
**Chromate conversion coatings on zinc,
cadmium, aluminium-zinc alloys
and zinc-aluminium alloys — Test methods**

*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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3613 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Methods of inspection and coordination of test methods*.

This second edition cancels and replaces the first edition (ISO 3613:1980), which has been technically revised.

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Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys — Test methods

WARNING — This International Standard calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves either the designer, the producer, the supplier or the user from statutory and all other legal obligations relating to health and safety at any stage of manufacture or use.

1 Scope

This International Standard specifies methods for the determination of the

- presence of colourless¹⁾ chromate conversion coatings;
- presence and quantity of hexavalent chromium in colourless and coloured coatings on zinc, cadmium, aluminium (55 % mass fraction)-zinc and zinc-aluminium (5 % mass fraction) alloys;
- total chromium content per unit area on zinc and cadmium;
- mass per unit area of both colourless and coloured coatings;
- satisfactory adhesion of chromate conversion coatings;
- quality of chromate coating.

These methods are applicable

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

1) 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 often called "chromating". The term passivation is not correct and is, therefore, not recommended.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3892:2000, *Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods*.

ISO 4520:1981, *Chromate conversion coatings on electroplated zinc and cadmium coatings*.

ISO 9227:1990, *Corrosion tests in artificial atmospheres — Salt spray tests*.

IEC 60068-2-30:1980, *Environmental testing — Part 2: Tests. Test Db and guidance: Damp heat, cyclic (12 + 12 hour cycle)*.

3 Reagents

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

3.1 Test solution A (see 5.2).

Dissolve 1 mg of diphenylcarbazide in a mixture of 60 ml glacial acetic acid and 40 ml distilled water contained in a beaker. Add 15 ml of concentrated hydrochloric acid (relative density 1,18), stir and add slowly 30 ml of sodium hypochlorite solution (10 % to 15 % available chlorine). Add 5 ml of hydrogen peroxide (30 %) 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.

NOTE The solution does not deteriorate with age and therefore can be kept in a bottle with a loosely fitting glass stopper over a period of several months.

3.2 Test solution B (see 5.3).

Dissolve 50 g of lead acetate trihydrate $[(CH_3COO)_2Pb \cdot 3H_2O]$ in 1 l of distilled or de-ionized water. 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.

3.3 Test solution C (see 5.5).

Dissolve 0,4 g of diphenylcarbazide in a mixture of 20 ml acetone and 20 ml 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.

3.4 Test solution D (see 5.6 and 5.7).

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.

3.5 Sulfuric acid, diluted 1 + 3.

Slowly add 1 volume of concentrated sulfuric acid (relative density 1,84) to 3 volumes of water.

3.6 Ammonium persulfate [(NH₄)₂S₂O₈].**3.7 Sodium hydroxide (NaOH)**, (240 g/l) solution.**3.8 Silver nitrate (AgNO₃)**, 17 g/l solution.**3.9 Potassium dichromate (K₂Cr₂O₇)**, standard solution.

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

3.10 Phosphate buffer solution

Dissolve 55 g of sodium dihydrogen orthophosphate monohydrate (NaH₂PO₄·H₂O) in 100 ml water.

4 Apparatus

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

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

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5 Test methods

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5.1 General

The test surface, prior to the following tests, shall be free of all contaminants, finger prints and other extraneous stains. If the surface is coated with thin oil, it shall be removed prior to the test by degreasing using a suitable solvent at room temperature (not exceeding 35 °C). 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.

The test methods for

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

are given in 5.2 to 5.7.

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

- minimum 24 h after the application of the chromate conversion coating for all tests (5.2 to 5.10);

- maximum 3 d for tests specified in 5.2 to 5.5²⁾;
- maximum 30 d for tests specified in 5.6 and 5.7.

The test methods for

- a) the determination of mass per unit area of chromate conversion coatings,
- b) adhesion by abrasion and
- c) the evaluation of the quality of chromate layer

are given in 5.8, 5.9 and 5.10, respectively.

5.2 Test for the presence of a colourless chromate coating on zinc

Apply 1 drop of test solution A (3.1) 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.

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

Place 1 drop of test solution B (3.2) 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.

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In the case of a cadmium substrate, observe the appearance of the spot for 1 min. The formation of a dark or black stain at least 5 s 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 1 min can be indicative of the presence of supplementary coatings, such as wax or oil.

For comparison purposes, test an untreated surface similarly. Untreated zinc and cadmium surfaces or the surfaces that have been treated for chromate conversion coating but do not have continuous coating will react with the lead acetate solution to form a black stain on the untreated or bare zinc and cadmium surfaces almost immediately, within 2 s to 5 s, after the application of the test solution.

NOTE 1 Because of the sensitivity of the method, the test is considered to be a quality control tool for the processor. Items with damaged chromate layers should not be tested by this method.

NOTE 2 Chromate conversion coatings exposed to temperatures above 60 °C will have considerably reduced resistance to corrosion in the accelerated test method (5.10), and will show black stain in less than 60 s on zinc, and less than 5 s on cadmium.

NOTE 3 No significance should be placed upon any differences in the reaction time to black spot formation between 1 min and 3 min on a zinc substrate, or between 5 s and 60 s on a cadmium substrate, with colourless chromate conversion treatment. Factors such as substrate surface texture (roughness), chromate film thickness variations (due to processing conditions), variations in the ambient temperature of the test, and precise pH control of the lead acetate solution have an influence on the reaction time and therefore preclude any rating based upon times beyond those already stated. Similarly, the test cannot be used to compare the corrosion protection of chromate conversion coatings made from various types of chromate treatment solutions.

2) This may require special arrangements to be made between the purchaser and the supplier.

5.4 Test for the presence of a colourless chromate coating on aluminium-zinc (55 % mass fraction aluminium) and zinc-aluminium (5 % mass fraction aluminium) alloys

Expose the test specimen for 24 h to an accelerated damp heat test in accordance with IEC 60068-2-30.

The formation of a dark grey to black stain over the main part of the area shall be deemed to indicate the absence of chromate conversion coating.

NOTE This test does not have any significant effect on the surface appearance if a chromate conversion coating is present.

5.5 Test for the presence of hexavalent chromium in both colourless and coloured chromate coatings

Place 1 drop to 5 drops of test solution C (3.3) on the surface to be tested. If hexavalent chromium is present, a red to violet colour will appear within a few minutes. Ignore any colour that appears much later, for example on drying. For comparison purposes, test an untreated surface similarly.

NOTE The treatment will not have any significant effect on the surface appearance.

5.6 Determination of hexavalent chromium content of coloured chromate coatings

5.6.1 Preparation of calibration graph

5.6.1.1 To a series of five 250 ml volumetric flasks, add respectively 0 ml (i.e. the compensation solution), 10 ml, 20 ml, 30 ml and 40 ml standard potassium dichromate solution (3.9). Add to each flask 100 ml water and 3 ml sulfuric acid solution (3.5). To one flask add 3 ml test solution D (3.4) and mix well. Exactly 2 min after the addition of test solution D, add 25 ml phosphate buffer solution (3.10), dilute to the mark and mix. Repeat this procedure with the other four flasks.

5.6.1.2 Transfer a portion of each solution to an absorption cell and measure the absorbance in the absorptiometer (4.1) or spectrophotometer (4.2). Complete each reading within 25 min after the addition of test solution D (3.4), as the colour is not stable. Subtract the compensation solution (see 5.6.1.1) from each reading and plot the differences against micrograms of chromium per 250 ml.

5.6.2 Blank test

Carry out a blank test in accordance with 5.6.3 and record the result.

NOTE Test sample for blank test: 0 ml standard potassium dichromate solution + addition of reagents and test solution as described in 5.6.6.1.

5.6.3 Determination

5.6.3.1 Cut a test piece measuring about 50 mm × 100 mm from the coated item. Bring to boiling 50 ml of distilled water in a suitable vessel and totally immerse the test specimen in this. Leach for exactly 5 min while the water continues to boil, remove the specimen and rinse over the vessel. Cool the vessel and its contents to room temperature, add 3 ml of sulfuric acid solution (3.5) and mix. Transfer the contents to a 250 ml volumetric flask. Rinse the vessel twice into the flask with 10 ml to 15 ml of distilled water, add 3 ml of test solution D (3.4) and mix. Exactly 2 min after addition of the test solution D add 25 ml of phosphate buffer solution (3.10), dilute to the mark and mix.

5.6.3.2 Transfer a portion of the solution to an absorption cell and measure the absorbance in the absorptiometer (4.1) or spectrophotometer (4.2). Complete the reading within 25 min of the addition of test solution D (3.4), as the colour is not stable. Subtract the blank test reading (see 5.6.2) from the instrument reading and using the calibration graph (see 5.6.1) convert the result into micrograms per 250 ml.

5.6.3.3 If the reading obtained falls within the portion of the calibration graph below 10 µg, repeat the determination using a larger test piece (1,5 × to 2 × the dimensions given in 5.6.3.1).