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

**Part 1:
Ferrous metals and alloys**

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

Partie 1: Métaux ferreux et alliages



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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 27831-1 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 1: 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 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;
- hot-dipped coatings;
- sprayed metal coatings;
- diffusion 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;
- de-rusting;
- chemical smoothing;
- electropolishing.

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

- non-corrosion-resisting steels, cast irons and pure irons;
- corrosion-resisting and heat-resisting steels.

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 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 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*

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 27831-1);
- b) the specification and metallurgical condition of the material of which the item is made;
- c) the tensile strength of the 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 7.1);
- 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 10.

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

7.1 Non-corrosion-resisting steels

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.

7.1.4 Preparation prior to electroplating

Degrease and clean in accordance with 7.1.3, taking into account the tensile strength of the steel, using:

- a) process A (method A1 or A2);
- b) process B (method B1, B2 or B3);
- c) processes D to F, as appropriate (method F, treatment in HCl, shall be avoided for spring temper and case-hardened items);
- d) process J or K, de-rusting;
- e) process G or, if inhibited solutions are used for acid pickling (process F), it may be necessary to remove adsorbed inhibitor from the surface by acid dipping (process G) or by a suitable alkaline solution (process B), before chemical smoothing (process L) and/or electroplating;
- f) process H, anodic pickling;
- g) process L, chemical smoothing.

For steels of tensile strength 1 800 MPa and above, see 7.1.3.4.

Tin, tin-zinc, lead and lead-tin electroplating is not recommended for steels of tensile strength over 1 000 MPa because of the danger of inter-granular penetration of the tin into the steel occurring at temperatures below the melting point of tin (232 °C) during embrittlement relief heat treatment at 190 °C to 220 °C as specified in ISO 9588 or as otherwise specified by the purchaser.

For steels of tensile strength 1 400 MPa and above, zinc and cadmium electrolyte shall not contain brightening agents, in order to avoid hydrogen embrittlement. Copper electrodeposition on steels of tensile strength 1 400 MPa and above is not recommended as it can introduce irreversible embrittlement and may adversely affect the fatigue properties.

7.1.5 Preparation prior to autocatalytic nickel plating

7.1.5.1 For steels having a tensile strength below 1 000 MPa, items may be treated as follows before autocatalytic nickel plating:

- a) degrease in accordance with process A (method A1) and process B (method B1) prior to the commencement of a cleaning sequence selected from processes D to F and K, as appropriate;
- b) clean by periodically reversed current (finishing anodically) in an alkaline solution;
- c) acid dip in a solution containing a 10 % volume fraction of sulfuric acid ($\rho = 1,84 \text{ g/cm}^3$) or in a solution containing a 10 % to 50 % volume fraction of hydrochloric acid ($\rho = 1,16 \text{ g/cm}^3$).

Some steels containing chromium and molybdenum may become passivated by anodic cleaning. For these steels, cathodic cleaning in place of cleaning by periodically reversed current, taking into account 7.1.3, may be employed.

A warm rinse may be found desirable to pre-heat large items immediately before autocatalytic nickel deposition, but care shall be taken to ensure that the items do not dry off before being immersed in the autocatalytic nickel solution.

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

7.1.5.2 For steels having a tensile strength in the range 1 000 MPa to 1 400 MPa, the following procedure shall be carried out:

- a) Degrease in accordance with process A (method A1) and process B (method B1) prior to the commencement of a cleaning sequence selected from processes D, F and K, as appropriate.
- b) Anodic etch (process H) for between 1 min and 3 min.

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

- c) Thoroughly rinse (see 5.5) to remove all viscous sulfate solution from the surface.

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

7.1.6 Preparation prior to phosphate treatment

Degrease and clean in accordance with the processes listed in 7.1.3, taking into account the tensile strength of the steel. Abrasive cleaning (process D) is preferred. If inhibited solutions are used for acid pickling (process F) or de-rusting (process J or K), it may be necessary to remove adsorbed inhibitors by an acid dip (process G) or by a suitable alkaline solution (process B) before rinsing and phosphating.

7.1.7 Preparation prior to sherardizing

Degrease and clean in accordance with the processes listed in 7.1.3, taking into account the tensile strength of the steel. For steels of tensile strength 1 400 MPa and above, sherardizing is unsuitable because it may adversely affect the properties of the component.

7.1.8 Preparation prior to hot-dip galvanizing ^[1]

7.1.8.1 Steel items other than castings

Degrease in accordance with process A (method A1 or A2) and process B (method B1) prior to one of the following:

- a) Acid pickling in accordance with process F (method F1 or F2). When necessary, a flux coating shall be applied.
- b) Abrasive blasting with chilled-iron or steel grit to process D (method D1) to remove welding slag or paint. After abrasive blasting, process F (method F1 or F2) may be used to remove residual iron dust from the surface prior to application of a flux coating if this is necessary.

For steels of tensile strength 1 400 MPa and above, hot-dip galvanizing is unsuitable as it may adversely affect the properties of the component.

7.1.8.2 Steel castings

Prepare using one of the following procedures:

- a) Abrasive blasting with chilled-iron or steel grit in accordance with process D (method D1) to remove moulding sand and iron scale. After the abrasive blasting, degrease in accordance with process A (Methods A1, A2) and process B (method B1) and clean by process F (method F1 or F2). When necessary, a flux coating shall be applied.
- b) Dilute a 30 % mass fraction solution of hydrofluoric acid ($\rho = 1,10 \text{ g/cm}^3$) with water to give a solution containing a 2 % to 10 % volume fraction of HF. Treat the items with this diluted solution, followed by hosing with water to remove any gelatinous products. After rinsing, degrease in accordance with process A (method A1 or A2) and process B (method B1). Clean in accordance with process F (method F2). When necessary, a flux coating shall be applied.

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

NOTE 2 Stresses induced by e.g. welding or hardening can result in grain boundary penetration of steels by zinc during hot dip galvanizing without appropriate stress-relieving treatments prior to galvanizing.

7.1.9 Preparation prior to metal spraying

7.1.9.1 Spraying for protection against corrosion and high-temperature oxidation

Degrease in accordance with process A (method A1 or A2) and process B (method B1) prior to abrasive cleaning by process D (method D1) with suitable abrasives (see 7.1.3). Metal spraying shall immediately follow abrasive cleaning.

For sheet steel of thickness 1,6 mm or less, some relaxation of the required degree of roughness may be acceptable in order to minimize distortion during surface preparation. Distortion can also be reduced by blast cleaning on both sides.

7.1.9.2 Spraying for reclamation (recycling) purposes

Degrease prior to the commencement of a cleaning sequence (see 7.1.9.1) which shall be in accordance with the processes and methods in 7.1.3 and appropriate to the subsequent metal-spraying process.

7.1.10 Preparation prior to the application of diffusion coatings

Degrease and clean the surfaces in question in accordance with one or more of the methods given in 7.1.3 appropriate to the tensile strength of the steel.

7.1.11 Preparation prior to vitreous enamelling

7.1.11.1 Conventional enamelling method

Degrease and clean as appropriate in accordance with a) to c):

- a) process A (method A1), especially items with welded seams;
- b) process B;
- c) process F (method F1 or F2), without wetting agents.

Deposit a coating of nickel by immersion in a solution of 10 g/l to 20 g/l nickel sulfate at a pH of 3,0 to 3,5 and at a temperature of 70 °C to 80 °C, followed by appropriate rinsing. The time of immersion shall be such that 0,40 g/m² to 0,60 g/m² of nickel is deposited on one side.

Immerse in the following neutralizing solution at a temperature of 70 °C to 80 °C:

sodium carbonate (Na ₂ CO ₃)	4,5 g/l;
borax (Na ₂ B ₄ O ₇ ·10H ₂ O)	1,5 g/l.

7.1.11.2 Alternative enamelling method

Degrease and clean in accordance with 7.1.11.1.

Deposit a nickel coating by immersion in either the solution specified in a) or that specified in b):

- a) Nickel reduction process:

nickel sulfate (NiSO ₄ ·6H ₂ O)	32 g/l;
sodium acetate (NaOOC-CH ₃)	12 g/l;
sodium hypophosphite (NaH ₂ PO ₂ ·H ₂ O)	7 g/l;
pH	4,5 to 5,8;
temperature	25 °C to 35 °C.

- b) Nickel replacement process:

nickel sulfate (NiSO ₄ ·6H ₂ O)	10 g/l to 20 g/l;
pH	3,0 to 3,5;
temperature	70 °C to 80 °C.

The time of immersion shall be such that 0,80 g/m² to 1,30 g/m² of nickel is deposited on one side of the sheet steel surface.

Following the nickel replacement process, immerse in neutralizing solution as described in 7.1.11.1.

7.1.12 Preparation prior to hot tinning

7.1.12.1 Preparation for ordinary mild-steel surfaces

Degrease and clean the items in accordance with 7.1.3.

7.1.12.2 Preparation of non-reactive mild-steel surfaces (TRI process) [2]

For steels having a non-reactive surface skin formed by the polymerization of certain lubricants present on the surface during low-temperature bright annealing, or during such operations as pressing and spinning, the following procedures are recommended:

- a) Heat treatment within the temperature range 700 °C to 850 °C for sufficient time for the component to reach the operating temperature of the furnace. The temperature employed shall be the lowest which will effectively oxidize the surface contamination and shall be determined by carrying out trials on test pieces from the same batch of material. Any loose scale formed shall be brushed off the component, which is then further treated in accordance with 7.1.3.
- b) Abrasive cleaning by process D (method D1 or D2).
- c) Etching in nitric acid, as follows. After carrying out 7.1.12.1, immerse in a solution containing an approximately 10 % volume fraction of nitric acid ($\rho = 1,42 \text{ g/cm}^3$) at a temperature of approximately 25 °C for 1 min to 3 min. Rinse (see 5.5) and then dip for approximately 1 min in dilute hydrochloric acid in accordance with process G (method G2). Rinse again (see 5.5) prior to fluxing and tinning.
- d) Etching in a nitric acid/sulfuric acid mixture, as follows. After carrying out 7.1.12.1, immerse for about 4 min to 6 min in a solution containing a 4 % mass fraction of nitric acid ($\rho = 1,42 \text{ g/cm}^3$) and a 20 % mass fraction of sulfuric acid ($\rho = 1,84 \text{ g/cm}^3$) at 25 °C to 40 °C.

7.1.13 Preparation prior to physical vapour deposition of cadmium and aluminium

Degrease and clean in accordance with 7.1.2 and 7.1.3, taking into account the cleaning procedures and requirements of ISO 22778 and ISO 22779.

7.2 Cast irons

7.2.1 General degreasing and cleaning

See 7.1.1 to 7.1.3.

7.2.2 Preparation prior to electroplating

See 7.1.4.

To overcome difficulties in electroplating certain castings with zinc, a strike with cadmium, tin, tin-zinc or acid zinc may be needed.

7.2.3 Preparation prior to autocatalytic nickel plating

See 7.1.5.

7.2.4 Preparation prior to phosphating and the application of other conversion coatings

See 7.1.6.

7.2.5 Preparation prior to hot tinning

7.2.5.1 “Direct chloride” method (TRI process) [2]

Before tinning, carry out the following procedure:

- a) Prepare the items mechanically by grinding, by machining (without cutting oils or coolants) or by coarse-abrasive blasting, in accordance with process D (method D1), with metallic shot or grit.
- b) Degrease in accordance with process A (method A1).
- c) Unless the mechanical preparation described in a) consists of fine grinding, either thoroughly grit blast with fine, angular chilled-iron grit which will pass through a sieve of 212 µm nominal aperture size or carry out fine-abrasive liquid blasting with alumina in the liquid.
- d) Immerse in an aqueous flux solution consisting of

zinc chloride (ZnCl_2)	400 g/l;
ammonium chloride (NH_4Cl)	25 g/l;
hydrochloric acid ($\rho = 1,16 \text{ g/cm}^3$)	100 ml/l (maximum).

When the items are tin-plated, use a flux cover, consisting of a 73 % mass fraction of zinc chloride, an 18 % mass fraction of sodium chloride and a 9 % mass fraction of ammonium chloride, on the first tinning bath. The initial flux cover shall be about 10 mm thick but, before use, it shall be thoroughly activated by spraying copiously with water, when it will become frothy and its thickness may increase to 50 mm to 75 mm. Lower the castings slowly through the flux cover into the tinning bath.

7.2.5.2 Preparation prior to electroplating with a readily tin-platable metal

To avoid the problems that can arise when tin-plating a graphite-contaminated surface, first electroplate with a readily tin-platable metal, such as iron, copper or nickel. For iron plating, use the following procedure:

- a) Carry out abrasive cleaning in accordance with process D (method D2) or tumble the items with a suitable fine abrasive (method D4).
- b) Degrease in accordance with process A (method A1).
- c) Acid pickle in accordance with process F (method F2).
- d) Electroplate using the following solution and conditions: [3]

ferrous ammonium sulfate $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ solution	300 g/l;
current density	1 A/dm ² to 1,5 A/dm ² ;
temperature	room temperature.

- e) Rinse (see 5.5), flux and tin the iron-plated castings immediately.

7.2.6 Preparation prior to hot-dip galvanizing

See 7.1.8.

7.2.7 Preparation prior to metal spraying

See 7.1.9.1.

7.2.8 Preparation prior to diffusion coating

See 7.1.10.

7.2.9 Preparation prior to vitreous enamelling

See 7.1.11.1.

8 Corrosion-resisting and heat-resisting steels

8.1 Corrosion-resisting steels

8.1.1 Stress-relieving heat treatment

Certain hardenable corrosion-resisting steels may need stress relieving after grinding or heavy machining. The stress relief heat treatment specified in 7.1.2 shall be used.

8.1.2 General degreasing and cleaning

Degrease prior to the selection of a cleaning sequence from 7.1.3, taking into account the tensile strength of the steel.

After degreasing, prepare the items in accordance with process M (method M1).

Electropolishing (method L2) or chemical polishing by a suitable method may also be used.

8.1.3 Preparation prior to electroplating

Degrease in accordance with process A (method A1) and process B (method B1) prior to the selection of a cleaning sequence from 7.1.3, taking into account the tensile strength of the steel. Then give the items a nickel strike in accordance with method M4, taking into account the tensile strength of the steel, before electrodeposition of the required metal(s).

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

8.1.4 Preparation prior to autocatalytic nickel plating

8.1.4.1 Degrease and clean in accordance with 7.1.3 and 7.1.5, taking into account the tensile strength of the steel, and then either give the items a nickel strike in accordance with method M4 or strike them electrolytically in the autocatalytic nickel solution until the surface is covered.

The surface may be activated by dipping for 1 min to 2 min in a solution containing a 50 % volume fraction of hydrochloric acid ($\rho = 1,16 \text{ g/cm}^3$) at a temperature of 40 °C to 50 °C.

8.1.4.2 After rinsing in water, transfer the items directly to the autocatalytic nickel bath for autocatalytic nickel plating in accordance with ISO 4527 or another appropriate International Standard.

8.1.5 Preparation prior to metal spraying

8.1.5.1 Preparation for metal spraying

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

8.1.5.2 Spraying for reclamation (recycling) purposes

Degrease the items thoroughly prior to the commencement of a cleaning sequence in accordance with the processes and methods in 7.1.3 and appropriate to the subsequent metal-spraying process.

8.1.6 Preparation prior to vitreous enamelling — Austenitic steels

8.1.6.1 Degrease and clean, taking into account the tensile strength of the steel, using either the sequence:

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

or the sequence:

- d) heat treatment at approximately 800 °C, provided this temperature is suitable for the material, to burn off any excess grease;
- e) process D (method D2);
- f) process A.

8.1.6.2 The following procedure may also be suitable:

- a) 8.1.6.1 a) to c), as appropriate;
- b) etch 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 by immersion for 5 min to 20 min;
- c) immerse in the following neutralizing solution for 3 min to 5 min at a temperature of 70 °C to 80 °C:

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.

8.1.6.3 The following procedure may also be suitable:

- a) 8.1.6.1 d) to f);
- b) 8.1.6.2 b) and c).

8.2 Heat-resisting steels

For nickel, cobalt and iron base heat-resisting alloys, a possible suitable procedure is to degrease in accordance with process A and process B and clean in accordance with process D (method D2), process F (method F5) and process K, as appropriate. For descaling, immersion in a fused sodium hydroxide bath containing oxidizing salts such as sodium nitrate at 480 °C to 490 °C for a few minutes and quenching in cold water followed by process F (method F5) may be suitable.

9 Pretreatment of metals prior to powder coating

9.1 General degreasing and cleaning

Degrease in accordance with 7.1.3.

9.2 Preparation of non-corrosion-resisting steels

- a) With steel items other than castings, degrease and clean in accordance with 7.1.3, taking into account the tensile strength of the steel. If, prior to powder coating, items are phosphated or given another conversion coating, degrease and clean in accordance with 7.1.3 and 7.1.6.
- b) With steel castings, degrease and clean in accordance with 7.1.8.2 a).

9.3 Preparation of corrosion-resisting steels

Degrease in accordance with process A (method A1 or A2) and process B (method B1, B2 or B3) and clean in accordance with process D (method D2).

10 Cleaning and preparation processes

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.

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.

10.1 Process A — Organic-solvent degreasing and cleaning

10.1.1 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.

10.1.2 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 ISO 27831-2), 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.

10.1.3 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 naphtha.

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.

10.1.4 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.

10.1.5 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.

10.1.6 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.

10.1.7 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.

10.2 Process B — Alkaline degreasing

10.2.1 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 7.1.1 to 7.1.3). 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.

10.2.2 Method B1 — General-purpose alkaline degreasing

The cleaning agent may contain any of the constituents given in 10.2.1. 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.

10.2.3 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 7.1.1 to 7.1.3).

10.2.4 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 7.1.1 to 7.1.3).

10.3 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.

10.4 Process D — Abrasive cleaning

10.4.1 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.

10.4.2 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 ferrous items.

10.4.3 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.

10.4.4 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 (see ISO 27831-2).

10.4.5 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.

10.4.6 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.

10.5 Process E — Caustic-alkali descaling

10.5.1 Method E1 — Sodium hydride descaling

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

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

sodium hydroxide (NaOH) mass fraction 98 %;

sodium hydride (NaH) mass fraction 2 %.

Remove from the molten-salt bath and quench in cold, running water with a final rinse (see 5.5) in hot water.

10.5.2 Method E2 — Alkali permanganate descaling

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

sodium hydroxide (NaOH) 200 g/l to 250 g/l;

potassium permanganate (KMnO₄) 10 g/l to 20 g/l.

The immersion time is dependent upon the extent of the scale. Following descaling, adequate rinsing and a short immersion in hydrochloric acid (mass fraction 10 %, $\rho = 1,16 \text{ g/cm}^3$), preferably inhibited, to remove any remaining loosened scale. In hot alkaline solution, potassium permanganate tends to decompose and needs to be replaced regularly.

10.6 Process F — Acid pickling without electrolysis

10.6.1 General

Immerse the items in one of the aqueous solutions specified in 10.6.2 to 10.6.6.

10.6.2 Method F1

Use an approximately 50 ml/l to 100 ml/l solution of concentrated sulfuric acid ($\rho = 1,84 \text{ g/cm}^3$), with or without inhibitors and/or wetting agents, between 20 °C and 45 °C. For pickling corrosion-resisting steels, the concentration of the sulfuric acid may need to be increased to approximately 200 ml/l. Sulfuric acid is unsuitable for galvanized steel.

10.6.3 Method F2

Use a 100 ml/l solution of concentrated hydrochloric acid ($\rho = 1,16 \text{ g/cm}^3$), with or without inhibitors and/or wetting agents, at room temperature.

10.6.4 Method F3

Use one of phosphoric acid solutions specified below, with or without inhibitors and/or wetting agents, at the temperature given:

- a) a solution containing a 30 % to 50 % volume fraction of phosphoric acid ($\rho = 1,75 \text{ g/cm}^3$), used at room temperature;
- b) a solution containing a 15 % to 30 % volume fraction of phosphoric acid ($\rho = 1,75 \text{ g/cm}^3$), used at a temperature between 60 °C and 70 °C.

More dilute solutions of phosphoric acid may be used to remove brazing fluxes.

The solution shall contain not more than

2 g/l of sulfate, calculated as sodium sulfate;

1 g/l of chloride, calculated as sodium chloride;

a mass fraction of 8×10^{-6} , in total, of arsenic and/or antimony (see 5.1).

Inhibitors may be added to the solution to reduce attack on the basis or substrate metal. Any inhibitor used shall be completely miscible with the solution and shall not decompose or separate on storage or dilution.

10.6.5 Method F4

The following solution and conditions are typical of those used for removing light scale from austenitic stainless steels:

ferric sulfate [$\text{Fe}_2(\text{SO}_4)_3$] liquor (mass fraction 40 %, $\rho = 1,50 \text{ g/cm}^3$)	200 ml/l to 300 ml/l;
hydrofluoric acid (HF) (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$)	50 ml/l to 75 ml/l;
temperature	60 °C to 70 °C;
immersion time	2 min to 30 min.

10.6.6 Method F5

Use a solution containing the following acids at the concentrations indicated:

nitric acid (HNO_3) ($\rho = 1,42 \text{ g/cm}^3$)	100 ml/l;
hydrofluoric acid (HF) (40 % mass fraction solution, $\rho = 1,128 \text{ g/cm}^3$)	50 ml/l.

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

NOTE 2 When items are to be subsequently chemically smoothed (process L), phosphated or electroplated, passive surfaces formed following a pickling operation can cause adhesion problems in the absence of an acid dip (process G).

10.7 Process G — Acid dipping

10.7.1 General

Immerse for not more than 2 min in one of the uninhibited aqueous solutions specified in 10.7.2 or 10.7.3.

10.7.2 Method G1

A solution containing 50 ml/l to 100 ml/l of sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$), used either warm or at room temperature.

10.7.3 Method G2

A solution containing 50 ml/l to 100 ml/l of hydrochloric acid (HCl) ($\rho = 1,16 \text{ g/cm}^3$), used at room temperature.

10.8 Process H — Anodic pickling

10.8.1 General

In anodic pickling, contamination of the pickling solution with chloride shall be avoided. The essential aim of anodic pickling of steel is to render the steel passive, a condition that will be indicated by a sharp rise in voltage between the steel 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.

Anodic pickling may not be effective and may cause pitting of the internal surfaces of tubular items unless auxiliary cathodes are employed. Where it is desired to clean only the external surfaces, such items shall be plugged to prevent ingress of the electrolyte.

Typical anodic pickling procedures are given in 10.8.2 to 10.8.4.

10.8.2 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. If the current density interruption method, which is not a safe procedure because of the high risk of hydrogen embrittlement, is used on steel of tensile strength in the range 1 400 MPa to 1 800 MPa (see 7.1.3), a safe procedure shall be established and agreed with the purchaser prior to its implementation.

10.8.3 Method H2

Immerse the items in an aqueous solution (ρ at least 1,70 g/cm³) containing 750 ml/l of sulfuric acid ($\rho = 1,84$ g/cm³), with or without a small addition of chromic acid. The solution strength shall be maintained by periodic additions of sulfuric acid to keep the relative density greater than 1,70 g/cm³. An initial current density of at least 10 A/dm² (4 V to 12 V) shall be used. The temperature shall not exceed 25 °C.

10.8.4 Method H3

An alternative aqueous solution (ρ at least 1,20 g/cm³) for use on corrosion-resisting steel prior to a nickel strike (process M, Method M4) contains 200 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 1,20 g/cm³. A current density of 20 A/dm² to 25 A/dm² (approximately 6 V) shall be used. The temperature shall not exceed 20 °C. A suitable immersion time is about 1 min to 3 min.

10.9 Process J — Acid de-rusting

10.9.1 Rust, i.e. iron and steel corrosion products, may be removed by immersion in any of the solutions described under process F (see 10.6). For steels of tensile strength over 1 000 MPa, see 7.1.3.

Process F shall not, however, be used for

- a) springs in stress;
- b) items where rusting has rendered them beyond repair.

Before subsequent chemical smoothing in accordance with process L (see 10.11), phosphating or electroplating, it is necessary to acid dip in accordance with process G (see 10.7).

10.9.2 Immersion in a solution containing a 3 % to 10 % mass fraction of triammonium citrate $[(\text{NH}_4)_3\text{C}_6\text{H}_5\text{O}_7]$ at pH 6,65 and a temperature of 70 °C to 80 °C for 5 min to 10 min removes most of the chloride ions from the fissures and crevices in the surface of the items [4].

10.10 Process K — Alkaline de-rusting

10.10.1 General

Rust, i.e. iron and steel corrosion products, may be removed and the surface prepared for electroplating by one of the methods specified in 10.10.2 to 10.10.4.

10.10.2 Method K1 (for light rust)

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

10.10.3 Method K2 (for heavy rust)

Immerse in an electrolyte consisting essentially of sodium hydroxide in water. The solution may also contain sodium cyanide or ethylenediaminetetraacetic acid and wetting agents.

Typical compositions are:

- | | | |
|----|---------------------------------|---------------------|
| a) | Sodium hydroxide (NaOH) | 200 g/l to 300 g/l; |
| | sodium cyanide (NaCN) | 25 g/l; |
| | wetting agent | 6 g/l; |
| | water | 4,5 l. |
| b) | Sodium hydroxide (NaOH) | 100 g/l; |
| | ethylenediaminetetraacetic acid | 100 g/l; |
| | non-ionic wetting agent | 1,5 g/l; |
| | water | 4,5 l. |
| c) | Sodium hydroxide (NaOH) | 200 g/l to 300 g/l; |
| | sodium gluconate | 50 g/l to 100 g/l. |

The operating temperature shall be between room temperature and 60 °C. With solutions containing cyanide, the operating temperature shall be below 40 °C to avoid decomposition of the cyanide. To enhance the de-rusting process, the items may, in addition, be made anodic or cathodic, or in certain circumstances periodically reversed current may be used, with current densities of the order of 2,5 A/dm² to 5 A/dm² (approximately 10 V to 12 V).

After de-rusting, items shall be rinsed thoroughly (see 5.5) in cold, running water, special attention being paid to any crevices.

When residual alkali on the surface is undesirable, e.g. on surfaces to be phosphated or given organic coatings, the cold-water rinse shall be followed by a rinse in phosphoric acid solution (containing a volume fraction of approximately 0,5 %).

This method is not applicable to

- steels of tensile strength over 1 000 MPa;
- assemblies containing aluminium or one of its alloys;
- springs;
- items susceptible to caustic embrittlement;
- items where rusting has rendered them beyond repair.

10.10.4 Method K3 (for heavy rust)

Use the solution specified for method K1 (see 10.10.2), but electrolytically.

10.11 Process L — Chemical smoothing and electropolishing of steels

10.11.1 Method L1 — Chemical smoothing process (for non-corrosion-resisting steels)

Treat the items as follows:

- a) Degrease in accordance with process A (Method A1) and/or alkaline clean in accordance with process B (method B1).
- b) Acid dip in accordance with process G (method G1).
- c) Rinse thoroughly in cold water (see 5.5).
- d) Immerse in an aqueous solution containing:

oxalic acid ($\text{H}_2\text{OOC-COOH}_2$)	25 g/l;
hydrogen peroxide (H_2O_2)	13 g/l (e.g. 40 ml/l of 30 % mass fraction H_2O_2 solution);
sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$)	0,053 ml/l.

Use this solution at room temperature.

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

Hydrogen peroxide decomposes quite rapidly, so additions need to be made after every 20 min of use to maintain the strength at its original level.

10.11.2 Method L2 — Electropolishing of corrosion-resisting steels

Following stages a) to c) of method L1 above, treat the corrosion-resisting (martensitic, ferritic or austenitic) steel items either in the solution specified in a) or that specified in b):

- a) immerse in a solution containing

phosphoric acid (H_3PO_4) ($\rho = 1,75 \text{ g/cm}^3$)	500 ml/l,
sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$)	450 ml/l

at a temperature of 70 °C to 90 °C and a current density of 20 A/dm² to 30 A/dm² (with the items anodic);

b) or immerse in a solution containing

phosphoric acid (H_3PO_4) ($\rho = 1,75 \text{ g/cm}^3$)	600 ml/l,
chromic acid (CrO_3)	150 g/l

at a temperature of 30 °C to 80 °C and a current density of 10 A/dm² to 100 A/dm² (with the items anodic).

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

10.12.1 Method M1 — Passivation

A suitable procedure consists of immersion for about 20 min at 20 °C to 50 °C in a solution containing

nitric acid (HNO_3) ($\rho = 1,42 \text{ g/cm}^3$)	190 ml/l to 210 ml/l;
sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$)	20 g/l to 30 g/l (may be omitted when “passivating” austenitic stainless steel).

This treatment shall be followed by rinsing in cold water and, in the case of ferritic and martensitic steels, by immersion for about 30 min in a 50 g/l solution of sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$) at about 6 °C and, finally, rinsing again in cold water.

10.12.2 Method M2 — Test for surface contamination

Swab the items for a few minutes with an aqueous solution containing

copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)	approximately 4 g/l;
sulfuric acid (H_2SO_4) ($\rho = 1,84 \text{ g/cm}^3$)	approximately 1,5 ml/l.

If, after drying, there are any areas of deposited copper present, this indicates that the surface is unsatisfactory and needs to be re-passivated and submitted for re-approval.

10.12.3 Method M3 — Alternative test for surface contamination

Immediately after the cleaning treatment, place one spot of the following solution on a flat area of the treated surface and allow it to remain there for 3 min:

palladium chloride ($\text{PdCl}_2 \cdot 2\text{H}_2\text{O}$)	0,5 g;
hydrochloric acid (HCl) ($\rho = 1,16 \text{ g/cm}^3$)	20 ml;
water	98 ml.

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

No trace, or only a slight trace, of a dark deposit shall be evident on the area tested showing that passivation has been performed successfully. This test may be carried out on a steel component or on a flat, smooth-surfaced test piece made from material of the same composition as the component which has been processed with the actual component in the same way.

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

Use one of the following (nickel strike) treatments, taking into account 7.1.1 to 7.1.3:

- 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 (see 5.5) and transfer them to the final plating bath.

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.

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