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Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods

Couches de conversion sur matériaux métalliques — Détermination de la masse de revêtement par unité de surface — Méthodes gravimétriques

Reference number
ISO 3892:2000(E)

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Foreword

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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 3892 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 3892:1980), which has been technically revised.

INTERNATIONAL STANDARD **ISO 3892:2000(E)**

Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods

WARNING — The materials, operations and equipment listed in this International Standard may be hazardous if suitable precautions are not observed. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies gravimetric methods for determining the coating mass per unit area of conversion coatings on metallic materials.

The methods are applicable to

- phosphate coatings on iron and steel;
- phosphate coatings on zinc and cadmium;
- phosphate coatings on aluminium and its alloys;
- chromate coatings on zinc and cadmium;
- chromate coatings on aluminium and its alloys.

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

The methods do not indicate the presence of bare spots or sites with thicknesses lower than the specified minimum in the measuring areas. In addition, the single values obtained from each measuring area is the mean thicknesses over that area. There can be no further mathematical analysis of this single value, e.g. for statistical control purposes.

2 Apparatus

2.1 Vessel, of glass or other appropriate material, in which the conversion coatings can be dissolved.

2.2 Analytical balance, capable of weighing to the nearest 0,1 mg, for weighing the test pieces under examination before and after dissolution of the conversion coatings.

2.3 Electrical equipment, for electrolytic dissolution (for chromate coatings on zinc and cadmium).

3 Test pieces

The test pieces shall have a maximum mass of 200 g and a total surface area large enough to give a loss of mass sufficient to be tested with adequate sensitivity, and conformity with the requirements of the relevant material or product specification. In order to achieve adequate accuracy in the determination, the total surface area shall be in accordance with the values given in Table 1.

Table 1 — Total surface areas of test pieces

Expected mass of coating per unit area g/m^2	Minimum total surface area of coated test piece cm^2
< 1	400
$\geqslant 1 \leqslant 10$	200
$> 10 \leqslant 25$	100
$> 25 \leqslant 50$	50
> 50	25

NOTE In order to achieve an overall measurement uncertainty (see 5.2) of 5 %, the surface areas should be measured to within 1 %.

4 Reagents and procedures

4.1 General

For the preparation of solutions, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

The sampling procedure shall be as specified in the product standard.

4.2 Phosphate coatings on iron and steel

4.2.1 Manganese phosphate coatings

4.2.1.1 Reagent

4.2.1.1.1 Chromium(VI) oxide (CrO_3), a 50 g/l aqueous solution.

4.2.1.2 Procedure

Dry the test piece (area A, in square centimetres, see 5.1) and weigh it, to the nearest 0,1 mg, using the analytical balance (2.2) (mass m_1, in milligrams, see 5.1). Then immerse the test piece for 15 min in the reagent (4.2.1.1.1), maintained at 75 °C ± 5 °C. Rinse the test piece immediately in clean running water and then in distilled water; dry rapidly and reweigh. Repeat the procedure until a reasonably constant mass (differing by < 0,1 mg) is obtained (mass m_2, in milligrams, see 5.1).

Use freshly prepared reagent for every test piece.

4.2.2 Zinc phosphate coatings

4.2.2.1 Reagent

4.2.2.1.1 Aqueous solution, containing, per litre, 100 g sodium hydroxide, 90 g ethylenedinitrilo tetra-acetic acid, tetrasodium salt dihydrate (EDTA tetrasodium salt) and 4 g triethanolamine.

4.2.2.2 Procedure

Dry the test piece (area A, in square centimetres, see 5.1), and weigh it, to the nearest 0,1 mg, using the analytical balance (2.2) (mass m_1, in milligrams, see 5.1). Then immerse the test piece for 5 min in the reagent (4.2.2.1.1), maintained at 75 °C ± 5 °C. Rinse the test piece immediately in clean running water and then in distilled water; dry rapidly and reweigh (mass m_2, in milligrams, see 5.1).

Use fresh reagent for every test piece.

4.2.3 Iron phosphate coatings

4.2.3.1 Reagent

4.2.3.1.1 Chromium(VI) oxide (CrO_3), a 50 g/l aqueous solution.

4.2.3.2 Procedure

Dry the test piece (area A, in square centimetres, see 5.1) and weigh it, to the nearest 0,1 mg, using the analytical balance (2.2) (mass m_1, in milligrams, see 5.1). Then immerse the test piece for 15 min in the reagent (4.2.3.1.1), maintained at 75 °C ± 5 °C. Rinse the test piece immediately in clean running water and then in distilled water; dry and reweigh. Repeat the procedure until a reasonably constant mass (differing by $< 0,1$ mg) is obtained (mass m_2, in milligrams, see 5.1).

Use freshly prepared reagent for every test piece.

4.3 Phosphate coatings on zinc and cadmium

4.3.1 Reagent

4.3.1.1 Ammonium dichromate, solution in 25 % to 30 % mass fraction ammoniac. During preparation of the solution, its temperature shall not exceed 25 °C.

4.3.2 Procedure

Dry the test piece (area A, square centimetres, see 5.1), and weigh it, to the nearest 0,1 mg, using the analytical balance (2.2) (mass m_1, in milligrams, see 5.1). Then immerse the test piece for 3 min to 5 min in the reagent (4.3.1.1) at room temperature. Carry out this operation in a fume cupboard. Rinse the test piece immediately in clean running water and then in distilled water; dry rapidly and reweigh (mass m_2, in milligrams, see 5.1).

Use freshly prepared reagent for every test piece.

4.4 Crystalline phosphate coatings on aluminium and its alloys

4.4.1 Reagent

4.4.1.1 Nitric acid solution, mass fraction 65 % to 70 %.

4.4.2 Procedure

Dry the test piece (area A, in square centimetres, see 5.1), and weigh it, to the nearest 0,1 mg, using the analytical balance (2.2) (mass m_1, in milligrams, see 5.1). Then immerse the test piece either for 5 min in the reagent (4.4.1.1) maintained at 75 °C ± 5 °C or for 15 min in the same reagent at room temperature. Rinse the test piece immediately in clean running water and then in distilled water; dry rapidly and reweigh (mass m_2, in milligrams, see 5.1).

Use freshly prepared reagent for every test piece.

4.5 Chromate coatings on zinc and cadmium

4.5.1 Reagent

4.5.1.1 Aqueous solution, containing, per litre, 50 g sodium (or potassium) cyanide and 5 g sodium hydroxide.

4.5.2 Procedure

Dry the test piece (area A, in square centimetres, see 5.1), aged naturally after application of the chromate coating for at least 24 h and not more than 14 d, and weigh it, to the nearest 0,1 mg using the analytical balance (mass m_1, in milligrams, see 5.1). Then immerse the test piece for approximately 1 min in the reagent (4.5.1.1) at room temperature and dissolve the coating under electrolytic conditions with the test piece as the cathode.

The anode shall be made from an insoluble material, e.g. graphite.

Immerse the test piece in the reagent, and withdraw it whilst the current is flowing. Use a cathodic current density of 15 A/dm^2. When the coating has been dissolved (after approximately 1 min), withdraw the test piece from the reagent, rinse it immediately in clean running water and then in distilled water, and then dry it rapidly and reweigh (mass m_2, in milligrams, see 5.1).

Use freshly prepared reagent for every test piece.

4.6 Chromate and amorphous phosphate coatings on aluminium and its alloys

4.6.1 Fresh coatings

4.6.1.1 General

Fresh coatings are those dried between 25 °C and 40 °C for at least 3 h and not longer than 5 h.

4.6.1.2 Reagent

4.6.1.2.1 nitric acid, diluted 1 + 1, prepared by adding 1 volume of nitric acid of mass fraction 65 % to 70 % to 1 volume of water.

4.6.1.3 Procedure

Air-dry the test piece (area A, in square centimetres, see 5.1) and weigh it, to the nearest 0,1 mg, using the analytical balance (2.2) (mass m_1, in milligrams, see 5.1). Then immerse the test piece for 1 min in the reagent (4.6.1.2.1) at room temperature. Rinse the test piece immediately in clean running water and then in distilled water; dry rapidly and reweigh (mass m_2, in milligrams, see 5.1).

Use freshly prepared reagent for every test piece.

4.6.2 Aged coatings

CAUTION — When using this method, wear a visor and protective clothing. When melting the reagent, keep away from the bath until the top crust has melted, as the reagent may spatter. Avoid all contact of the reagent with organic matter as such mixtures can be explosive.

4.6.2.1 Reagent

4.6.2.1.1 Mixture, of 98 parts by mass of solid sodium nitrate and 2 parts by mass of solid sodium hydroxide.

4.6.2.2 Procedure

Place the reagent (4.6.2.1.1) in a vessel of a resistant material, e.g. nickel, and heat slowly, from the bottom and sides of the vessel, until the mixture has completely melted.

Dry the test piece (area A, in square centimetres, see 5.1), and weigh it, to the nearest 0,1 mg, using the analytical balance (2.2) (mass m_1, in milligrams, see 5.1). Then immerse the test piece in the molten reagent for 2 min to 5 min at a minimum temperature of 370 °C. A temperature of 370 °C may be adequate for certain coatings but, in general, increasing the temperature to 500 °C will ensure complete stripping of the coating in all cases. When using higher stripping temperatures, it is desirable to determine any loss of mass due to attack on the basis aluminium or its alloy by running a blank determination on an uncoated test piece and deducting this figure from the mass loss obtained on the coated test piece.

Rinse the test piece in clean running water. **BEWARE OF THE RISK OF SPATTERING.**

Immerse the test piece in the diluted nitric acid solution (4.6.1.2.1) for 15 s to 30 s at room temperature. Rinse the test piece immediately in clean running water and then in distilled water; dry rapidly and reweigh (mass m_2, in milligrams, see 5.1).

5 Expression of results

5.1 Calculation

The mass per unit of surface area, m_A, in grams per square metre, shall be calculated from the following equation:

$$m_A = \frac{m_1 - m_2}{A} \times 10$$

where

m_1 is the mass, in milligrams, of the coated test pieces;

m_2 is the mass, in milligrams, of the test piece after the coating has been dissolved;

A is the area, in square centimetres, of the coated surface of the test piece.

If the determinations have been carried out in duplicate or triplicate, the mean shall be reported.

5.2 Measurement uncertainty

The uncertainty of the methods depends on the accuracy in measuring the total surface area and in weighing the test pieces, i.e. on the possibility of carrying out the determinations on total surface areas that are large enough with respect to the mass of the coatings. Under optimum conditions, the measurement uncertainty of the methods is within 5 %.

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