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## Metallic and other inorganic coatings — Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys — Test methods

*Revêtements métalliques et autres revêtements inorganiques — Couches de conversion au chromate sur zinc, cadmium et alliages d'aluminium-zinc et de zinc-aluminium — Méthodes d'essai*  
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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 [www.iso.org/directives](http://www.iso.org/directives)).

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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 262, *Metallic and other inorganic coatings, including for corrosion protection and corrosion testing of metals and alloys*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 3613:2010), which has been technically revised. The main changes compared with the previous edition are as follows:

- [Table 1](#) has been revised;
- analysis method described in [6.5](#) was aligned with the method described in IEC 62321-7-1;
- procedure for the preparation of test solution C 2 in [4.3.2](#) has been revised and a second standard solution has been added;
- limiting time has been amended in [6.1](#).
- procedure for testing with test solution C 2 has been revised in [6.5.2](#).
- description of the calibration of spectrophotometer has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

This document specifies methods for the qualitative determination of the presence of chromate conversion coatings as well as the total chromium content of chromate conversion coatings.

The application of very thin, colourless, practically invisible chromate conversion coatings is frequently called “passivation”, while the application of thicker, coloured chromate conversion coatings is called “chromating”. The term “passivation” is not correct, as it does not conform to the ISO 2080 designation, and is therefore deprecated.

Chromate conversion coatings are based on a solution containing Cr(VI). Both the process solution and the coating contain hexavalent chromium. The term passivation is nowadays often used for Cr(VI)-free coatings replacing chromate conversion coatings. Products containing Cr(VI) are not allowed within EU and processes using Cr(VI) are strictly regulated.

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# Metallic and other inorganic coatings — Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys — Test methods

**WARNING** — This document calls for the use of substances and/or procedures that can be injurious to health if adequate safety measures are not taken. This document does not address any health hazards, safety or environmental matters associated with its use. It is the responsibility of the user of this document to establish appropriate health, safety and environmentally acceptable practices and take suitable actions for any national and international regulations.

## 1 Scope

This document specifies methods for the determination of

- the presence of colourless chromate conversion coatings,
- the presence of hexavalent chromium in colourless and coloured coatings on zinc or cadmium or aluminium-zinc (mass fraction of aluminium: 55 %, within a range of 54 % to 56 % mass fraction) and zinc-aluminium (mass fraction of aluminium: 5 %) alloys,
- the total chromium content per unit area on zinc and cadmium,
- the mass per unit area of both colourless and coloured coatings,
- the satisfactory adhesion of chromate conversion coatings, and
- the quality of chromate coatings.

These methods are applicable to

- colourless and coloured chromate conversion coatings containing trivalent and hexavalent chromium in varying proportions and produced by either chemical or electrochemical processes, and
- chromate coatings that are free from any supplementary coatings, such as oil, water or solvent-based polymers or wax.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3892, *Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods*

ISO 4520, *Chromate conversion coatings on electroplated zinc and cadmium coatings*

IEC 60068-2-30, *Environmental testing — Part 2-30: Tests — Test Db: Damp heat, cyclic (12 h + 12 h cycle)*

## 3 Terms and definitions

No terms and definitions are listed in this document.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

### 4 Reagents

Use only reagents of recognized analytical grade and distilled water or water of equivalent purity, unless otherwise specified, for analysis.

#### 4.1 Test solution A (see 6.2).

Dissolve 1 g of diphenylcarbazine in a mixture of 20 ml of acetone, 60 ml of glacial acetic acid and 40 ml of distilled water contained in a beaker. Add 15 ml of concentrated hydrochloric acid ( $\rho = 1,18 \text{ g/cm}^3$ ), stir and add slowly 30 ml of sodium hypochlorite solution (10 % to 15 % available chlorine). Add 5 ml of hydrogen peroxide (30 % volume fraction) slowly with continuous stirring. Leave the solution in the open beaker for 24 h in a fume cupboard, to allow excess chlorine to escape, before use.

The solution does not deteriorate with age and can be kept in a bottle with a loosely fitted stopper. However, there can be losses due to evaporation and the concentration can alter so it is discarded after six months.

#### 4.2 Test solution B (see 6.3).

Dissolve 50 g of lead acetate trihydrate  $[(\text{CH}_3\text{COO})_2\text{Pb}\cdot 3\text{H}_2\text{O}]$  in 1 l of distilled water or water of equivalent purity. Ensure that the pH of the solution is between 5,5 and 6,8 as prepared. If the pH of the solution is outside this range, discard the solution and obtain a new supply of lead acetate.

Any white precipitate formed during the initial preparation of the solution may be dissolved by small additions of glacial acetic acid, provided that the pH is not reduced to a value below 5,5. Discard the stock solution if the white precipitate does not disappear.

#### 4.3 Test solution C (see 6.5)

##### 4.3.1 Test solution C 1

Dissolve 0,4 g of diphenylcarbazine in a mixture of 20 ml of acetone and 20 ml of ethanol (96 %). After dissolution, add 20 ml of 75 % orthophosphoric acid solution and 20 ml of distilled water. Prepare this solution not more than 8 h prior to use.

##### 4.3.2 Test solution C 2

Add 700 ml of orthophosphoric acid, of specific gravity 1,7, to 250 ml of distilled water or water of equivalent purity and make up to 1 000 ml.

Dissolve 0,5 g of 1,5-diphenylcarbazine of analytical reagent grade in 50 ml of acetone of analytical reagent grade. Dilute slowly, while stirring, with 50 ml of distilled water or water of equivalent purity (rapid mixing can result in precipitation of diphenylcarbazine). Keep the solution in a dark glass bottle in the refrigerator. The solution shall be discarded after four weeks or earlier when the solution becomes discoloured.

For the Cr(VI) stock solution, dissolve 0,113 g of  $\text{K}_2\text{Cr}_2\text{O}_7$  of analytical reagent grade (dried for 1 h at 100 °C before use) in distilled water or water of equivalent purity and make up to the mark in a 1 000 ml volumetric flask. The solution shall be discarded after nine months.

From this stock solution, prepare two Cr(VI) standard solutions.



For 0,10 µg/ml Cr(VI): Measure 2,5 ml of this solution into a second 1 000 ml volumetric flask and make up to the mark. 1 ml of this standard solution contains 0,10 µg of Cr(VI).

For 0,13 µg/ml Cr(VI): Measure 3,3 ml of this solution into a third 1 000 ml volumetric flask and make up to the mark. 1 ml of this standard solution contains 0,13 µg of Cr(VI).

For the preparation of the comparison solution for 0,10 µg/cm<sup>2</sup> Cr(VI), add 1 ml of orthophosphoric acid and 1 ml of diphenylcarbazide solution to 50 ml of the standard Cr(VI) solution with 0,10 µg/ml Cr(VI) and mix thoroughly. Then add 2 ml of the diphenylcarbazide solution and mix. Allow the solution to stand for 10 min for the colour reaction to be completed.

For the preparation of the comparison solution for 0,13 µg/cm<sup>2</sup> Cr(VI), add 1 ml of orthophosphoric acid to 50 ml of the standard Cr(VI) solution with 0,13 µg/ml Cr(VI) and mix thoroughly. Then add 2 ml of the diphenylcarbazide solution and mix. Allow the solution to stand for 10 min for the colour reaction to be completed.

#### 4.4 Test solution D (see 6.6 and 6.7).

Dissolve 0,50 g of diphenylcarbazide in 50 ml of acetone. Dilute slowly, while stirring, with 50 ml of water (rapid mixing can result in precipitation of diphenylcarbazide).

For maximum stability, store the solution under refrigeration in an amber-coloured glass bottle.

#### 4.5 Sulfuric acid, diluted 1 + 3.

Slowly add one volume of concentrated sulfuric acid ( $\rho = 1,84 \text{ g/cm}^3$ ) to three volumes of water.

#### 4.6 Ammonium persulfate [(NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>].

#### 4.7 Sodium hydroxide (NaOH), 240 g/l solution.

#### 4.8 Silver nitrate (AgNO<sub>3</sub>), 17 g/l solution.

#### 4.9 Potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>), standard solution.

Dilute 2 ml of standard volumetric potassium dichromate solution (4,9 g/l) to 1 000 ml.

#### 4.10 Phosphate buffer solution.

Dissolve 55 g of sodium dihydrogen orthophosphate monohydrate (NaH<sub>2</sub>PO<sub>4</sub>·H<sub>2</sub>O) in 100 ml of water.

## 5 Apparatus

Use normal laboratory apparatus and the following.

Glassware shall be of grade A. Pipettes shall be calibrated.

Clean all glassware thoroughly. It is recommended that cleaning be done by boiling the glassware with nitric acid (HNO<sub>3</sub>) of 30 % mass fraction, and then rinsing thoroughly with distilled water or water of equivalent purity. Adequate cleanliness should be demonstrated through the analysis of method blanks.

**5.1 Photoelectric absorptiometer (colorimeter)**, with a filter having a mean transmission of 520 nm. The absorptiometer shall be used with an absorption cell having an optical path length of 10 mm.

**5.2 Spectrophotometer**, with wavelength set at 540 nm. The spectrophotometer shall be used with an absorption cell having an optical path length of 10 mm.

5.3 **Analytical balance**, with an accuracy of 0,10 mg.

## 6 Test methods

### 6.1 General

The test surface, prior to the following tests, shall be free of all contaminants, fingerprints and other extraneous stains. If the surface is coated with a thin oil film, this shall be removed prior to the test by degreasing using a suitable solvent at room temperature (not exceeding 35 °C). If it is necessary to store samples, they shall be stored at temperatures not exceeding 40 °C and at a relative humidity below 70 %. For test purposes, the samples shall not be subjected to forced drying at temperatures in excess of 35 °C. Treatment in alkaline solutions shall not be performed as chromate conversion coatings are broken down by alkalis. Spot tests are not always a precise means of determining the presence of chromate coatings.

The test methods for the following are given in [6.2](#) to [6.7](#):

- a) the presence of a colourless chromate coating on zinc, cadmium and aluminium-zinc alloys;
- b) the presence of hexavalent chromium in both coloured and colourless chromate coatings;
- c) the determination of hexavalent chromium content;
- d) the determination of total chromium content.

The tests shall be carried out within the following time limits:

- a minimum of 24 h after the application of the chromate conversion coating for almost all tests ([6.2](#) to [6.4](#), [6.5.2](#) and [6.6](#) to [6.10](#));
- a minimum of 5 d after the application of the chromate conversion coating for the test specified in [6.5.3](#) to [6.5](#);
- a maximum of 3 d for the tests specified in [6.2](#) to [6.4](#) and [6.5.2](#).
- a maximum of 30 d for the tests specified in [6.6](#) and [6.7](#). It could be recommendable to apply this limit also to the test specified in [6.5.3](#) to [6.5.5](#).

The test methods for the following are given in [6.8](#), [6.9](#) and [6.10](#), respectively:

- the determination of mass per unit area of chromate conversion coatings;
- adhesion by abrasion;
- the evaluation of the quality of chromate layer.

### 6.2 Test for the presence of a colourless chromate coating on zinc

Apply one drop of test solution A ([4.1](#)) to the chromated zinc surface. The formation of a red or red-violet colour in the drop shall be deemed to indicate the presence of chromate film.

### 6.3 Test for the presence of a colourless chromate coating on zinc and cadmium

Place one drop of test solution B ([4.2](#)) on the surface to be tested.

In the case of a zinc substrate, observe the appearance of the spot for 3 min. The formation of a dark or black stain, at least 1 min after the application of the test solution, shall be deemed to indicate the presence of a colourless chromate conversion coating.

Black stain formation after more than 3 min can be indicative of the presence of supplementary coatings, such as wax or oil.