
International Standard



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Chromate conversion coatings on zinc and cadmium — Test methods

Couches de conversion au chromate sur zinc et cadmium — Méthodes d'essai

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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Poland

Chromate conversion coatings on zinc and cadmium — Test methods

1 Scope and field of application

This International Standard specifies methods for the determination on zinc and cadmium of :

- the presence of colourless¹⁾ chromate conversion coatings;
- the presence and quantity of hexavalent chromium in colourless and coloured coatings;
- the total chromium content per unit area;
- the satisfactory adhesion of coloured coatings.

These methods are applicable only to chromate conversion coatings which are free from any supplementary coating, such as oil, water or solvent-based polymers, or wax.

A method for the determination of mass per unit area of both colourless and coloured chromate conversion coatings and for the neutral salt spray test are specified in ISO 3892 and ISO 3768 respectively and are listed in 3.5 and 3.7 for completeness.

2 References

ISO 3768, *Metallic coatings — Neutral salt spray test (NSS test)*.

ISO 3892, *Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods²⁾*.

3 Test methods

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 (3.1 to 3.7);
- maximum 3 days for tests specified in 3.1 and 3.2³⁾;
- maximum 30 days for tests specified in 3.1 and 3.4

3.1 Test for presence of a colourless chromate coating

3.1.1 Test solution

Dissolve 50 g of lead acetate trihydrate $[(\text{CH}_3\text{COO})_2\text{Pb}\cdot 3\text{H}_2\text{O}]$ in 1 litre of distilled or deionized water. The pH of the solution shall be between 5,5 and 6,8.

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

3.1.2 Procedure

Place 1 drop of the lead acetate solution (3.1.1) on the surface to be tested.

In the case of a zinc substrate, observe the spot for 3 min. The presence of a colourless chromate conversion coating will be indicated when the formation of a dark or black stain is delayed by at least 1 min after application of the test solution. Delay of black stain formation by more than 3 min may be indicative of supplementary coatings, such as wax or oil (see note).

In the case of a cadmium substrate, observe the spot for 1 min. The presence of a colourless chromate conversion coating will be indicated when the formation of a dark or black stain is delayed by at least 6 s. Delay of black stain formation by more than 1 min may be indicative of supplementary coatings, such as wax or oil.

Zinc and cadmium surfaces which have not been treated with a chromate conversion coating will react with the lead acetate solution to form a black stain within 2 to 5 s.

No significance should be placed upon any differences in reaction time to black spot formation between 1 and 3 min on a zinc substrate with colourless chromate conversion treatment, or between 6 to 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

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 deprecated.

2) At present at the stage of draft.

3) This may require that special arrangements be made between the purchaser and supplier.

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 various types of chromate treatment solutions.

NOTE — Chromate conversion coatings which have lost their protective value through dehydration by exposure to temperatures higher than 60 °C will form black stain in less than 60 s on zinc, and less than 5 s on cadmium.

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

3.2.1 Test solution

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

3.2.2 Procedure

Place 1 to 5 drops of the test solution (3.2.1) 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 which appears much later, for example on drying.

For comparative purposes, a surface that is known not to have been chromate treated may be similarly tested.

3.3 Determination of hexavalent chromium content of coloured chromate coatings — Diphenylcarbazide colorimetric method

3.3.1 Reagents

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

3.3.1.1 Sulphuric acid, 9 N solution (3 volumes of water + 1 volume of sulphuric acid, ρ , 1,84 g/ml).

3.3.1.2 Diphenylcarbazide, solution.

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

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

3.3.1.3 Potassium dichromate standard solution.

Dilute 2 ml of 0,1 N standard volumetric potassium dichromate solution to 1 litre.

1 ml of this solution contains 3,47 µg of chromium. A solution containing 9,90 mg/l of potassium dichromate contains 3,5 µg/ml of chromium.

3.3.1.4 Phosphate, buffer solution.

Dissolve 55 g of sodium dihydrogen orthophosphate monohydrate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$) in 100 ml of water.

3.3.2 Apparatus

Ordinary laboratory apparatus and

3.3.2.1 Photoelectric absorptiometer (colorimeter) or spectrophotometer.

With a photoelectric absorptiometer, use a filter having a mean transmission of 520 nm. A wavelength of 540 nm shall be set for a spectrophotometer. Use an absorption cell with an optical path length of 1 cm in both cases.

3.3.3 Procedure

3.3.3.1 Preparation of calibration graph

To a series of five 250 ml volumetric flasks, add respectively 0 - 10 - 20 - 30 and 40 ml of the standard potassium dichromate solution (3.3.1.3). Add to each flask 100 ml of water and 3 ml of the sulphuric acid solution (3.3.1.1). Take each flask through the following steps, one flask at a time : add 3 ml of the diphenylcarbazide solution (3.3.1.2) and mix well. Exactly 2 min after addition of the diphenylcarbazide solution, add 25 ml of the phosphate buffer solution (3.3.1.4), dilute to the mark and mix.

Transfer a portion of each solution to an absorption cell and measure the absorbance in the absorptiometer or spectrophotometer (3.3.2.1). Complete each reading within 25 min after addition of the diphenylcarbazide solution, because the colour is not stable. Subtract the blank (0 ml) from each reading and plot the differences against micrograms of chromium per 250 ml.

3.3.3.2 Blank test

Carry out a blank test on the reagents simultaneously with the determination.

3.3.3.3 Determination

Prepare a test piece measuring about 50 mm × 100 mm. Heat 50 ml of distilled water to boiling in a suitable vessel, and totally immerse the test piece. Leach for exactly 5 min while the water continues to boil, remove the test piece, and rinse into the vessel. Cool the vessel and its contents to room temperature, add 3 ml of the sulphuric acid solution (3.3.1.1), and mix. Transfer to a 250 ml volumetric flask. Rinse the vessel twice into the flask with 10 to 15 ml of distilled water, add 3 ml of the diphenylcarbazide solution (3.3.1.2), and mix. Exactly 2 min after addition of the diphenylcarbazide solution, add 25 ml of the phosphate buffer solution (3.3.1.4), dilute to the mark, and mix.

Transfer a portion of the solution to an absorption cell, and measure the absorbance in the absorptiometer or spectrophotometer (3.3.2.1). Complete the reading within 25 min

after addition of the diphenylcarbazide solution, because the colour is not stable. Subtract the blank test reading (3.3.3.2) from the instrument reading, and, using the calibration graph (3.3.3.1), convert into micrograms per 250 ml.

In the event that the reading obtained falls within the portion of the calibration graph below 10 µg, repeat the determination using an appropriately larger test piece.

3.3.4 Expression of results

Determine the total surface area of coating on the test piece, in square centimetres. Divide the hexavalent chromium content obtained in 3.3.3.3 by the surface area of the coating to obtain micrograms of hexavalent chromium per square centimetre.

3.4 Determination of total chromium content of coloured chromate coatings — Diphenylcarbazide colorimetric method

3.4.1 Reagents

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

3.4.1.1 Ammonium persulphate [(NH₄)₂S₂O₈].

3.4.1.2 Sodium hydroxide, 6 N solution (240 g/l).

3.4.1.3 Sulphuric acid, 9 N solution (3 volumes of water + 1 volume of sulphuric acid, ρ 1,84 g/ml).

3.4.1.4 Silver nitrate, 0,1 N solution (17 g/l).

3.4.1.5 Phosphate buffer solution.

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

3.4.1.6 Diphenylcarbazide solution.

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

For maximum stability, keep under refrigeration in an amber-coloured glass bottle.

3.4.1.7 Potassium dichromate standard solution.

Dilute 2 ml of 0,1 N standard volumetric potassium dichromate solution to 1 litre.

1 ml of this solution contains 3,47 µg of chromium. A solution containing 9,90 mg/l of potassium dichromate contains 3,5 µg/ml of chromium.

3.4.2 Apparatus

Ordinary laboratory apparatus and

3.4.2.1 Photoelectric absorptiometer (colorimeter) or spectrophotometer.

With a photoelectric absorptiometer, use a filter having a mean transmission of 520 nm. A wavelength of 540 nm shall be set for a spectrophotometer. An absorption cell with an optical path length of 10 mm shall be used in either case.

3.4.3 Procedure

3.4.3.1 Preparation of calibration graph

To a series of five 250 ml volumetric flasks, add respectively 0 - 10 - 20 - 30 and 40 ml of the standard potassium dichromate solution (3.4.1.7). Add to each flask 100 ml of water and 3 ml of the sulphuric acid solution (3.4.1.3). Take each flask through the following steps, one flask at a time: add 3 ml of the diphenylcarbazide solution (3.4.1.6), and mix well. Exactly 2 min after addition of the diphenylcarbazide solution, add 25 ml of the phosphate buffer solution (3.4.1.5), dilute to the mark, and mix.

Transfer a portion of each solution to an absorption cell and measure the absorbance in the absorptiometer or spectrophotometer (3.4.2.1). Complete each reading within 25 min after addition of the diphenylcarbazide solution, because the colour is not stable. Subtract the blank (0 ml) from each reading and plot the differences against micrograms of chromium per 250 ml.

3.4.3.2 Blank test

Carry out a blank test on the reagents simultaneously with the determination.

3.4.3.3 Determination

Prepare a test piece measuring approximately 50 mm × 100 mm. Heat 25 ml of the sulphuric acid solution (3.4.1.3) to 40 to 50 °C and pour into a vessel containing the test piece. Swirl continuously so that all surfaces are exposed to acid for 10 to 15 s. Remove the test piece, and rinse into the vessel. Dilute to about 175 ml, and add about 0,1 g of the ammonium persulphate (3.4.1.1) and 3 ml of the silver nitrate solution (3.4.1.4). Then add anti-bumping granules and boil for 30 min. Cool and adjust the pH to 1,5 to 1,8 with the sodium hydroxide solution (3.4.1.2) (about 20 ml; to measure the pH, transfer a drop to pH paper do not immerse the paper in the solution). Transfer to a 250 ml one-mark volumetric flask. Rinse the vessel twice into the flask with 10 to 15 ml of water, add 3 ml of the diphenylcarbazide solution (3.4.1.6), and mix. Exactly 2 min after addition of the diphenylcarbazide solution add 25 ml of the phosphate buffer solution (3.4.1.5), dilute to the mark and mix.

Transfer a portion of the solution to an absorption cell and measure the absorbance in the absorptiometer or spectrophotometer (3.4.2.1). Complete the reading within 25 min after addition of the diphenylcarbazide solution, because the colour is not stable. Subtract the blank test reading (3.4.3.2) from the instrument reading, and, using the calibration graph (3.4.3.1), convert into micrograms per 250 ml.

3.4.4 Expression of results

Determine the total surface area of coating on the test piece, in square centimetres. Divide the total chromium content obtained in 3.4.3.3 by the surface area of the coating to obtain micrograms of total chromium per square centimetre.

3.5 Determination of mass per unit area of both colourless and coloured chromate coatings

Carry out the determination by the method specified in ISO 3892.

3.6 Paper and eraser test for adhesion of coloured chromate coatings

Test the adhesion of a coloured chromate coating by rubbing the surface with a soft white tissue paper or with a gritless gum eraser, using normal pressure (about 10 strokes). There shall be no appreciable staining on the tissue paper when this is used for the test, nor shall the chromate coating be removed or worn through to the underlying zinc or cadmium when rubbed with the paper or the eraser.

3.7 Corrosion test

Carry out a neutral salt spray test by the method specified in ISO 3768.

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