International Standard 3892

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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 par unité de surface — Méthodes gravimétriques

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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.

International Standard ISO 3892 was developed by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the member bodies in August 1977.

It has been approved by the member bodies of the following countries :

Australia	India	Romania
Austria	Israel	Spain
Brazil	Italy	Sweden
Czechoslovakia	Japan	Turkey
France	Mexico	United Kingdom
Germany, F. R.	Netherlands	USA
Hungary	Poland	USSR

The member bodies of the following countries expressed disapproval of the document on technical grounds :

South Africa, Rep. of
Switzerland

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

1 Scope and field of application

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 which are free from any supplementary coating such as oil, water- or solvent-based polymers, or wax.

2 Apparatus

Ordinary laboratory apparatus and

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 a precision of 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, in the case of 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 test, with adequate sensitivity, conformity with the requirements of the relevant material or product specification.

In order to achieve an adequate accuracy in the determination, the total surface area shall be in conformity with the following table :

Table — Total surface areas of test pieces

Expected mass of coating per unit area	Minimum total surface area of test piece
g/m^2	cm^2
less than 1	400
1 to 10	200
over 10 to 25	100
over 25 to 50	50
over 50	25

In order to achieve an overall precision (see 5.2) of 5 %, the surface areas should be measured to an accuracy of 1 %.

4 Reagents and procedures

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

If a sufficient number of test pieces is available, carry out each determination in duplicate or, better, in triplicate.

4.1 Phosphate coatings on iron and steel

4.1.1 Manganese phosphate coatings

4.1.1.1 Reagent

An aqueous solution containing 50 g of chromium(VI) oxide (CrO_3) per litre.

4.1.1.2 Procedure

Dry the test piece (area A) and weigh it on the analytical balance (mass m_1, in milligrams), to the nearest 0,1 mg. Then immerse the test piece for 15 min in the reagent (4.1.1.1), main-

tained at 75 ± 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 sensibly constant mass is obtained (mass m_2, in milligrams).

Use fresh reagent for every test piece.

4.1.2 Zinc phosphate coatings

4.1.2.1 Reagent

An aqueous solution containing 100 g of sodium hydroxide, 90 g of EDTA tetrasodium salt (ethylenedinitrilo tetraacetic acid, tetrasodium salt dihydrate) and 4 g of triethanolamine per litre.

4.1.2.2 Procedure

Dry the test piece (area A), and weigh it on the analytical balance (mass m_1, in milligrams), to the nearest 0,1 mg. Then immerse the test piece for 5 min in the reagent (4.1.2.1), maintained at 70 ± 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).

Use fresh reagent for every test piece.

4.1.3 Iron phosphate coatings

4.1.3.1 Reagent

An aqueous solution containing 50 g of chromium(VI) oxide (CrO_3) per litre.

4.1.3.2 Procedure

Dry the test piece (area A), and weigh it on the analytical balance (mass m_1, in milligrams), to the nearest 0,1 mg. Then immerse the test piece for 15 min in the reagent (4.1.3.1), maintained at 75 ± 5 °C. Rinse the test piece immediately in clean running water and then in distilled water, dry and reweigh. Repeat the procedure until a sensibly constant mass is obtained (mass m_2, in milligrams).

Use fresh reagent for every test piece.

4.2 Phosphate coatings on zinc and cadmium

4.2.1 Reagent

A solution containing 20 g of ammonium dichromate per litre of 25 to 30 % (m/m) ammonia solution. During the preparation of the solution, its temperature shall not exceed 25 °C.

4.2.2 Procedure

Dry the test piece (area A), and weigh it on the analytical balance (mass m_1, in milligrams), to the nearest 0,1 mg. Then immerse the test piece for 3 to 5 min in the reagent (4.2.1) 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).

Use fresh reagent for every test piece.

4.3 Crystalline phosphate coatings on aluminium and its alloys

4.3.1 Reagent

Nitric acid, 65 to 70 % (m/m) solution.

4.3.2 Procedure

Dry the test piece (area A), and weigh it on the analytical balance (mass m_1, in milligrams), to the nearest 0,1 mg. Then immerse the test piece either for 5 min in the reagent (4.3.1) maintained at 75 ± 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).

Use fresh reagent for every test piece.

4.4 Chromate coatings on zinc and cadmium

4.4.1 Reagent

An aqueous solution containing 50 g of sodium (or potassium) cyanide and 5 g of sodium hydroxide per litre.

4.4.2 Procedure

Dry the test piece (area A), aged naturally after application of the chromate coating for at least 24 h and not more than 14 days, and weigh it on the analytical balance (mass m_1, in milligrams), to the nearest 0,1 mg. Then immerse the test piece for approximately 1 min in the reagent (4.4.1) 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, for instance graphite. Immerse the test piece in the reagent, and withdraw it, while 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).

Use fresh reagent for every test piece.

4.5 Chromate and amorphous phosphate coatings on aluminium and its alloys

4.5.1 Fresh coatings (aged not longer than 3 h) dried below 70 °C.

4.5.1.1 Reagent

A solution containing 1 part by volume of 65 to 70 % (m/m) nitric acid solution, and 1 part by volume of water.

4.5.1.2 Procedure

Air-dry the test piece (area A) and weigh on the analytical balance (mass m_1, in milligrams), to the nearest 0,1 mg, within a period of 3 h following the application of the chromate coating. Then immerse the test piece for 1 min in the reagent (4.5.1.1) 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).

Use fresh reagent for every test piece.

4.5.2 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 is melted, as the reagent may spatter. Avoid all contact of the reagent with organic matter as such mixtures can be explosive.

4.5.2.1 Reagent

A mixture of 98 parts by mass of solid sodium nitrate and 2 parts by mass of solid sodium hydroxide.

4.5.2.2 Procedure

Place the reagent (4.5.2.1) in a vessel of a resistant material, for instance nickel, and heat slowly, from the bottom and sides of the vessel, until the mixture is completely melted.

Dry the test piece (area A), and weigh it on the analytical balance (mass m_1, in milligrams), to the nearest 0,1 mg. Then immerse the test piece in the molten reagent for 2 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 (**caution - risk of spattering**), then immerse it in the nitric acid solution (4.5.1.1) for 15 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).

5 Expression of results

5.1 Calculation

The mass per unit of surface area, m_A, expressed in grams per square metre, is given by the formula

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

where

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

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 Precision

The precision 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 large enough in relation to the mass of the coatings. Under optimum conditions, the precision of the methods will be within 5 %.

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