## INTERNATIONAL STANDARD

ISO 19598

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# Metallic coatings — Electroplated coatings of zinc and zinc alloys on iron or steel with supplementary Cr(VI)-free treatment

Revêtements métalliques — Revêtements électrolytiques de zinc et d'alliages de zinc sur du fer ou de l'acier avec traitement supplémentaire sans Cr(VI)





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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.

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 <a href="www.iso.org/directives">www.iso.org/directives</a>).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: <a href="www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

The committee responsible for this document is ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 3, *Electrodeposited coatings and related finishes*.

### Introduction

The chromium(VI) free systems differ in 2 points from the chromium(VI)-containing systems:

- a) there is no self-healing of the system;
- b) higher temperature resistance (> 150 °C), the limit for chromium(VI) containing systems, is  $\leq$  70 °C.

# Metallic coatings — Electroplated coatings of zinc and zinc alloys on iron or steel with supplementary Cr(VI)-free treatment

#### 1 Scope

This International Standard applies to electrodeposited zinc and zinc-alloy coatings on iron and steel with Cr(VI)-free passivation. The zinc-alloy coatings contain nickel or iron as alloying elements (referred to as zinc/nickel and zinc/iron coatings, respectively).

The main purpose of the coatings or coating systems is protecting iron and steel components against corrosion.

This International Standard specifies

- the designations to be used for the above coating systems,
- the minimum corrosion resistance to be achieved in specified test procedures, and
- the minimum coating thicknesses required.

#### 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 3497, Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods

ISO 3613:2010, Metallic and other inorganic coatings — Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys — Test methods

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

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 27830:2008, Metallic and other inorganic coatings — Guidelines for specifying metallic and inorganic coatings

#### 3 Terms and definitions

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

#### 4 Designation

#### 4.1 Electrodeposited coatings

The electrodeposited coatings shall be composed of zinc or zinc-alloys as listed in <u>Table 1</u>.

Table 1 — Designation of electrodeposited coatings

Symbol	Definition
Zn	Zinc coating without alloying element
ZnFe	Zinc-alloy containing a mass fraction of 0,3 % to 1,0 % iron
ZnNi	Zinc-alloy containing a mass fraction of 12 % to 16 % nickel

#### 4.2 Passivation

Passivation is the production of conversion coatings by treating coatings with suitable Cr(VI)-free solutions in order to improve their corrosion resistance. The conversion coating may optionally be dyed.

Since the Cr(VI)-free passivations are new coating systems, the new nomenclature is listed in <u>Table 2</u>.

Table 2 — Passivations

Type of passivation	Code	Colouration, surface appearance <sup>a</sup>	Notes
Transparent	An	Colourless to coloured and iridescent	Often referred to as "thin-layer passivation"
Iridescent	Cn	Coloured and iridescent	Often referred to as "thick-layer passivation"
Black	Fn	Black	
a Tints are permissible.			

#### 4.3 Post treatment

Application of a sealant or top coats increases corrosion resistance.

Sealant layers normally show a thickness of up to 2  $\mu$ m and are composed of Cr(VI)-free organic and/or inorganic compounds. Coatings that can be removed with cold cleaning agents, such as those with an oil, grease or wax base, are not considered as sealants in the context of this International Standard.

Top coats are normally thicker than 2  $\mu$ m and are Cr(VI)-free organic thin coatings which may need elevated temperature curing. Particularly for coatings with black passivation, a postdip can be used for increasing corrosion resistance and improving colour depth.

The impact of the post treatments mentioned to component properties such as contact resistance, weldability, compatibility with fuels, performance in bonded joints, etc., when in service shall be assessed on a case by case basis.

Since the range of surface modifications is very large, the use of a post treatment mentioned and the type shall be agreed on in the case of surface coatings that are subject to particular requirements.

NOTE The post treatment mentioned normally remove the interference colours produced by passivation.

Table 3	— Post	treatmen	ıt
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Symbol	Meaning
Tx	Sealant may or may not be present <sup>a</sup>
Т0	No sealant
T2nL	Sealant without integrated lubricant <sup>b</sup>
T2yL	Sealant with integrated lubricant <sup>b</sup>
T4	Subsequently added lubricant
T7nL	Top coat without integral lubricant <sup>b</sup>
T7yL	Top coat with integral lubricant <sup>b</sup>

NOTE T4 is not considered.

#### 4.4 Significant (functional) surfaces

Components with complex shapes, particularly those having cavities, may not be capable of meeting the corrosion resistance and minimum thickness criteria required in the accelerated corrosion tests at all the points on the electroplated surface. In such cases, the significant surfaces for the coating system shall be indicated on the drawing by a dot-and-dash line.

If the purchaser does not specify a significant surface, the definition given in ISO 27830:2008, Clause 6 shall apply.

#### 4.5 Examples of designations

Designation of a zinc/nickel alloy coating on a steel (Fe) component having a minimum local coating thickness of  $8 \mu m$  (8) and iridescent passivation (Cn):

#### Electroplated coating ISO 19598 - Fe//ZnNi8//Cn//T0

Designation of a zinc/iron alloy coating on a steel (Fe) component having a minimum local coating thickness of 8  $\mu$ m (8) with black passivation (Fn) and sealant (T2):

#### Electroplated coating ISO 19598 - Fe//ZnFe8//Fn//T2

Designation of a zinc coating on a steel (Fe) component having a minimum local coating thickness of  $12 \mu m$  (12) and black passivation (Fn); application of a subsequent sealant is left to the choice of the coater:

#### Electroplated coating ISO 19598 - Fe//Zn12//Fn//Tx

#### 5 Information to be supplied by the purchaser to the electroplater

The minimum information to be provided by the purchaser to the electroplater shall comprise the following:

- a) the tensile strength of the component (to enable the requirements in 7.4 to be taken into account);
- component information (basis material, production method, heat treatment);
- c) information on significant surfaces as required by 4.4;
- d) designation of the coating to be applied (see 4.5).

If desired, further requirements, such as appearance, friction properties and media resistance, may be imposed on the properties of the coatings to be applied and the testing extended accordingly.

a Left to the choice of the coater.

b Optional designation of nL or yL, when required.

#### ISO 19598:2016(E)

If necessary, additional information relating to requirements or restrictions to be imposed on the coating process shall be provided.

#### 6 Basis materials

The application of coatings complying with this International Standard to low-alloyed steels is state of the art. In the case of other iron-based materials, such as cast iron or sintered materials, materials with a substantial content of passivatable alloying elements or materials with special strength properties, it may be necessary to specially adapt the treatment processes (pre-treatment, coating, supplementary treatment) and possibly to take additional measures in order to fulfil the requirements of this International Standard. For this reason, the electroplater shall have detailed information about the composition, properties and production of the components to be coated.

If steel components having tensile strengths of 1 000 N/mm<sup>2</sup> and above are to be coated, steps will have to be taken even at the early stages of production (e.g. when choosing the material, the hardening process and the assembly procedure) to eliminate with a high degree of reliability the possibility of damage resulting from internal hydrogen embrittlement.

The components to be coated shall not exhibit any material, processing or surface defects that may affect the corrosion protection and/or appearance of the coatings adversely or in an unpredicted way.

It shall be possible to remove any impurities (e.g. corrosion products or scale, oil, grease, dirt, etc.) from the surface of the components without leaving any residue by the standard cleaning and pre-treatment procedures.

It may be necessary to draw up an agreement on surface quality.

#### 7 Coating and processing

#### 7.1 Surface preparation and deposition of zinc or zinc-alloy coating

To ensure that the process is reliable and reproducible, the entire pre-treatment and coating cycle, together with the associated physical data, e.g. treatment times and temperatures, and all the processing chemicals, shall be recorded, documented and, if necessary, optimized. The individual process control limits shall be defined along with the frequency of monitoring and the analytical procedures. The actions these require shall be described and recorded by the electroplater.

A typical process cycle would be as set out below:

- a) alkaline degreasing (adapted to suit the oil- or grease-based films encountered);
- b) pickling (usually in hydrochloric acid, inhibited);
- c) electrolytic alkaline degreasing (preferably anodic);
- d) electroplating;
- e) supplementary treatment comprising passivation and possibly sealing;
- f) drying.

#### 7.2 Supplementary treatments

#### 7.2.1 Passivation layers

Passivation layers are conversion coatings produced by dipping components in, or spraying them with, passivation solutions. The deposited coating reacts with the passivation solution to form a thin film that protects the metallic coating. Some of the deposited coating is normally dissolved as a result of the reaction.

#### 7.2.2 Post treatment

The post treatment is carried out by applying additional organic and/or inorganic substances to, or incorporating them in, the passivation.

Depending on the geometry of the component and the process, this may result in an accumulation of several post treatments. The latter shall be kept to a minimum, e.g. by spraying in the case of rack-electroplated components or agitation in the case of barrel-electroplated components.

#### 7.3 Barrel-/rack-plating (handling of parts)

#### 7.3.1 Barrel electroplating

Among the parts typically treated in coating barrels are screws, nuts and other small components. After the parts have been loaded into the barrels in bulk, they are pre-treated and coated with the barrel rotating. The rotation ensures that the coating of all the components is more or less identical, but it may give rise to surface damage. This damage can be minimized, for example, by reducing the barrel rotation or the drop height when the barrels are emptied. The corrosion resistance is normally less than that achieved by rack electroplating.

#### 7.3.2 Rack electroplating

Parts may have to be coated in rack technology because of their size or shape or possibly because they have to meet special requirements. In this process, the components are placed on racks for coating. The characteristics of the coatings (especially the thickness of the metallic coating) may vary with the position of the components on the rack, but optimization may be possible, for example, by designing racks for particular components.

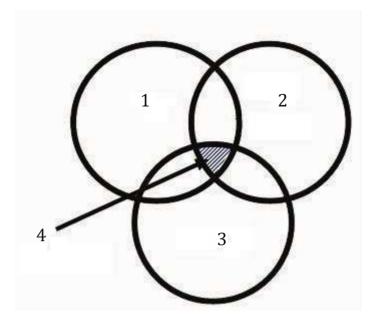
#### 7.4 Hydrogen embrittlement

#### 7.4.1 Basic factors

During processes such as pickling, electrolytic cleaning and electrodeposition of metal coatings, steel parts coated by the procedure described in this International Standard may absorb hydrogen as a result of hydrogen diffusing preferentially through the metal lattice to energetically favourable positions, such as structural defects in the lattice and points with high concentrations of stress.

This may result in delayed hydrogen-induced brittle fractures. Particular attention has to be paid to the critical interaction of

- the material and its characteristics (strength, hardness),
- hydrogen absorption during the pre-treatment and coating processes, and
- mechanical stresses in the components, including those that vary locally as a function of shape.



#### Key

- 1 material
- 2 mechanical stress (including internal and local stress)
- 3 hydrogen in the material
- 4 fracture in the event of critical interaction

Figure 1 — Interaction of material, mechanical stress and hydrogen absorption

The crucial material properties are strength and toughness, the risk of hydrogen embrittlement increasing with the strength of the material.

All steel parts having a tensile strength Rm  $\geq 1~000~N/mm^2$  (even if localized as in case-hardened or cold-worked structures or in the vicinity of welds) are considered high-strength and classified as critical.

NOTE Rm is the symbol of the tensile strength. The dimension of tensile strength is force per unit area; the commonly used units are  $N/mm^2$  or MPa (megapascal).

#### 7.4.2 Choice of procedure

#### 7.4.2.1 Basis material strength <1 000 N/mm<sup>2</sup>

The choice of treatment procedures is unrestricted, provided the requirements of this International Standard are fulfilled and service performance is not adversely affected.

#### 7.4.2.2 Basis material strength ≥1 000 N/mm<sup>2</sup>

In coating high-strength components, the prevention of delayed brittle fractures (hydrogen embrittlement) is of paramount importance.

The surface treatment shall be performed in such a way as to prevent damage due to delayed hydrogen-induced brittle fractures with a high degree of certainty. How to deal with potentially defective coating (e.g. by stripping and re-coating) shall be investigated and the outcome documented.

The steps taken to minimize the risk of delayed hydrogen-induced brittle fractures shall be agreed between the electroplater and the purchaser. (The baking time and temperature shall be agreed between the supplier and purchaser, based on the coating material and properties.)

The necessary process investigation and process testing associated with the manufacture may be performed, for example, by strain testing an adequate number of suitable hydrogen-sensitive specimens.

The advice given in ISO 9587 and ISO 9588 shall be borne in mind.

To prevent brittle fractures, heat treatment processes shall be performed after electroplating to promote hydrogen effusion, and possibly even before electroplating, to relieve internal component stresses. In this connection, see ISO 9587 and ISO 9588.

Particular attention shall be paid to ensuring that the deposited metallic coating does not act as a diffusion barrier and prevent hydrogen effusion. The properties of the components shall not be adversely affected.

NOTE Guideline values for heat treatment to promote hydrogen effusion are given in <u>Table 4</u>.

Table 4 — Guideline values for heat treatment to promote hydrogen effusion after electroplating

Tensile strength,  R <sub>m</sub> N/mm <sup>2</sup>	Heat treatment conditions in hot-air circulation oven Minimum holding time at component temperature (215 $\pm$ 15) °C $$\rm h$$				
1 000 to 1 250	6				
>1 250 to 1 450	12				
>1 450 to 1 600	20				
>1 600 to 2 000	24				

In addition to the measures described above, components potentially subject to hydrogen damage shall be tested as specified in ISO 9588.

#### 8 Requirements to be met by coatings and test methods

#### 8.1 Thickness

<u>Table 5</u> shows the minimum zinc or zinc-alloy coating thicknesses  $(d_{min})$  provided on significant surfaces by the specified processes.

The thicknesses of zinc and zinc-alloy coatings shall preferably be determined by X-ray fluorescence as in ISO 3497.

Examples of other methods that can be used are as follows:

- a) microscopic determination as in ISO 1463;
- b) coulometric determination as in ISO 2177;
- c) magnetic determination as in ISO 2178;
- d) eddy current permeability as in ISO 2360 and ISO 15549.

No account is taken of the thicknesses of passivation and sealant layers.

#### 8.2 Adhesion

There shall be no peeling or blistering if the test pieces are kept at  $(220 \pm 10)$  °C for 30 min and then immediately quenched in water at 15 °C to 25 °C (thermal shock test based on ISO 2819).

Bending or burnishing, if feasible, is recommended as a further adhesion test.

#### 8.3 Absence of Cr(VI)

The coating deposited shall be free of Cr(VI) when analysed as specified in ISO 3613:2010, 5.5.2.

#### 8.4 Accelerated corrosion testing

#### 8.4.1 General

The process stability of the coatings can be controlled by salt spray test according to ISO 9227. For qualification of coated parts, the guidelines in, e.g. ISO/TR 16335, can be used.

NOTE Since the temperature, humidity, flow conditions, etc. may be very different from the limited test atmospheres specified in the accelerated corrosion tests, the results of accelerated corrosion tests cannot be used without qualification to predict the corrosion behaviour of components when in service.

#### 8.4.2 Minimum corrosion resistance of passivated zinc and zinc-alloy coatings

No corrosion products, such as white corrosion in the coating or red rust in the basis material, shall appear within the minimum test times specified for a given coating system and test. The evaluation shall be based on the significant surfaces of the component.

The minimum corrosion resistances apply in the "coated state" and also after thermal ageing at 120 °C for 24 h prior to the corrosion test. Thermal ageing is unnecessary for the Zn//An//T0 coating system.

Treatments such as waxing and greasing, which have not been agreed but which could improve resistance in the corrosion test, are not permitted.

Operations such as sorting, transportation, assembly or exposure to aggressive media shall be avoided prior to corrosion testing since they may adversely affect the corrosion resistance properties of the coatings. Assessment or limitation of such damage is not covered by this International Standard.

The corrosion resistance that can be achieved by coatings may depend not only on the coating system and the quality of the coating, but also to a considerable extent on the component to be coated (e.g. on the material and the geometry). If optimum coating quality cannot readily be achieved in the case of a component (e.g. because of defects in the material or complicated component geometry), it may be necessary to agree a reduced corrosion resistance.

 $Table\ 5-Minimum\ coating\ thicknesses\ and\ minimum\ test\ times\ for\ passivated\ transparent\ or\ iridescent\ zinc\ and\ zinc\ alloy\ coatings\ subjected\ to\ the\ ISO\ 9227\text{-NSS}\ test$ 

	Coating designation	Type of electroplating	Minimum test time h			
Type of surface protection coating			Without coating corrosion	Without basis material corrosion (as a function of Zn or Zn-alloy coating thickness)		
				5 μm	8 µm	12 μm
Transparent-passivated	Zn//An//T0	Barrel	8	48	72	96
electroplated zinc coating		Rack	16	72	96	120
Iridescent-passivated	Zn//Cn//T0	Barrel	72	144	216	288
electroplated zinc coating	,,,,,	Rack	120	192	264	336
Iridescent-passivated		Barrel	120	192	264	360
and sealed electroplated zinc coating	Zn//Cn//T2	Rack	168	264	360	480
Transparent-passivated	ZnFe//An//T0	Barrel	96	168	240	312
electroplated zinc-iron alloy coating		Rack	168	240	312	384
Transparent-passivated	ZnFe//An//T2	Barrel	144	216	288	384
and sealed electroplated zinc-iron alloy coating		Rack	216	312	408	528
Iridescent-passivated	ZnFe//Cn//T0	Barrel	96	168	240	312
electroplated zinc-iron alloy coating		Rack	168	240	312	384
Iridescent-passivated	ZnFe//Cn//T2	Barrel	144	216	288	384
and sealed electroplated zinc-iron alloy coating		Rack	216	312	408	528
Transparent-passivated	ZnNi//An//T0	Barrel	120	480	720	<b>720</b> a
electroplated zinc- nickel alloy coating		Rack	192	600	720	<b>720</b> <sup>a</sup>
Transparent-passivated		Barrel	168	600	720	<b>720</b> a
and sealed electroplated zinc-nickel alloy coating		Rack	360	720	720 <sup>a</sup>	<b>720</b> <sup>a</sup>
Iridescent-passivated	ZnNi//Cn//T0	Barrel	120	480	720	720
electroplated zinc- nickel alloy coating		Rack	192	600	720	<b>720</b> <sup>a</sup>
Iridescent-passivated	ZnNi//Cn//T2	Barrel	168	600	720	<b>720</b> a
and sealed electroplated zinc-nickel alloy coating		Rack	360	720	720a	720

Table 6 — Minimum coating thickness and test times for passivated black zinc and zinc-alloy coatings subjected to the ISO 9227-NSS test

	Coating designation	Type of electroplating	Minimum test time h			
Type of surface protection coating			Without coating corrosion	Without basic material corrosion (as a function of Zn or Zn-alloy coating thickness)		
				5 μm	8 µm	12 μm
Black passivated	Zn//Fn//T0	barrel	24	48	72	96
electroplated zinc coating		rack	48	72	96	120
Black passivated and	Zn//Fn//T2	barrel	72	144	216	288
sealed electroplated zinc coating		rack	120	192	264	360
Black-passivated and	ZnFe//Fn//T2	barrel	120	192	264	360
sealed electroplated zinc-iron alloy coating		rack	168	264	360	480
Black-passivated and	ZnNi//Fn//T2	barrel	168	480	720	<b>720</b> a
sealed electroplated zinc-nickel alloy coating		rack	240	600	720	<b>720</b> <sup>a</sup>
Black-passivated	ZnNi//Fn/T0	barrel	48	480	720	<b>720</b> a
electro-plated zinc- nickel alloy coating		rack	72	600	720	<b>720</b> <sup>a</sup>
a To limit the testing costs, the requirement has been reduced to 720 h.						

Faint visual variations (a hazy grey veil) that are not voluminous in nature are permitted and do not adversely affect the corrosion protection layer.

#### 9 Test report

#### 9.1 General information

The electroplater shall provide a test report that:

- a) refers to this International Standard, i.e. ISO 19598;
- b) certifies conformance with the requirements of this International Standard;
- c) gives the name of the electroplater;
- d) records process details (rack or barrel plating and coating system deposited).

#### 9.2 Coatings on materials having a tensile strength ≥1 000 N/mm<sup>2</sup>

The test report shall certify that designated steps have been taken to minimize the risk of delayed hydrogen-induced brittle fracture.

#### 9.3 Test results

The tests in 8.2, 8.3 and 8.4 shall be carried out concurrently with the process.

The test report shall include the following details:

- a) the results of the technological tests specified in Clause 8;
- b) by whom the tests were performed (the supplier and/or subcontractor and/or an independent testing laboratory).

## **Bibliography**

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- [2] ISO 2177, Metallic coatings Measurement of coating thickness Coulometric method by anodic dissolution
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- [7] ISO/TR 16335, Corrosion of metals and alloys Corrosion tests in artificial atmospheres Guidelines for selection of accelerated corrosion test for product qualification

