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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

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

This third edition cancels and replaces the second edition (ISO 9717:2010), which has been technically revised. The following changes have been made:

— the Scope has been clarified;

— the Normative references have been updated;

— the Terms and definitions have been replaced by a reference to ISO 2080;

— Clause 4 has been revised;

— the terms in Clause 5 have been revised;

— requirements for the phosphate layer have been revised;

— statements on corrosion resistance have been shifted to a new Annex A;

— Annex B on salt spray testing has been revised;

— Annex C to the properties of the phosphate layers has been 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

— impart corrosion resistance,

— improve adhesion to paints and other organic finishes,

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

— 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 and zinc, and differ in coating mass per unit area and apparent density, depending on

— the construction material and surface condition of the components,

— previous mechanical and chemical treatment of the components, and

— processing conditions for phosphating.

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

Metallic and other inorganic coatings — Phosphate conversion coating of metals

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1 Scope

This document specifies a process for the confirmation of requirements for phosphate coatings which are usually destined for application on ferrous materials, zinc, cadmium and their alloys (see Annex B).

2 Normative references

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

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

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 9227, *Corrosion tests in artificial atmospheres — Salt spray tests*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2080 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Information to be supplied by the purchaser to the processor

The following information shall be provided by the purchaser.

a) A description of the coating according to this document, i.e. ISO 9717 (see 5.2).

b) In cases of phosphating steel parts with tensile strength ≥ 1 000 MPa, possibly also locally restricted, e.g. for case-hardened or cold-formed structures or in weld seam areas, the safety against brittle fracture (hydrogen embrittlement) is of primary importance. The phosphatising process shall be carried out in such a manner that any damage caused by hydrogen-induced brittleness is excluded. Technical measures to minimize the risk of hydrogen-induced brittleness shall be defined by the user and provided by the supplier/customer. Heat treatment in accordance

with any recommendations cannot guarantee full freedom from hydrogen embrittlement. The performance of the heat treatment shall be demonstrated by the supplier.

c) The sampling procedure, the acceptable quality limit or any other requirements and tests that deviate from ISO 4519.

d) The surface treatment or phosphating.

e) The appearance of the surface.

f) The corrosion resistance.

g) The quality, appearance and condition of basis metal, if any of these properties affect the usability and/or appearance of coatings.

h) Where applicable, technical measures to minimize the risk of hydrogen induced brittleness as defined by the user.

5 Coating types and their importance

5.1 Coating types

The conversion coating shall be declared according to Table 1.

Table 1 — Declaration of conversion coatings

Elements detected	Coating type
Iron (II)	Feph
Manganese	Mnph
Zinc (no Calcium)	Znph
Zinc and Calcium	ZnCaph

There are other coating types of zinc phosphate that are altered because of the build-in of iron and/or nickel and/or manganese. This altered metal is usually present as a double salt, such as $Zn_2ME(PO_4)_2$ x 4 H_2O, whereby ME can be Fe(II), Ni or Mn. Zn will continue to be the main metal element of those coatings, which do not have separate markings in order to avoid confusion. It should also be noted that metal taken from the basis material is included in the conversion coating.

Information for coating characteristics can be found in Annex B and the identification methods can be found in Annex C.

5.2 Designation of conversion coating

The designation of the conversion coating shall consist of the following information:

a) number of this document, i.e. ISO 9717, and the designation “phosphate coating”;

b) followed by a hyphen;

c) designation of basis material: by means of the material chemical symbol (or the symbol of the primary alloy components);

d) followed by a solidus(/);

e) a symbol describing the type of coating;

f) followed by a solidus (/);

g) a symbol, which indicates the function of the conversion coating as follows:

   1) r = adhesion promoter and/or corrosion protection;

   2) z = simplification of cold forming;

   3) g = reduction of friction;

   4) e = electrical insulation;

h) followed by a solidus (/);

i) a number, which indicates the surface-related mass per square metre with a measurement uncertainty of ±30 %.

If the phosphate coating receives a supplementary treatment, the following information shall be added to the designation:

j) solidus (/)

k) a symbol, which defines the supplementary treatment (see Table 2).

Repeat this process if necessary.

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 is either not required or has been omitted (see ISO 27830).

6 Requirements

6.1 Appearance

Zinc phosphate, zinc calcium phosphate and manganese phosphate coatings shall evenly cover the metal surface and shall not show any white stains, corrosion products or fingerprints.

NOTE Slight fluctuations in the appearance of phosphate coatings because of contact with frames, properties of the base material or through minor contact inside the drum do not constitute any reason for claim.

6.2 Coating mass per unit area

The coating mass is measured in accordance with the procedures specified in ISO 3892.

6.3 Post treatments

Untreated phosphate coatings do not provide a corrosion protection. A suitable after-treatment can effect a temporary protection. Table 2 shows the symbols when post-treatments are required.

Table 2 — Symbols for post after-treatments

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

The corrosion resistance of the after-treated components shall be verified in accordance with the corrosion test specified by the customer. If there is no test method prescribed, the components shall be tested according to the neutral salt spray (NSS) test described in ISO 9227 NSS test and Annex A. The test duration shall be agreed between the contractual parties. The minimum testing periods specified by the customer shall be reached before the first appearance of corrosion effect.

A coating of zincphosphate type Znph, which has been applied on a ferrous material to prevent corrosion, with an area related mass of 3 g/m^2 ± 0,9 g/m^2 and an after-treatment with a sealing (T2) and a painting (T1), has the following designation:

Phosphate coating ISO 9717 — Fe/Znph/r/3/T2/T1

6.4 Correlation of coating thickness and area-related mass

The thickness and the related mass in gram per square metre can be measured according to the following methods:

— magnetic inductive, according to ISO 2178;

— dissolving method, according to ISO 2177;

— x-ray according to ISO 3497.

7 Heat treatment

Heat treatment after the phosphating is not recommended, because the phosphate layer will be damaged at a temperature higher than 120 °C.

Annex A
(normative)

Determination of phosphate conversion coating resistance to neutral salt spray test

A.1 Determination of corrosion resistance

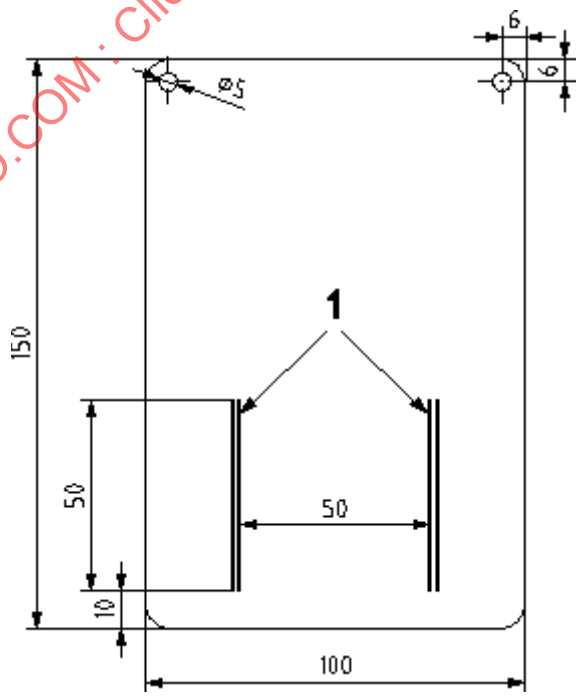
A neutral salt spray (NSS) test in accordance with ISO 9227 NSS is the standard test for determining corrosion resistance. If required, other test methods may be agreed between the contracting parties. The test should be performed with original components.

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 they represent, and shall be placed in the same production lot of, and be processed along with, the coated article they represent. Original components or test plates can be used either for testing the coating systems.

The salt spray test provides a means of controlling for the quality and continuity of coating.

The test panels shall measure 150 mm × 100 mm (see Figure A.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 A.1 — Test panel

A.2 Determination of resistance of phosphate conversion coatings to neutral salt spray test without supplementary treatments or organic coatings

Use the neutral salt spray test method (see ISO 9227) to provide a means of controlling the quality and continuity of the phosphate conversion coatings by a continuous moving moisture film. The test duration shall be specified by the user or purchaser.

A.3 Determination of resistance of phosphate conversion coatings, which have been subsequently treated with corrosion-preventing mediums, to neutral salt spray test

A.3.1 Principle

Phosphated test pieces are treated with an appropriate corrosion-protecting medium (e.g. oil) under specified conditions and these treated test pieces are subjected to the neutral salt spray test (see ISO 9227). 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 medium.

A.3.2 Supplementary treatment

A.3.2.1 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 forms 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.

A.3.2.2 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 plastic hooks or plastic-coated steel hooks and immerse them vertically in the 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 that forms 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.

A.3.3 Procedure

Subject the oil-treated components or test panels (see A.3.2) to the neutral salt spray test (see ISO 9227).

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.

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 B
(informative)

General information

B.1 General

Phosphate conversion coatings are produced by treatment with solutions, the main constituents of which are specified in Table B.1. These coatings are usually applied on ferrous material, aluminium, zinc and cadmium. They differ in the surface related mass and the apparent density depending on the following:

a) the raw material and surface quality of the component;

b) the mechanical and chemical pre-treatment of the component;

c) the working conditions of phosphating.

Phosphate coatings are more or less porous, but they can be compacted with an appropriate retreatment

Table B.1 — Characteristics of phosphate 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 $(NH_4)^+$.

B.2 Surface preparations

Components need to be removed from scale, grease, oil and corrosion products by using a suitable pretreatment.

Phosphating is usually characterized by dipping the components (if necessary in moving fluids), flooding or spraying with a phosphating solution. In cases of handling with tape material, it is possible to use the roll application process.

B.3 Application of phosphate coatings

B.3.1 Cold forming

Zinc phosphate conversion coatings are preferred to assist with cold forming. Guidance on the coating mass per unit area for various intended use is given in Table B.2.

The deformation rate is achieved by using an adequate lubricant.

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

Intended 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	2 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

B.3.2 Sliding action

Manganese phosphate coatings are generally preferred to facilitate sliding action. Table B.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 B.3 — Manganese-phosphate-conversion-coating mass per unit area to influence tribological properties

Intended use	Coating mass per unit area g/m^2	Notes
Components with small clearances, e.g. pistons of refrigerator compressors	2 to 5	Coating of mainly manganese phosphate
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)

B.3.3 Application of phosphate conversion coatings to improve corrosion resistance

Table B.4 reviews the significance of coating mass per unit area of phosphate conversion coatings regarding their application.

Table B.4 — Use 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 and/or storage.
	ZnCaph	> 5		Corrosion 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 that 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).