperature not exceeding 40°C in an aqueous solution containing:

| | |
|--|-------------------------|
| hydrofluoric acid (HF) (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$) | approximately 100 ml/l; |
| nitric acid (HNO_3) ($\rho = 1,422 \text{ g/cm}^3$) | approximately 100 ml/l. |

Renew the solution when its action becomes sluggish.
 - 2) Treat in chromic acid/sulfuric acid solution in accordance with process O.

NOTE This treatment is possibly slightly less satisfactory than the acid fluoride treatment described in procedure 1) above for promoting adhesion but, because of its less vigorous attack on the material, provides a smoother cleaned surface.

Following acid pickling, carry out an alkaline etching and de-smutting treatment in accordance with process P (method P4).

- c) Rinse thoroughly (see 5.5) and proceed immediately to the zincate treatment.
- d) Zincate treatment:
- 1) Immerse for 1 min in an aqueous solution containing approximately 500 ml/l of nitric acid ($\rho = 1,42 \text{ g/cm}^3$).
 - 2) Rinse (see 5.5).
 - 3) Immerse for about 3 min at room temperature in an aqueous solution containing:

| | |
|-------------------------|------------------------|
| zinc oxide (ZnO) | approximately 100 g/l; |
| sodium hydroxide (NaOH) | approximately 540 g/l. |
 - 4) Rinse (see 5.5).
 - 5) Repeat 1) to 4).
- e) Electroplate an undercoat of copper (or another metal) directly onto the zincate film.

In the case of copper, transfer the items to a Rochelle copper-plating bath of the following composition and plate for 10 min at 1 A/dm²:

| | |
|--|-------------------|
| sodium cyanide (NaCN) | 30 g/l to 45 g/l; |
| copper cyanide [Cu(CN) ₂] | 25 g/l to 35 g/l; |
| sodium potassium tartrate (KNaC ₄ H ₄ O ₆ ·4H ₂ O) (Rochelle salt) | 40 g/l to 60 g/l; |
| sodium carbonate (Na ₂ CO ₃) | 25 g/l to 50 g/l. |

Maintain the pH of the bath between 9,5 and 10,5 (using an electrometric technique) and the temperature between 35 °C and 45 °C. Make electrical contact with the items before immersing them in the bath.

- f) After copper deposition, rinse the items thoroughly (see 5.5) and transfer immediately, without drying, to the final plating bath.

22.23 Process X — Preparation of nickel alloys prior to electroplating

Degrease and clean in accordance with 13.1, followed by the following sequence of operations:

- a) Etch for a maximum of 1 min at room temperature in an aqueous solution containing:
- | | |
|---|-----------------------|
| ferric chloride (FeCl ₃) | 150 g/l to 200 g/l; |
| [or ferric chloride hexahydrate (FeCl ₃ ·6H ₂ O)] | 250 g/l to 330 g/l]; |
| hydrochloric acid (HCl) ($\rho = 1,16 \text{ g/cm}^3$) | 155 ml/l to 170 ml/l. |
- b) Carry out a nickel strike (not required prior to chromium plating) as follows:

Make the items cathodic in an aqueous solution containing:

| | |
|--|-----------------------------------|
| nickel chloride (NiCl ₂ ·6H ₂ O) | approximately 300 g/l to 400 g/l; |
| hydrochloric acid (HCl) ($\rho = 1,16 \text{ g/cm}^3$) | approximately 100 ml/l. |

Plate at room temperature with nickel anodes at a cathode current density of approximately 15 A/dm² for not more than 2 min.

- c) After the nickel strike, rinse the items thoroughly (see 5.5) and transfer to the final plating bath.

Surfaces previously nickel-plated shall be treated as the anode in a fresh solution containing a 30 % mass fraction of sulfuric acid ($\rho = 1,84 \text{ g/cm}^3$) at room temperature and a current density of 22 A/dm^2 for 5 min. On completion of this high-current-density treatment, make the items cathodic for about 2 s by current reversal, remove from the solution, rinse thoroughly (see 5.5) and transfer to the plating bath.

22.24 Process Y — Preparation of titanium alloys prior to electroplating

22.24.1 Method Y1 — General method for titanium alloys

This procedure is suitable for plating some titanium alloys, e.g. Ti-2Cu, Ti-4Al-4Mn, Ti-5Al-2,5Sn, Ti-2,5Al-11Sn-4Mo and Ti-3Al-11Cr-13V.

- a) Degrease in accordance with process A (method A1 or A2) and process B (method B2, without current) (see 14.1).
- b) Scour (method D3) using pumice powder moistened with an aqueous solution containing:

sodium hydroxide (NaOH) approximately 50 g/l;

sodium carbonate (Na_2CO_3) approximately 50 g/l.

Following scouring, rinse (see 5.5) and drain off the excess water.

- c) Etch in analytical-reagent grade concentrated hydrochloric acid ($\rho = 1,16 \text{ g/cm}^3$) at approximately 30°C . For some alloys, addition of 0,05 g/l of chloroplatinic acid [$\text{H}_2(\text{PtCl}_6)$] may improve coating adhesion. The optimum immersion time depends upon the composition of the alloy, and may range from 10 min to 2 h. If the solution is maintained at the boiling point, an immersion time of approximately 5 min may be adequate.
- d) Transfer rapidly to a rinsing solution containing 50 g/l of Rochelle salt (potassium sodium tartrate) in water. Use at room temperature and agitate the item well.
- e) Transfer without delay to a copper tartrate plating bath at room temperature containing:

copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) approximately 60 g/l;

Rochelle salt ($\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$) approximately 160 g/l;

sodium hydroxide (NaOH) approximately 50 g/l.

Plate at $0,4 \text{ A/dm}^2$ for 5 min. Make electrical contact with the items before immersing them in the bath.

- f) Rinse in one of the following two solutions:
- dilute sulfuric acid (approximately 1 % volume fraction solution) if subsequent plating is to be from an acid electroplating bath (e.g. nickel or chromium);
 - an approximately 5 % solution of sodium or potassium cyanide (expressed in terms of mass of cyanide salt per volume of solution) if subsequent plating is to be from a cyanide electroplating bath (e.g. silver).

Further electrodeposits may be applied by conventional methods to the thin copper undercoat. For maximum adhesion, the items shall be heat-treated at 450°C for 1 h after final plating.

Thicker copper plating is not recommended because of the risk of formation of brittle intermetallic alloys between the copper and the titanium on subsequent heat treatment.

22.24.2 Method Y2 — Method for titanium and selected titanium alloys

This method is suitable for commercially pure titanium and for Ti-6Al-4V, Ti-4Al-4Mn, Ti-3Al-11Cr-13V and Ti-2Cu only. It is not suitable for Ti-4Al-4Mo-2Sn-0,5Si or Ti-4Al-4Mo-4Sn-0,5Si.

- a) Degrease in accordance with process A (method A1 or A2) and process B (method B2, without current) (see 14.1).
- b) Immerse in hydrochloric acid ($\rho = 1,16 \text{ g/cm}^3$) for approximately 5 min at 90 °C to 110 °C or for approximately 1 h at room temperature, followed by rinsing in cold, running water (see 5.5).
- c) Transfer to a nickel strike solution of the following composition:

| | |
|---|---------------------|
| nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) | 150 g/l to 250 g/l; |
| hydrochloric acid (HCl) ($\rho = 1,16 \text{ g/cm}^3$) | 75 g/l to 125 g/l. |

Apply the strike at room temperature using nickel anodes and a cathode current density of 2,5 A/dm² to 2,9 A/dm². A minimum coating thickness of 1 µm is needed when the nickel strike is to be the base for subsequent electrodeposits.

- d) Rinse in cold, running water (see 5.5).
- e) Electroplate with the appropriate metal.

22.24.3 Method Y3 — Chemical-etching method

This method is suitable for commercially pure titanium and for Ti-6Al-4V, Ti-4Al-4Mn, Ti-3Al-11Cr-13V, Ti-5Al-2,5Sn and Ti-4Al-4Mo-2Sn-0,5Si.

- a) Degrease in accordance with process A (method A1 or A2) and process B (method B2, without current) (see 14.1).
- b) Etch in a solution of hydrofluoric acid/nitric acid in accordance with process S [method S1 (light etch) or method S2 (deep etch)] for 5 min at room temperature. The temperature of the solution shall not be allowed to exceed 30 °C.
- c) Rinse in cold, running water (see 5.5).
- d) Rinse in one of the following two solutions:
 - dilute sulfuric acid (1 % to 2 % volume fraction solution) if copper, nickel or chromium is to be the subsequent deposit;
 - dilute hydrochloric acid (2 % to 5 % volume fraction solution) if any other metal is to be electrodeposited.

If any electrodeposit other than copper, chromium or nickel is to be applied, a nickel strike coating of minimum thickness 1 µm shall be used.

- e) Electroplate with the required metal.

22.25 Process Z — Preparation of zinc and zinc alloys prior to electroplating

22.25.1 Method Z1

- a) Degrease in accordance with process A (method A1) and process B (method B1), followed by rinsing.
- b) Immerse in a 0,5 % to 5 % volume fraction sulfuric acid solution or a 1 % mass fraction hydrofluoric acid solution and then immediately rinse (see 5.5) in clean water to remove all traces of acid. The acid dip shall not be longer than 1 min or it may give rise to tarnishing.
- c) Immerse in a solution containing 50 g/l to 55 g/l of potassium cyanide, followed by rinsing in cold, running water (see 5.5).
- d) Deposit a minimum of 5 µm of copper from a cyanide solution. Make electrical contact with the items before immersing them in the plating bath.
- e) Rinse in cold, running water (see 5.5).
- f) Deposition of other metals can follow immediately.

22.25.2 Method Z2

- a) Degrease in accordance with process A (method A1) and process B (method B1).
- b) Rinse in cold, running water (see 5.5).
- c) Immerse for between 30 s and 2 min in the following solution:

| | |
|---------------------------------------|---------------------|
| sodium bisulfate (NaHSO_4) | mass fraction 90 %; |
| sodium fluoride (NaF) | mass fraction 10 %. |
- d) Immerse in a solution containing 50 g/l to 55 g/l of potassium cyanide.
- e) Rinse in cold, running water (see 5.5).
- f) Deposit a minimum of 5 µm of copper from a cyanide solution. Make electrical contact with the items before immersing them in the plating bath.
- g) Rinse in cold, running water (see 5.5).
- h) Deposition of other metals can follow immediately.

Annex A **(informative)**

Maintenance of organic and vapour degreasing baths

A.1 General

Baths used for organic-solvent immersion or for vapour degreasing need regular maintenance to ensure consistently satisfactory cleaning.

A.2 Solvent baths

A.2.1 Baths become heavily contaminated with use and hence become less effective. Replacement of the contaminated solvent with clean solvent is necessary at intervals depending on the extent of use.

A.2.2 With heavily soiled items, it is good practice to use two baths, one for the removal of the bulk of the grease, etc., and the second to complete the cleaning process. When the first bath becomes too heavily soiled, it should be discarded. The lightly soiled second bath can then be used as the first bath and a new second bath prepared with clean solvent.

A.2.3 Used solvent should be discarded or regenerated, depending on the circumstances. If it is regenerated, tests should be carried out to ensure that there is no progressive deterioration of the solvent (e.g. development of acidity due to breakdown products) or decrease in the inhibitor level.

A.3 Vapour and alkaline degreasing

Successful operation depends on the following factors:

- a) Maintenance of the solvent at the correct level in the sump.
- b) The redistillation of the solvent at sufficiently frequent intervals to prevent the accumulation of oil and grease. Both insoluble and soluble soil will accumulate in the sump. The insoluble matter may insulate heating elements and local overheating can result. The soluble matter will raise the boiling point of the solvent and the temperature will gradually rise. Temperatures above 120 °C for trichloroethylene or 160 °C for perchloroethylene are to be avoided or decomposition of the solvent may occur, producing acidity.
- c) The early removal of solid matter from the sump.
- d) Keeping all internal surfaces, particularly those through which the liquid is heated, clean and free from deposits.
- e) Ensuring that the solvent is free from acidity (see Annex B).
- f) The exclusion of water and water vapour so that there is no contact with the solvent or its vapour.

Annex B

(informative)

The control of acidity in vapour cleaning baths

B.1 Chlorinated solvents intended for use in vapour degreasing plants contain additives which absorb any acidity formed by degradation of the solvent during use. Knowledge of the absorptive capacity of such additives remaining in used solvent can be utilized to determine when the solvent requires replacement.

B.2 Suitable test kits, intended for use by semi-skilled operators, are now commercially available. Such kits enable an approximate acid acceptance value to be determined from which a reasonable assessment of the remaining working life of a solvent may be established without recourse to laboratory testing. The names and addresses of the suppliers of such kits can be obtained from solvent suppliers.

Annex C

(normative)

Method for the determination of oxidizable material in sulfuric acid

Add 15 ml of concentrated sulfuric acid ($\rho = 1,84 \text{ g/cm}^3$) carefully to 60 ml of water, cool and add to the mixture 0,10 ml of a 3,3 g/l potassium permanganate (KMnO_4) solution and stir. The pink colour should persist for 5 min, indicating the presence of a mass fraction of oxidizable material of not more than 11×10^{-6} , calculated as sulfur dioxide, in the sulfuric acid.

Annex D

(informative)

Method for determining free acidity

Weigh out $25 \text{ g} \pm 0,1 \text{ g}$ of the solution and transfer to a 100 ml graduated flask. Make up to 100 ml with water and mix. Stopper the flask and allow to settle for 2 h, then filter and titrate 50 ml of the filtrate with 1 M sodium hydroxide solution using methyl orange as indicator, matching the end point against methyl orange in a 2 % mass fraction solution of sodium dihydrogen phosphate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$).

1 ml of 1 M NaOH solution is equivalent to 0,098 g of H_3PO_4 .

Annex E (normative)

Selection of method of cleaning according to the tensile strength of the steel in zinc-coated steel items prior to chromate conversion coating

The following text is taken from ISO 27831-1:2008, Subclauses 7.1.1 to 7.1.3.

7.1.1 Selection of method of cleaning

The choice of cleaning methods for steels of tensile strength 1 000 MPa and above is restricted by the requirements of 7.1.2 and 7.1.3 in order to avoid embrittlement by hydrogen absorption during cathodic or acid treatments. In general, the methods given are suitable for both steel and iron. Treatment of cast iron in acid solutions is, however, liable to set up preferential etching around the graphite, especially around flake graphite, which may become detached, leaving holes which may trap treatment solution and give rise to subsequent staining or corrosion.

7.1.2 Stress relief heat treatment

Items made from steel of tensile strength 1 000 MPa and above which have been ground or subjected to heavy machining after final tempering shall be stress relieved in accordance with ISO 9587 or as specified by the purchaser. Any stress-relieving treatment shall be applied after degreasing (process A) and before any treatment liable to cause embrittlement (see 7.1.1). Items which have been treated in order to introduce beneficial compressive stresses into any part of the surface, e.g. by shot peening or cold working, shall not be heated above 230 °C.

7.1.3 General degreasing and cleaning

7.1.3.1 Steels of tensile strength up to 1 000 MPa

Degrease in accordance with process A (method A1 or A2) and process B (method B1, B2 or B3) prior to the commencement of a cleaning sequence selected from processes D to L, as appropriate.

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

Any form of cathodic cleaning treatment of these steels is incompatible. Degrease in accordance with process A (method A1 or A2) and process B (method B1, B2 or B3) prior to the commencement of a cleaning sequence selected from processes D, F, H and K, as appropriate.

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

Degrease in accordance with process A (method A1 or A2) and process B (method B1), with an applied anodic current, if needed. Alkaline de-rusting (method K1) may also be used. Neither cathodic- nor alternating-current treatment is applicable for these steels.

For cleaning, use abrasive cleaning (process D) and anodic pickling in a sulfuric acid solution (process H). If electrolytic de-rusting is needed, an alkaline electrolyte and anodic current shall be used.

7.1.3.4 Steels of tensile strength over 1 800 MPa

Degrease in accordance with process A (method A1 or A2) and process B (method B1), and for cleaning use abrasive cleaning process D (method D1) or anodic pickling in a sulfuric acid solution (process H).

Abrasive cleaning, especially grit blasting (method D1), leaves the surface readily corrodible. Therefore, further processing shall be carried out without delay.

If sulfuric acid cleaning is used instead of process D, the items shall be connected up and the current switched on before the items are immersed in the solution and, after treatment, the items shall be withdrawn rapidly before the current is finally switched off, and rinsed immediately (see 5.5). The sulfuric acid used to make up the solution shall at no time contain a mass fraction of more than 11×10^{-6} of oxidizable material, calculated as sulfur dioxide, determined by the method described in Annex C. This precaution is necessary because reduced sulfur, phosphorous and arsenic compounds in the acid promote hydrogen absorption during rinsing.

Process H and electrolytic de-rusting shall not be used for steels of tensile strength 1 800 MPa or greater. For these steels, abrasive cleaning (process D) shall only be used following process A and/or process B, in order to avoid any risk of hydrogen embrittlement. For electrolytic de-rusting of steels of tensile strength over 1 000 MPa, cathodic current or alternating current shall not be used.

Annex F (normative)

Method M4

The following text is taken from ISO 27831-1:2008, Subclause 10.12.4.

10.12.4 Method M4 — Preparation of corrosion-resisting steels prior to electroplating

Use one of the following (nickel strike) treatments, taking into account Annex E:

- a) Pickle the items anodically (process H) and then treat cathodically, with nickel or lead anodes, for 5 min at 16 A/dm² to 22 A/dm² in an aqueous solution, at 35 °C to 40 °C, containing

nickel sulfate (NiSO₄·6H₂O) approximately 225 g/l;

sulfuric acid (H₂SO₄) ($\rho = 1,84 \text{ g/cm}^3$) approximately 27 ml/l.

- b) Make the items anodic in an aqueous solution of the following approximate composition for not more than 2 min, then reverse the current so that they are cathodic for 6 min, at room temperature and with a current density of 3 A/dm²:

nickel chloride (NiCl₂·6H₂O) 240 g/l;

hydrochloric acid (HCl) ($\rho = 1,16 \text{ g/cm}^3$) 85 ml/l.

Use depolarized nickel electrodes without bags.

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

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

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

Bibliography

- [1] *General Galvanizing Practice*, Galvanizers Association, Sutton Coldfield, West Midlands, UK
- [2] THWAITES, C.J.: *Practical Hot Tinning*, Publication No. 575, ITRI Ltd, Unit 3, Curo Park, Frogmore, St Albans, Herts, UK
- [3] CLARKE, M., and CHAKRABARTY, A.M.: *Trans. Inst. Met. Finishing*, **48**, p. 122, 1970
- [4] RAY, G.P.: *Trans. Inst. Met. Finishing*, **56**, p. 168, 1978
- [5] Defence Standard 03-2, *Cleaning and preparation of metal surfaces*, Ministry of Defence, Directorate of Standardization, Kentigern House, 65 Brown Street, Glasgow, UK
- [6] BS 7773, *Code of practice for cleaning and preparation of metal surfaces*
- [7] *Pretreatments of Metals for Powder Coatings*, Paint Research Association, UK
- [8] RAY, G.P.: Hydrogen Embrittlement — A General Observation, *Proceedings of Hydrogen Embrittlement Seminar*, AESF, Orlando, FL, 30 January 2002, p. 42
- [9] RAY, G.P.: Hydrogen Embrittlement and Standardization, *Proceedings of Corrosion 2005, International Conference on Science and Economy — New Challenges*, Warsaw, Poland, Vol 1, p. 143, 2005
- [10] SHREIR, L.L., JARMAN, R.A., and BURNSTEIN, G.T.: *Corrosion*, Vols 1 and 2, Butterworth Heinemann, 1994
- [11] ASTM B 201, *Standard Practice for Testing Chromate Coatings on Zinc and Cadmium Surfaces*
- [12] ASTM B 117, *Standard Practice for Operating Salt Spray (Fog) Apparatus*
- [13] ASTM B 368, *Standard Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)*

