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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

Reference number
ISO 9717:2010(E)

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Contents

Page

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.

6.3.1 Rinsing

After phosphating, thorough rinsing with water is necessary in order to remove soluble salts which would otherwise tend to promote blistering under organic coatings. Parts, especially those with crevices, folds, or seams, as well as those treated with the accelerated phosphating process, shall be rinsed as follows:

a) After treatment by an accelerated process, items are rinsed thoroughly, first in flowing cold water and then in hot water at a minimum temperature of 75 °C, and finally washed in the hot dilute “chromic” solution described below. The accumulation of treatment chemicals in the rinse waters is avoided by maintaining a sufficient flow of cold water and by periodic renewal of hot water. The acidity of the hot water shall not be allowed to exceed that corresponding to 0,75 ml of 0,4 % (mass fraction) sodium hydroxide solution when tested by a suitably calibrated pH meter or by any other appropriate method.

Alternative processes to the chromic rinse such as those based on silane acid systems may be used provided that the efficiency and appropriate control of the process can be confirmed.

b) After treatment by an unaccelerated process, items are rinsed in flowing cold water, then in either hot water (65 °C minimum) or the hot dilute “chromic” solution (65 °C minimum).

The “chromic” wash solution normally consists of the range of 15 g to 50 g of chromic acid, or of alkali metal chromate or alkali metal dichromate, or of a mixture containing approximately equal parts of chromic and phosphoric acids, per 100 litres of water. A higher concentration than the maximum quoted above is not to be used on surfaces which will be in contact with, or in proximity to, hazardous substances. In other cases, higher concentrations of chromic acid, chromate or dichromate may be used up to a maximum of 500 g per 100 litres of water, where such concentrations will give overall beneficial results and where the effect of local enrichment or drainage will not be detrimental under organic-based systems.

WARNING — The repeated handling with bare hands of items that have been given a “chromic” wash may constitute a health hazard.

c) The wash waters, hot or cold, used for unaccelerated and accelerated processes shall not contain chloride, calculated as chloride ion, in excess of 0,5 g/l, when determined by the method described in Annex B.

6.3.2 Staining

Parts that are to be stained by water soluble pigment are dyed at this stage [see 4.1 j)]. Staining by spirit dye is dyed after drying.

6.3.3 Drying

Parts are dried after thorough rinsing. Forced drying is also used, especially for those with crevices, seams, etc.

6.3.4 Identification of type of phosphate conversion coating

Details of the procedures of identification of types of phosphate conversion coatings are described in Annex C.

6.4 Thickness of coating

The coating mass, rather than thickness, is measured in accordance with ISO 3892 and shall be determined on an item after treatment by phosphating, washing, and drying and before any heat treatment and sealing (see 5.4).

For quality assurance purposes, instruments which are available and which give a direct reading of coating mass per unit area may be used. It is, however, essential that they are calibrated against standard coatings having a coating mass per unit area of the same order as those being inspected.

In the case of heavy coatings, the amount of phosphate deposited is sometimes expressed in terms of coating thickness, particularly for quality-control purposes. The use of this alternative (and selection of a relevant test method) shall be agreed by the purchaser [see 4.1 e)].

If required, other properties of the coating, such as surface profile and crystal size, may also be specified [see 4.2 d)].

If proprietary processes are used, dimensional changes resulting from phosphating treatments are normally within a maximum increase of 8 µm/surface, depending on the particular process selected and a strict adherence to the process manufacturer's operating instructions. Deviation from the specified operating conditions of the phosphating solution can result in excessive attack of the item, with a possible decrease in dimensions and uneven coatings of inferior properties.

The dimensions of the finished item shall be those of the item after phosphating and before the application of any supplementary cover. Where phosphating is used to give anti-wear properties to sliding surfaces, the coating is consolidated during the running-in period, and consequently no machining allowance is normally made for phosphating.

6.5 Stress-relief heat treatment before cleaning and phosphating

Steel parts that have an ultimate tensile strength equal to or greater than 1 000 MPa and that contain tensile stresses caused by machining, grinding, straightening or cold-forming operations shall be given a stress-relief heat treatment, prior to cleaning and phosphating, and the procedures and classes for stress-relief heat treatment shall be in accordance with ISO 9587, unless otherwise specified by the purchaser.

When a heat treatment for stress relief prior to phosphating is specified, the time and temperature of the heat treatment process shall be included in the coating designation, as illustrated in 5.3.

Steels with oxide or scale have to be cleaned before application of the coatings. For high-strength steels (equal to or greater than 1 000 MPa), non-electrolytic alkaline and anodic alkaline cleaners, as well as mechanical cleaning procedures, are preferred to avoid the risk of producing hydrogen embrittlement during cleaning procedures.

6.6 Hydrogen-embrittlement-relief heat treatment after phosphating

Steel parts having an ultimate tensile strength equal to or greater than 1 000 MPa, as well as surface-hardened parts, shall receive hydrogen-embrittlement heat treatment according to the procedures and classes of ISO 9588, unless otherwise specified by the purchaser.

Any heat treatment for the relief of hydrogen embrittlement shall be carried out before application of any supplementary cover.

When a heat treatment for hydrogen-embrittlement relief after phosphating is specified, the time and temperature of the heat treatment process shall be included in the coating designation, as illustrated in 5.3.

The effectiveness of the hydrogen-embrittlement-relief treatment is determined by test methods described in ISO 10587[4] and ISO 15724, unless otherwise specified by the purchaser.

Embrittlement relief heat treatment at higher temperatures may cause colour changes and dehydration of phosphate coatings with consequent lowering of corrosion resistance.

6.7 Treatments for supplementary coatings

Where the items are required to be finished by varnish, lacquer, or organic coating systems, and an interval between the phosphating and the first stage of the treatment for supplementary coating is unavoidable, the phosphated items shall be kept under conditions which preclude contamination or condensation of moisture on the surface.

Immediately after rinsing, drying, and the embrittlement-relief heat treatment where applied, the phosphate coating is sealed by the application of a supplementary coating (see Table F.1). The type of coating required (e.g. oil, grease, organic coating, etc.) and, in the case of varnish, lacquer, or organic coatings, the mass or thickness of the coating, shall be as agreed by the purchaser [see 4.1 e) and 4.1 i)].

When heavily pigmented organic coatings are applied to a Class 1 or Class 2 phosphate coating, complete impregnation may not be achieved, leading to a serious lack of adhesion, particularly under dynamic conditions. When such phosphate films are given treatments for supplementary coating, consideration should be given to using a low-viscosity impregnating coat of clear lacquer compatible with the normal organic coating system, followed by the normal coating system.

6.8 Adhesion of phosphate conversion coatings

When the function of the phosphate conversion coating is corrosion resistance, an ancillary treatment is always required. Guidance for phosphate conversion coatings to ensure good adhesion of organic coatings, varnishes and related finishes, and to improve corrosion resistance of the basis metal, is given in Annex A.

If phosphated components are to be given organic coating systems, varnishes or similar coatings, they shall first be rinsed in clean tap water and then preferably in deionized water, so as to ensure that the surfaces are free from any water-soluble residues from previous processing, etc., which could give rise to blister formation in the applied film. It is equally important that the phosphated surface intended to have organic coatings shall be free of additional contamination, such as dust or finger prints.

6.9 Quality-evaluation test of phosphate conversion coatings

For applications suitable for intense local action rather than that produced by a continuous moving moisture film, the salt droplet test (see Annex D and Reference [1] in the Bibliography) shall be used, unless otherwise specified by the purchaser [see 4.1 j)].

For a test method to provide a means of controlling the continuity and quality of the coating by a continuous moving moisture film, use the neutral salt spray (Fog) test method (see Annex E and References [2] and [7] in the Bibliography), unless otherwise specified by the purchaser [see 4.1 j)].

In all cases, the minimum exposure time before the first appearance of the corrosion product of the substrate, mode of assessment and whether removal of supplementary coatings is needed prior to examination of tested specimens shall be specified by the purchaser [see 4.1 j)].

Depending on the composition of the material used for supplementary coating, considerable scatter of exposure times can occur before the first appearance of corrosion. Therefore, minimum exposure time shall always be specified for the supplementary coating.

Salt droplet and neutral salt spray (fog) tests provide means of controlling the continuity and quality of the coatings and are not corrosion tests on metals.

The duration and results of these tests may bear little relationship to the service life of the finished article and, therefore, the results obtained shall not be regarded as a direct guide to the resistance to exposure to an aggressive atmosphere of the tested coatings in all environments where these coatings may be used.

Additionally, performance of different materials in the test shall not be taken as a direct guide to the relative resistance to aggressive environments of these materials in service.

7 Sampling

The sampling method shall be selected from the procedures specified in ISO 2859 or ISO 4519, unless otherwise specified by the purchaser [see 4.1 k)]. The purchaser shall specify the acceptance levels.

Annex A
(informative)

Methods of application, characteristics and significance of phosphate conversion coatings

A.1 General

Phosphate conversion coatings are produced by treatment with solutions, the main constituents of which are the appropriate dihydrogen orthophosphates. All phosphate conversion coatings are more or less porous but can be sealed significantly by appropriate sealing and supplementary treatments.

A.2 Methods of application

Phosphating is usually carried out by immersing the component in a coating bath, with agitation of the solution if necessary, or by copious application or spraying of the component with the coating solution. Roller application may, however, be used in the case of galvanized or ungalvanized steel strip.

In the case of certain low alloy steels, e.g. chromium-molybdenum-vanadium and copper-silicon-molybdenum-vanadium steels, the best phosphate coatings are obtained using accelerated solutions. In the past, considerable difficulties were experienced in obtaining acceptable phosphate coatings on some of these types of steel particularly in the forged form, and it may be necessary to consider an alternative method of protection.

The free-acid concentration of the phosphate solution can markedly affect the quality of the phosphate coating of alloy steels, for example, nickel-chromium-molybdenum and maraging (nickel-cobalt-molybdenum-titanium)-type steels. It is expected that the pointage to the free-acid ratio be maintained at not less than 6 to 1 to obtain acceptable coatings and prevent unacceptable attack and/or pitting of the steel during the phosphate treatment. The free-acid level of the phosphate process solution, which tends to increase when the bath is maintained at the operating temperature but is not used, may be reduced by the treatment of clean steel scrap, steel wool, or finely divided iron powder. Addition of iron powder to the phosphate process solution may result in a vigorous reaction. The iron powder should be added in small quantities and the reaction allowed to subside after each addition, before further iron powder is added.

The phosphated components are then rinsed with water, dried and after-treated according to their intended end-use.

Sludge is formed as a normal by-product of the phosphating reaction. The sludge does not usually interfere with processing but forms of agitation which cause sludge to be deposited on the component should be avoided. Filtration of the bath prevents sludge deposition on the work pieces.

A.3 Characteristics of various types of phosphate conversion coatings

The usual appearance, crystallinity, colour and uniformity of phosphate coating of the principal types of phosphate conversion coatings, at magnification ×6, are given in Table A.1.

Table A.1 — Characteristics of phosphate conversion coatings

Main constituents of phosphating bath	Coating-type produced	Coating symbol	Usual appearance of coating	Coating mass per unit area, g/m^2			
				Fe	**Al**	**Zn**	**Cd**
$Zn(H_2PO_4)_2$	Zinc phosphate	Znph	Light grey to dark grey	1 to 30	0,3 to 10	1 to 20	
$Zn(H_2PO_4)_2$ $Ca(H_2PO_4)_2$	Zinc-calcium phosphate	ZnCaph	Light grey to dark grey	1 to 30	—	1 to 10	—
$Mn(H_2PO_4)_2$	Manganese phosphate	Mnph	Dark grey to black	1 to 30	—	—	—
$Me(I)(H_2PO_4)_2$[a]	Phosphate of treated metal (plus oxides of iron in the case of ferrous materials)	Feph	Amorphous coatings of about 0,1 g/m^2 to 1 g/m^2: iridescent, e.g. yellowish to bluish grey Coatings over about 1 g/m^2: grey	0,2 to 1,5	$< 0,5$	0,2 to 2	—

[a] Me(I) denotes a cation of alkali metal or $(NH_4)^+$.

A.4 Significance and adhesion of phosphate conversion coatings

A.4.1 Cold forming

Zinc phosphate conversion coatings are preferred to assist cold forming. Guidance on the coating mass per unit area for various end-uses is given in Table A.2.

Zinc phosphate coatings shall be neutralized, after rinsing, by treatment with a weakly alkaline aqueous solution.

Soap lubrication can be performed by dipping in a suitable soap solution (tube drawing, cold heading and cold extrusion, deep drawing) or with dry soap powder (wire drawing). In the case of dip application, the soap will react with the zinc phosphate layer, giving rise to zinc stearate on top of the zinc phosphate coating. Prior to dry-soap lubrication, wires may be immersed in a strong solution of borax or lime which acts as a lubricant carrier.

Table A.2 — Zinc-phosphate-conversion-coating mass per unit area to assist cold forming

End-use	Coating mass per unit area g/m^2
Drawing of steel wire	5 to 15
Drawing of welded steel tubes	3 to 10
Drawing of steel precision tubes	4 to 10
Cold heading and cold extrusion	5 to 20
Deep drawing without wall-thickness reduction	2 to 5
Deep drawing with wall-thickness reduction	5 to 15

A.4.2 Sliding action

Manganese phosphate coatings are generally preferred to facilitate sliding action. Table A.3 recommends coating masses per unit area for various end-uses.

The factors listed below should also be taken into consideration.

a) Although manganese phosphate coatings are generally preferred, other types of coatings, for example zinc phosphate, are also suitable, particularly at lower coating levels. The type of coating selected will depend on the stresses in the coating involved for the intended end-use.

b) The coating level used depends on the dimensional tolerances of the coated components in the assembled unit.

Such coatings are normally used in conjunction with a suitable lubricant.

Table A.3 — Manganese-phosphate-conversion-coating mass per unit area to assist sliding action

End-use	Coating mass per unit area g/m^2	Notes
Components with small clearances, e.g. pistons of refrigerator compressors	3 to 5	Coating of mainly manganese phosphate (no iron in bath)
Components with large clearances, e.g. gears, crown wheels and pinions of gear units and differentials	5 to 20	Coating of manganese iron phosphates (iron present in bath)

A.4.3 Adhesion of phosphate conversion coatings to improve corrosion resistance

Table A.4 reviews the significance of coating mass per unit area of phosphate conversion coatings to ensure good adhesion of organic coating systems, varnishes and related coatings to improve corrosion resistance.

Table A.4 — Adhesion of phosphate conversion coatings to improve corrosion resistance

Basis metal	Phosphate coating		Supplementary covers	Typical applications and end-uses
	Preferred type of treatment	Coating mass per unit area g/m^2		
Ferrous materials	Znph Mnph Feph	> 5, but preferably > 10	Protective oils or waxes, as required, after dyeing of the coating	Corrosion protection in transit during long-term storage.
	ZnCaph	> 5		Long-term protection in dry environment (no condensation). Temporary protection outdoors with overhead cover.
Ferrous materials, zinc, aluminium, cadmium	Znph ZnCaph	1 to 10	Organic coatings, varnishes and related coatings	Motor-vehicle bodies, refrigerator and washing-machine cabinets
Zinc	Znph	1,5 to 4,5		Motor-vehicle bodies, sheets and strips which are formed after application of organic coatings and, in particular, where flexing of an organic after-treatment is involved.
Ferrous materials	Feph Znph	0,2 to 1,5 1,5 to 4,5	None	Temporary protection in dry environment (no condensation). Short-term, in-plant storage of machine components (< 24 h).

Annex B
(informative)

Determination of rinse-water contamination

B.1 Test for carry-over of treatment solution

B.1.1 Solutions required

a) Sodium hydroxide: 0,4 % (mass fraction) sodium hydroxide solution.

b) Phenolphthalein indicator: dissolve 0,5 g of phenolphthalein in 50 ml of ethanol and dilute to 100 ml with distilled water.

B.1.2 Procedure

Stir the contents of the rinsing bath; withdraw about 100 ml of the solution and cool to room temperature. Measure 50 ml of the cooled solution into a 300 ml conical flask or other suitable glass container, add a few drops of phenolphthalein indicator and titrate with the 0,4 % (mass fraction) sodium hydroxide solution until a permanent faint-pink colouration is obtained. The titration value shall not exceed 0,75 ml.

B.2 Test for chloride in rinsing water

B.2.1 The following test is designed to determine when the rinsing water has a chloride concentration greater than 0,5 g/l. Alternative tests may be used, provided that their efficiency and reproducibility can be demonstrated.

B.2.2 Solutions required

a) Standard sodium chloride: solution containing 0,5 g/l of chloride ion. Dissolve 0,825 g of sodium chloride in distilled water and dilute to 1 litre.

b) Acidic silver nitrate solution: 1 % (mass fraction) silver nitrate in 20 % (volume fraction) of nitric acid. Dissolve 10 g of silver nitrate in a little distilled water, add 200 ml of nitric acid (ρ = 1,42 g/cm^3) and dilute to 1 litre.

B.2.3 Apparatus

Use the following apparatus.

a) Two 100 ml tubes.

b) One 5 ml measuring cylinder.

c) Two glass rods approximately 200 mm long and 5 mm in diameter for stirring the solutions.

B.2.4 Procedure

a) Mark the two 100 ml tubes “S” and “T” respectively, in any convenient way.

b) To the tube marked “S”, add 5 ml of the standard sodium chloride solution and dilute to the 100 ml mark with water.

c) To the tube marked “T”, add 5 ml of the rinsing water and dilute to the 100 ml mark with water.

If the rinsing water contains suspended matter, it shall be filtered through a Whatman No. 1 paper[1] (or equivalent) before using it for the test.

d) To each tube, add 5 ml of the silver nitrate solution. Stir the contents of each tube with the glass rods, and set aside for 1 min.

e) Compare the turbidities of the two solutions. If the solution in tube “T” is more turbid than that in tube “S” then the rinse has a chloride concentration greater than 0,5 g/l.

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[1] Whatman No. 1 paper is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

Annex C
(informative)

Identification of phosphate conversion coating

C.1 General

This annex describes methods for identifying the type of phosphate conversion coating. They are applicable to coatings containing phosphorus, iron, manganese, zinc and calcium. They are not applicable to the detection of iron or zinc if these metals are present in the basis material.

C.2 Method 1

C.2.1 Principle

Removal of the phosphate conversion coating from a test specimen by treatment with sodium hydroxide solution. Detection of the elements present in the solution by any suitable instrumental analytical technique, e.g. atomic absorption spectrometry.

C.2.2 Reagents

Use sodium hydroxide solution, 50 g/l.

During the analysis, use only reagents of recognized analytical grade and distilled water, or water of equivalent purity.

C.2.3 Apparatus

Use ordinary laboratory apparatus and any appropriate instrument suitably equipped for the detection of phosphorus, manganese, zinc and calcium, e.g. an atomic absorption spectrometer for metals.

C.2.4 Test specimen

Use a test specimen having a total coated surface area of approximately 100 cm^2.

C.2.5 Procedure

Immerse the test specimen (C.2.4) in 100 ml of the sodium hydroxide solution (C.2.2) maintained at a temperature of 80 °C to 90 °C, until the coating has been removed or has at least undergone obvious attack. If necessary, remove the coating by rubbing with a rubber squeegee. Using the appropriate analytical instrument (C.2.3), detect which of the elements, phosphorus, manganese, zinc and calcium, are present in the test solution.

C.3 Method 2

As an alternative to Method 1 (C.2), a scanning electron microscope equipped for energy dispersive spectroscopy (EDS) may be used for the direct detection of metal elements in the coating.

C.4 Interpretation of results

Taking account of the elements detected, identify the types of coating in accordance with Table C.1.

Table C.1 — Identification of type of phosphate conversion coating

Elements detected	Coating type
Phosphorus	Phosphate
Manganese	Mnph
Zinc (no calcium)	Znph
Zinc and calcium	ZnCaph

For ferrous basis materials, the absence of manganese or zinc in the conversion coating indicates almost certainly that the coating consists of iron(II) phosphate (Feph).

NOTE The presence of a satisfactory coating can be deduced by

a) rubbing the treated surface with fine steel wool; untreated steel shows a bright burnished appearance, or

b) scraping the surface once with a blunt knife; coated surfaces show a whitish streak, or

c) scraping a small area with a sharp knife; coated surfaces yield a light grey powder.

Test methods b) and c) may not give very marked indications of the presence of a Class III or IV coating (see 5.4).

Annex D
(normative)

Salt droplet test

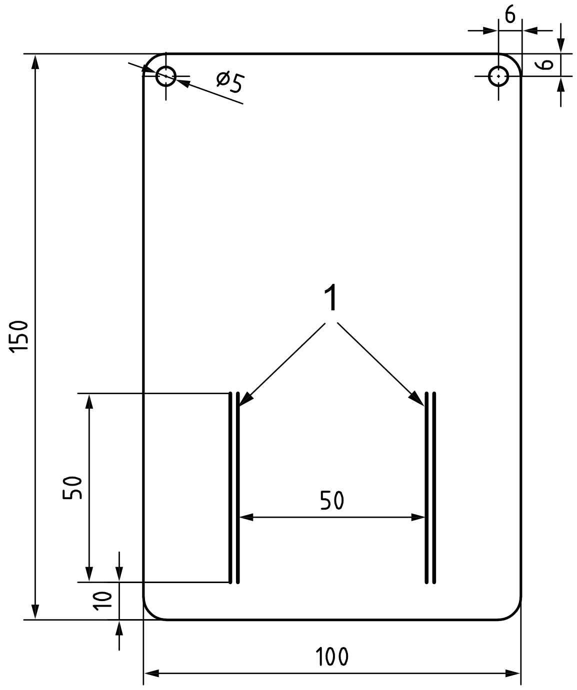
D.1 Test method

D.1.1 For applications suitable for intense local action rather than that produced by a continuous moving moisture film (see 6.9) the salt droplet test shall be used, unless otherwise specified by the user or purchaser [see 4.1 k)].

Where it is not possible to carry out the test on the actual item, special test panels can be used. If special test specimens are used to represent the coated articles in a test, the specimens shall be of the same nature, same surface condition, and same material and in the same metallurgical condition as the article it represents, and shall be placed in the same production lot of, and be processed along with, the coated article they represent.

D.1.2 The test panels shall measure 150 mm × 100 mm (see Figure D.1). Two holes of diameter 5 mm and lightly countersunk on each side shall be drilled at 6 mm from each edge near adjacent corners at the ends of a 100 mm side, to enable the specimens to be suspended vertically during the test. All cut edges and corners shall be lightly rounded off and smoothed.

Dimensions in millimetres



Key

1 scratches

Figure D.1 — Test panel

D.2 Test solution

The reagents used shall be of analytical quality and deionized water or water of equivalent purity shall be used. The composition of the solution shall be as follows:

Sodium chloride, NaCl	26,5 g
Magnesium chloride, $MgCl_2$	2,4 g
Magnesium sulfate, $MgSO_4$	3,3 g
Potassium chloride, KCl	0,73 g
Sodium hydrogen carbonate, $NaHCO_3$	0,20 g
Sodium bromide, NaBr	0,28
Calcium chloride, $CaCl_2$ (to be added last)	1,1 g
Deionized water or water of equivalent purity to complete to	1 000 ml
The volume of solution shall be between 1 litre and 1,5 litres per	24 h

D.3 Test procedure

D.3.1 The items or panels (see Figure D.1) shall be suspended vertically, by means of hooks of glass or other non-corrodible insulators passing through the holes in two corners, over large dishes partly filled with water and the whole assembly shall be placed within a non-metallic container. The distance between the surface of the water and the lower edge of the items or panels shall be 50 mm to 75 mm and the items or panels shall be one behind the other and all facing the same way. They shall be at a convenient distance (not more than 25 mm) apart to prevent touching when being moved and a dummy item or panel shall be placed at each end of each row.

D.3.2 Once daily, for five days a week, all the items or panels, including the dummies, shall be removed temporarily from their position on the rack and sprayed individually on both sides with the salt solution, using a hand-operated atomizer. The aim shall be to cover the surface with discrete droplets, by giving a number of puffs with the nozzle at a suitable distance (150 mm to 300 mm) from the specimens, the spray being directed, in turn, at different parts of the surface. The spraying shall not be so heavy that the droplets coalesce; in order to avoid this it may prove necessary on occasions, where the surface is still wet from the previous spraying, to refrain from applying the full weight of spray specified below. It is essential to keep the items or panels covered with droplets. Immediately after spraying, the items or panels shall be returned to their positions on the rack and the whole collection shall be covered with a boxlike cover of non-metallic or other suitable material, to prevent the drying of the droplets. Any cover shall be suitably protected on the inside to prevent contamination of the item or panel.

The spray may be absorbed by non-glossy paint films; if so, it may be impossible to check that the desired distribution of droplets has been obtained. The intensity of spraying required is shown in ISO 4536[1]. This difficulty can be overcome by spraying dummy items or panels of a material (on which the droplets can be seen) in such a way as to produce the standard distribution of droplets and then spraying the test items or panels in exactly the same way. Steel or glass coated with a hard glossy finish is suitable for this purpose.

D.3.3 The intensity of spraying can be checked by weighing the solution deposited on both sides of a blank 150 mm × 100 mm freshly degreased bare steel panel. The mass should lie between 0,5 g and 1,0 g. The temperature throughout the test shall be between 5 °C and 20 °C and the test assembly shall be protected against external sources of heat tending to produce local variations in temperature. The spray solution shall be a synthetic sea water prepared as in D.2.

D.3.4 Any atomizing nozzle capable of producing fine droplets (not a mist) may be used for the test.

D.3.5 There shall be two scratches, each 50 mm long, parallel and at a distance of 50 mm apart (see Figure D.1), unless the size or shape of the article does not permit this, in which case alternative arrangements shall be permissible. In all cases, the scratch shall penetrate the paint film to the substrate. The article shall be placed for testing in such a way that the scratches run vertically, thus eliminating any tendency for rust stains to spread laterally from the scratches under the action of gravity. No test shall commence within 24 h of the completion of the coating process and, if the items or panels are scratched before testing, a further interval of 24 h shall elapse between the infliction of the scratches and the first spraying.

D.4 Loss of mass of control specimens

D.4.1 At the end of the agreed test period, the loss in mass of the control items or panels shall be determined after derusting: The average loss in mass shall be not less than the values given in Table D.1.

Table D.1 — Loss of mass of control specimens

Test duration days	**Average loss of mass per** 150 mm × 100 mm **panel** g
5	1,0
7	1,4
14	2,4
21	3,3
28	4,1

D.4.2 The items or panels shall be derusted by means of Clarke's solution, which consists of 20 g of antimonious oxide and 50 g of stannous chloride dissolved in 1 litre of hydrochloric acid ($\rho = 1{,}16$ g/cm^3). The solution shall be used cold and the items or panels kept moving in it until derusting is complete. The items or panels shall then be washed in running water, dried and weighed. The figure to be recorded is the difference between the mass of the freshly degreased items or panels and the corresponding mass after derusting.

It is essential that the items or panels be kept moving in the solution, so as to ensure rapid reduction by the reagent of ferric chloride produced by solution of the rust; otherwise, attack on the steel itself may occur.

D.5 Assessment

D.5.1 At the end of the agreed test period, the items or panels shall be rinsed in cold water, visually inspected, and allowed to dry at room temperature. Immediately the items or panels are dry, a final inspection shall be made.

D.5.2 The same procedure shall be followed if failure is suspected at an earlier stage, and if inspection confirms failure, the items or panels concerned may be removed from test.

Annex E
(normative)

Determination of resistance of phosphate conversion coating to neutral salt spray (fog) test

E.1 Determination of resistance of phosphate conversion coatings to neutral salt spray (fog) test without supplementary treatments or organic coatings

For test methods to provide means of controlling the continuity and quality of the phosphate conversion coatings by a continuous moving moisture film, use the neutral salt spray (fog) test method (see 6.9), unless otherwise specified by the user or purchaser [see 4.1 k)].

The phosphated components or test panels to be examined are placed in the test cabinet, exposed to the specified environment during the periods stated in Table E.1, and removed for evaluation.

Table E.1 gives the minimum exposure times, in hours, which have to be attained by phosphated ferrous components without any supplementary treatments before the first evidence of corrosion appears.

Table E.1 — Minimum exposure time, in hours, before first appearance of red rust of phosphated ferrous components without any treatments for supplementary cover

Type of phosphate coating	Zinc phosphate coatings	Manganese phosphate coatings	Iron(II) phosphate coatings
Classification code	Znph	Mnph	Feph
Coating mass per unit area, g/m^2	> 10	> 15	> 15
Minimum exposure time, h	2	1,5	1,5

E.2 Determination of resistance of phosphate conversion coatings, which have been subsequently treated with corrosion-preventing oils, to neutral salt spray (fog) test

E.2.1 Principle

Phosphated test pieces are treated with an appropriate corrosion-protecting medium under specified conditions and these treated test pieces are subjected to the neutral salt spray (fog) test (see 6.9). The test may be used either to evaluate a specific corrosion-protecting system or for comparative testing of a range of phosphate conversion coatings in conjunction with a specific corrosion-preventing oil.

E.2.2 Supplementary treatment

E.2.2.1 Specific treatment

Immerse the phosphated component or test panel in the corrosion-protecting medium under examination, carefully following the appropriate instructions and ensuring that the liquid, semi-solid or waxy film which is thereby formed on the phosphated surface is free from air bubbles and discontinuities.

Before testing, suspend the test pieces for at least 24 h at a temperature of 23 °C ± 5 °C and a relative humidity not exceeding 65 %, in a dust and draught-free atmosphere, so as to allow the surplus of oil to drip off and the solvents to evaporate.

E.2.2.2 Comparative testing

Suspend the phosphated test pieces (previously dried at a temperature of between 100 °C and 120 °C and cooled to room temperature) from suitable plastics hooks or plastics-coated steel hooks and immerse them vertically in the selected oil for 1 min at a temperature of 25 °C ± 2 °C. During this time, move the test panels gently to and fro and then withdraw them gradually from the oil in about 30 s. The oil film so formed on the phosphated surface shall be free from air bubbles and discontinuities.

Suspend the test panels for at least 24 h at a temperature of 23 °C ± 5 °C and a relative humidity not exceeding 65 %, in a dust and draught-free atmosphere, prior to testing.

E.2.3 Procedure

Subject the oil-treated components or test panels (see E.2.2) to the neutral salt spray (fog) test (see 6.9).

This test determines the exposure times attainable by a specific corrosion-protecting system before the first evidence of corrosion of metal becomes visible.

For this purpose, take test pieces from the test chamber at predetermined intervals and examine them visually (with corrected vision if necessary) for evidence of corrosion. Whether or not the after-treatment film (or layer) is to be removed before visually examining the components or test panels is subject to agreement [see 4.2 d)].

For a specific phosphate coating, considerable scatter occurs in the exposure times up to the first occurrence of corrosion, depending on the composition of the supplementary treatment medium and the coating level. Therefore, minimum exposure times for supplementary-treated phosphate conversion coatings shall always be referred to a specific product from the category of corrosion-preventing oils, greases and waxes, of known coating level, in grams per square metre, as determined by mass difference.

Annex F
(normative)

Treatments for supplementary coating

Table F.1 shows the symbols that are used when specifying a supplementary coating.

Table F.1 — Types of supplementary coating

Symbol	Type of treatment
T1	Application of varnishes or organic coatings Application of inorganic or non-film-forming organic sealants
T2	Application of inorganic or organic sealants
T3	Dyeing
T4	Application of grease or oil or other lubricants
T5	Application of wax
T6	Application of soap

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