INTERNATIONAL STANDARD

ISO 9717

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Metallic and other inorganic coatings — Phosphate conversion coating of metals

Revêtements métalliques et autres revêtements inorganiques — Couches de conversion au phosphate sur métaux

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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 9717 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 8, *Chemical conversion coatings*.

This second edition cancels and replaces the first edition (ISO 9717:1990), which has been technically revised.

Introduction

Phosphate conversion coatings are applied to ferrous metals, aluminium, zinc and their alloys (including zinc- and zinc-alloy-plated steel, cadmium and their alloys) either as an end finish or as an intermediate layer for other coatings. They are intended to

a) impart corrosion resistance,

b) improve adhesion to paints and other organic finishes,

c) facilitate cold-forming operations, such as wire drawing, tube drawing and extrusion, and

d) modify surface frictional properties so as to facilitate sliding.

Phosphate conversion coatings are produced by treatment with solutions, the main constituents of which are the appropriate dihydrogen orthophosphates. These coatings are applied principally to ferrous materials, aluminium, zinc and cadmium and differ in coating mass per unit area and apparent density, depending on

a) the construction material and surface condition of the components,

b) previous mechanical and chemical treatment of the components, and

c) processing conditions for phosphating.

All phosphate conversion coatings are more or less porous but can be sealed substantially by subsequent sealing processes.

Metallic and other inorganic coatings — Phosphate conversion coating of metals

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1 Scope

This International Standard specifies requirements for the processing of ferrous metals, aluminium, zinc, cadmium and their alloys to produce coatings consisting essentially of inorganic phosphates, which are intended to be used in conjunction with supplementary treatments for the protection of the basis metal against corrosion and to provide anti-wear properties to sliding surfaces, adhesion to organic finishes and ease of cold-forming operations.

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 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

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 2819, *Metallic coatings on metallic substrates — Electrodeposited and chemically deposited coatings — Review of methods available for testing adhesion*

ISO 2859 (all parts), *Sampling procedures for inspection by attributes*

ISO 3892, *Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods*

ISO 4519, *Electrodeposited metallic coatings and related finishes — Sampling procedures for inspection by attributes*

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 15724, *Metallic and other inorganic coatings — Electrochemical measurement of diffusible hydrogen in steels — Barnacle electrode method*

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

ISO 27831-2, *Metallic and other inorganic coatings — Cleaning and preparation of metal surfaces — Part 2: Non-ferrous metals and alloys*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1463, ISO 2064, ISO 2080, ISO 2819, ISO 9587 and ISO 9588 and the following apply.

3.1
free acid of a phosphating solution
number of millilitres of 0,4 % mass fraction of sodium hydroxide solution required to neutralize 10 ml of the phosphating solution to pH 4, using methyl orange or an equivalent indicator system

3.2
pointage
measure of the total acidity of a phosphating solution, i.e. the number of millilitres of 0,4 % mass fraction of sodium hydroxide solution required to neutralize 10 ml of the phosphating solution to pH 10 using phenolphthalein as an indicator

3.3
accelerated process
process where the processing solution is of the metal phosphate/phosphoric acid type containing additions such as oxidizing agents, e.g. nitroguanidine, nitrate, nitrite, or chlorate, which accelerate the coating formation

3.4
unaccelerated process
process where the processing solution is of the plain metal phosphate/phosphoric acid type and no accelerating agents are employed

3.5
sealing
application of a supplementary coating to the phosphate surface immediately after phosphating, rinsing and drying, to form a complete protective or lubrication system

3.6
supplementary coating
covering material, usually of an organic nature

EXAMPLES oil, grease, lacquer, varnish, paint or dry lubricant

4 Information to be supplied by the purchaser to the processor

4.1 Essential information

When ordering articles to be phosphated in accordance with this International Standard, the purchaser shall provide the following information in writing, in, for example, the contract or purchase order, or on engineering drawings:

a) a reference to this International Standard, ISO 9717:2010, and the designation (see Clause 5);

b) the nominal composition, specification, nature and metallurgical condition of the basis metal, if they are likely to affect the serviceability and/or the appearance of the coating;

c) the significant surfaces, to be indicated on drawings of the parts or by providing suitably marked specimens;

d) the coating classification code (see 5.4);

e) the coating mass per unit area, or the coating thickness with any tolerance limit required, and the relevant test method (see 6.4);

f) tensile strength of parts and the requirements of heat treatment before and/or after the phosphating process (see 6.5 and 6.6);

g) the surface preparation required prior to phosphate conversion coating (see 6.1);

h) the surface appearance (see 6.1);

i) the requirements for, and type of, supplementary treatments, e.g. staining, oil, grease, organic coatings, etc. (see 6.7);

j) the requirement for a quality-evaluation test for controlling the quality and continuity of the coating, test method to be used and minimum exposure time (see 6.9);

k) the sampling methods, acceptance levels and/or any other inspection requirements (see Clause 7).

4.2 Additional information

The following additional information shall also be provided by the purchaser, when appropriate:

a) for creviced items, the phosphate process and/or nature of accelerator employed;

b) for high-strength steels, details of any special process requirements;

c) the properties of the coating, such as surface profile and crystal size;

d) any other requirements, such as cascaded process in rinsing or whether removal of supplementary coatings is needed prior to examination of corroded test specimens (see 6.3.1, 6.8, 6.9 and Annex F).

5 Designation

5.1 General

The designation shall appear on engineering drawings, in the purchase order, in the contract or in the detailed product specification.

The designation specifies, in the following order, the basis metal, the specific alloy (optional), stress-relief requirements, the thickness (or mass) and composition of the phosphate coating, heat treatment to reduce susceptibility to hydrogen embrittlement, and treatments for, including the type of, supplementary coating.

5.2 Designation specifications

The coating designation specifies the basis metal and the types and thickness of coatings appropriate for each service condition number and comprises the following:

a) the term, “Phosphate conversion coating”, the number of this International Standard, ISO 9717, followed by a hyphen;

b) the chemical symbol for the basis metal (or for the principal metal if an alloy) followed by a solidus (/) as follows:

   — Fe for iron or steel;

   — Zn for zinc or zinc alloys;

   — Al for aluminium or aluminium alloys;

   — Cd for cadmium;

c) the designation SR (see 5.3), if necessary, followed by a solidus;

d) a symbol describing the type of coating;

e) a number indicating the coating mass per unit area, in grams per square metre, followed by a solidus;

f) a symbol indicating additional treatments for supplementary coating of the phosphate layer followed by a solidus (see Table E.1);

g) the designation ER (see 5.3), if necessary, followed by a solidus;

h) a symbol indicating any additional treatments for supplementary coating of the phosphate layer.

Solidi (/) shall be used to separate data fields in the designation corresponding to the different sequential processing steps. Double separators or solidi indicate that a step in the process either is not required or has been omitted.

If supplementary treatments other than, or in addition to, sealing are used, the designation shall be Fe/ZnMeph25/X/Y, where ZnMeph represents appropriate metal phosphate if zinc remains as the main metal constituent of the coating or phosphate of a double salt with, for example, 25 g/m^2 coating mass per unit area, and X and Y represent the supplementary coating codes given in Table F.1.

It is recommended that the specific alloy be identified by its standard designation following the chemical symbol of the basis metal; for example, its UNS number, or the national or regional equivalent, may be placed between the symbols < >. For example, Fe<G43400> is the UNS designation for one high-strength steel. (See Reference [6] in the Bibliography).

5.3 Designation of heat treatment requirements

The heat treatment requirements shall be in brackets and designated as follows:

a) the letters SR, for stress-relief heat treatment prior to electroplating, and/or the letters ER, for hydrogen-embrittlement-relief heat treatment after electroplating;

b) in parenthesis, the minimum temperature, in degrees Celsius;

c) the duration of the heat treatment, in hours.

For example, [SR(210)1] designates, without square brackets, stress-relief heat treatment at 210 °C for 1 h.

EXAMPLE 1 Designation of a zinc-phosphate-type coating (Class II) on iron or steel at a mass per unit area of 5 g/m^2 with after-treatments of inorganic sealants (T2) and organic coating or varnishes (T1):

Phosphate conversion coating ISO 9717 – Fe/Znph5/T2/T1

EXAMPLE 2 Designation of a zinc phosphate coating of 5 g/m^2 on iron or steel (Fe) which is stress relieved at 200 °C for 3 h prior to phosphate coating, is heat-treated after phosphate coating for hydrogen-embrittlement relief for 8 h at 190 °C [ER(190)8] and has been given an inorganic sealant (T2) and a treatment for supplementary coating, such as an organic coating (T1):

Phosphate conversion coating ISO 9717 – Fe/SR(200)3/Znph5/ER(190)8/T2/T1

5.4 Classification of phosphate conversion coating

The classification of phosphate coatings for steel parts is used by the purchaser to specify the degree of protection required or where parts are deformed during further fabrication after application of supplementary coatings:

I) Maximum corrosion protection (not less than 7,5 g/m^2)

A coating consisting essentially of inorganic phosphates of manganese or iron and having a mass of not less than 7,5 g/m^2 of treated surface. The coating is produced by certain immersion-type accelerated or unaccelerated processes. This class is used normally when sealing with oil or grease supplementary treatments is specified, and maximum corrosion protection (or wear resistance) is desired. It is not recommended for use under organic coatings, varnish, and lacquer finishes on sheet materials less than 1 mm thick.

II) General protective purpose (not less than 4,5 g/m^2)

A coating consisting essentially of inorganic phosphates of zinc and having a mass of not less than 4,5 g/m^2 of treated surface. The coating is usually produced by an immersion-type accelerated process. This class will normally be used for general protective purposes under organic coating systems. A Class I coating may be used when a Class II coating is called for, except on thin sheet materials.

III) Pretreatment of thin sheet-steel sections (1,5 g/m^2 to 4,5 g/m^2)

A coating consisting essentially of inorganic phosphates of zinc or iron and having a mass within the range 1,5 g/m^2 to 4,5 g/m^2 of treated surface. The coating is usually produced by a spray-type accelerated process. This class will normally be used only for the pretreatment of thin sheet-steel sections under organic coatings, varnish, or lacquer coatings of high protective value.

IV) For fabrication following organic or lacquer coatings (0,2 g/m^2 to 1,5 g/m^2)

A coating consisting essentially of inorganic phosphates of zinc or iron having a mass within the range 0,2 g/m^2 to 1,5 g/m^2 of treated surface. This class is normally used where items are to be deformed during further fabrication after the application of organic coatings, varnish, or lacquer.

Other coating types exist, including zinc phosphate modified with iron and/or nickel and/or manganese. The modifying metal will normally be present in the form of a double salt such as $Zn_2Me(P0_4)_2{\cdot}4H_20$, where Me represents Fe(II), Ni or Mn., if zinc remains the main metal constituent of these coatings, which, to avoid confusion, have not been given separate designations. Metal from the substrate material will often be incorporated in the conversion coating.

6 Requirements

6.1 Surface preparation

All items shall be prepared in accordance with ISO 27831-1 or ISO 27831-2, as appropriate, to produce a chemically clean surface, unless otherwise specified by the purchaser. Items with folds, seams, or crevices shall receive special attention to ensure removal of oil, grease, or other foreign matter. The method of removal shall be chosen with due regard to its effect on the properties of the item and the formation of high-quality phosphate coatings.

After alkali or acid cleaning, the items shall be rinsed thoroughly in cold or hot water to remove all residues of cleaning materials that would otherwise affect the quality of the coating or the efficacy of the phosphating solution. Where acid pickling has been employed, it may be necessary to follow the use of pickling solutions containing wetting agents or inhibitors by dipping in an acid solution without a wetting agent or inhibitor, or by dipping in a suitable alkaline solution in order to remove adsorbed films.

Both acid and strong alkali treatments can result in the formation of coarse crystalline phosphate coatings of poor quality. Post-cleaning conditioning treatments to prevent such coarse coatings may be used. The post-cleaning conditioning treatments are intended to remove all traces of residual acid or alkali solution, i.e. a mild alkali solution to remove all traces of acid and a mild acid solution to remove all traces of alkali [see 4.1 g)].

A conditioning rinse is normally used immediately prior to phosphating, to favour the formation of fine grain coatings without further rinsing. Materials based on titanium salts are also used and are widely available from proprietary sources. It is also possible to incorporate such materials in mildly alkaline spray cleaners, thus obviating the need for a separate conditioning rinse. However, in this case, rinsing before phosphating is essential.

6.2 Phosphate conversion coating

Only processes capable of meeting the requirements of this International Standard shall be used.

Composite items made up of ferrous and non-ferrous (such as aluminium, magnesium, nickel) items shall normally have their ferrous items phosphated before assembly. These can be zinc phosphated, provided that the baths are appropriately modified for this type of applications. Exceptions can be allowed in cases of composite items containing zinc-base material or copper-base material (the copper-base material should not constitute more than about 10 % of the total surface) provided that the joint is unlikely to be penetrated by the phosphating solution.

For high-strength steels of a tensile strength greater than or equal to 1 000 MPa, a normally accelerated, copper-free process is used.

Normally, plants are constructed of steel and care should be taken to avoid the use of unsuitable materials in plant construction, e.g. copper or brass heating coils, which will contaminate the phosphating solution and adversely affect the quality of the phosphate coating.

The phosphate layer shall be uniform and matt in appearance, free from spots, uncoated areas, scratches, powdery and white residues. Differences in colour or shade in different areas, or from piece to piece, are not considered as causes for rejection.

Minor variations in the appearance of phosphate coatings caused, for example, by variations in the surface of the basis material or by contact with racks during phosphating, are common and are not normally indicative of important fluctuations in performance.

Methods of application and characteristics of phosphate conversion coatings are given in Annex A.

6.3 Processing after phosphating

Following phosphating, components are rinsed, stained, if specified, and dried as follows.