BRITISH STANDARD

BS EN ISO
3892 : 1995

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

The European Standard EN ISO 3892 : 1994 has the status of a British Standard

STANDARDS

Amendment No. 1
published and effective from 15 April 1995
to BS 5411 : Part 14 : 1982

Methods of test for metallic and related coatings
Part 14. Gravimetric method for determination of coating mass per unit area of conversion coatings on metallic materials

NOTE. The European Committee for Standardization (CEN) has accepted ISO 3892 : 1980 as a European Standard designated as EN ISO 3892 : 1994. This amendment implements EN ISO 3892 : 1994 as a British Standard in the BS EN series.

Implementation of European Standard

Front cover

Delete the existing outside front cover and substitute the attached new cover page.

AMD 8497/April 1995

National foreword

At the end of paragraph 1 insert the following new paragraph.

'In 1994 the European Committee for Standardization (CEN) accepted ISO 3892 : 1980 as European Standard EN ISO 3892 : 1994. As a consequence of implementing the European Standard this British Standard is renumbered as BS EN ISO 3892 and any reference to BS 5411 : Part 14 should be read as a reference to BS EN ISO 3892.'

AMD 8497/April 1995

New EN title page and foreword

Immediately after the national foreword insert the attached new EN title page and foreword page.

AMD 8497/April 1995

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 3892

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Descriptors: Non-metallic coatings, conversion coatings, chromate coatings, phosphate coatings, oxide coatings, anodic coatings, physical tests, density measurement, specific surface

English version

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

(ISO 3892 : 1980)

Couches de conversion sur matériaux métalliques — Détermination de la masse par unité de surface — Méthodes gravimétriques
(ISO 3892 : 1980)

Konversionsschichten auf metallischen Werkstoffen — Bestimmung der Masse er Schichten pro Flächeneinheit — Gravimetrische Verfahren
(ISO 3892 : 1980)

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

This European Standard was taken over by the Technical Committee CEN/TC 262, Protection of metallic materials against corrosion, from the work of ISO/TC 107, Metallic and other inorganic coatings, of the International Organization for Standardization (ISO).

CEN/TC 262 has decided to submit the final draft for formal vote. The result was positive.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 1995, and conflicting national standards shall be withdrawn at the latest by April 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

BSI

BS 5411 : Part 14 : 1982
ISO 3892-1980

UDC 669.058 : 531.751.3

Methods of test for

Metallic and related coatings

Part 14. Gravimetric method for determination of coating mass per unit area of conversion coatings on metallic materials

[ISO title : Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods]

Méthodes d'essai des revêtements métalliques et des revêtements apparentés
Partie 14. Méthode gravimétrique de détermination de la masse par unité de surface des couches de conversion sur matériaux métalliques

Prüfmethoden für metallische und ähnliche Beschichtungen
Teil 14. Gravimetrisches Verfahren zur Bestimmung des Beschichtungsgewichts pro Flächeneinheit der Umformungs-Beschichtungen auf metallischen Stoffen

British Standards Institution

British Standard Methods of test for

Metallic and related coatings

Part 14. Gravimetric method for determination of coating mass per unit area of conversion coatings on metallic materials

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 %.

BS 5411 : Part 14 : 1982
ISO 3892-1980

This British Standard, having been prepared under the direction of the Surface Coatings (other than Paints) Standards Committee, was published under the authority of the Board of BSI and comes into effect on 26 February 1982.

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Contract requirements
Attention is drawn to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

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The following BSI references relate to the work on this standard:
Committee reference SRC/14 Draft for comment 76/50826 DC

Cooperating organizations

The Surface Coatings (other than Paints) Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following:

*Aluminium Federation
Assay Offices Committee of Great Britain
*Association of Metal Sprayers
*British Jewellers' Association
British Jewellery and Giftware Federation Ltd.
British Metal Finishing Suppliers' Association
British Steel Industry
Chemical Industries Association
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Engineering Equipment Users' Association
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*Metal Finishing Association
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Motor Cycle Association of Great Britain
*Society of Motor Manufacturers and Traders Limited
Vitreous Enamel Development Council
Zinc Development Association

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the Technical Committee entrusted with the preparation of this British Standard:

Association of Manufacturers of Domestic Electrical Appliances
British Anodising Association
British Industrial Fasteners Federation
City of London Polytechnic
Department of Industry (National Physical Laboratory)
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