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# *Indian Standard*

# METHOD FOR DETERMINATION OF COATING MASS PER UNIT AREA OF CONVERSION COATINGS ON METALLIC MATERIALS BY GRAVIMETRIC METHOD

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# *Indian Standard*

# METHOD FOR DETERMINATION OF COATING MASS PER UNIT AREA OF CONVERSION COATINGS ON METALLIC MATERIALS BY GRAVIMETRIC METHOD

## 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 4 June 1981, after the draft finalized by the Metallic Finishes Sectional Committee had been approved by the Structural and Metals Division Council.

**0.2** Conversion coatings are applied on basis metals such as zinc, cadmium, aluminium, and iron and steel in order to retard formation of corrosion products on the exposed surface of the coatings, such as phosphate coatings on iron and steel or on zinc and cadmium; and chromate coatings on zinc, cadmium, and aluminium and its alloys. Need was felt to prepare a comprehensive standard relating to method of determination of mass of such conversion coatings on metallic materials.

**0.3** While preparing the standard, necessary assistance has been derived from ISO 3892-1979 Conversion coatings on metallic materials — Determination of coating mass per unit area -- Gravimetric method, issued by the International Organization for Standardization.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

## 1. SCOPE

**1.1** This standard specifies gravimetric method for determining the coating mass per unit area of conversion coatings on metallic materials.

**1.2** 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.

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*Rules for rounding off numerical values ( *revised* ).

## 2. APPARATUS

**2.1 Vessel** — Of glass or other appropriate material, in which the conversion coatings may 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

**3.1** 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, in conformity with the requirements of the relevant specification.

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

| *Expected Mass of Coating Per Unit Area*, g/m$^2$ | *Minimum Total Surface Area of Test Piece*, 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* **10.2** ) of 5 percent, the surface area should be measured to an accuracy of one percent.

## 4. QUALITY OF REAGENTS AND NUMBER OF TEST PIECES

**4.1** For the preparation of solutions, use only reagents of recognized analytical grade and only distilled water ( IS : 1070-1977* ) or water of equivalent purity.

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

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*Specification for water for general laboratory use ( *first revision* ).

## 5. PHOSPHATE COATINGS ON IRON AND STEEL

### 5.1 Manganese Phosphate Coatings

**5.1.1** *Reagent* — An aqueous solution containing 50 g of chromium (VI) trioxide ( $CrO_3$ ) per litre.

**5.1.2** *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 minutes in the solution, maintained 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 solution for every test piece.

### 5.2 Zinc Phosphate Coatings

**5.2.1** *Reagent* — An aqueous solution containing 100 g of sodium hydroxide, 90 g of EDTA tetrasodium salt (ethylenedinitrilo tetra-acetic acid, tetrasodium salt dihydrate ), and 4 g of triethanolamine per litre.

**5.2.2** *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 minutes in the reagent 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.

### 5.3 Iron Phosphate Coatings

**5.3.1** *Reagent* — An aqueous solution containing 50 g of chromium (VI) trioxide ( $CrO_3$ ) per litre.

**5.3.2** *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 minutes in the reagent 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.

## 6. PHOSPHATE COATINGS ON ZINC AND CADMIUM

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

**6.2 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 minutes in the reagent 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.

## 7. CRYSTALLINE PHOSPHATE COATINGS ON ALUMINIUM AND ITS ALLOYS

**7.1 Reagent** — Nitric acid, 65 to 70 percent ( *m*/*m* ) solution.

**7.2 Procedure** — Dry the test piece ( Area *A* ) and weigh it on the analytical balanee ( mass $m_1$, in milligrams ) to the nearest 0·1 mg. Then immerse the test piece either for 5 minutes in the reagent maintained at 75±5°C or for 15 minutes 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.

## 8. CHROMATE COATINGS ON ZINC AND CADMIUM

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

**8.2 Procedure** — Weigh the test piece ( Area *A* ), aged naturally after application of the chromate coating for at least 24 hour 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 minute in the reagent 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 cathode current density of 1 500 A/m$^2$. When the coating has been dissolved ( after approximately one minute ), 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.

## 9. CHROMATE AND AMORPHOUS PHOSPHATE COATINGS ON ALUMINIUM AND ITS ALLOYS

**9.1** Fresh coating ( aged not more than three hours ) dried below 70°C.

**9.1.1** *Reagent* — A solution containing one part by volume of 65 to 70 percent ( *m*/*m* ) nitric acid solution and one part by volume of water.

**9.1.2** *Procedure* — Air-dry the test piece ( Area $A$ ) and weigh it on the analytical balance ( mass $m_1$, in milligrams ) to the nearest 0·1 mg within a period of three hours following the application of the chromate coating. Then immerse the test piece for one minute in the 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.

### 9.2 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 may be explosive.

**9.2.1** *Reagent* — A mixture of 98 parts by mass of solid sodium nitrate and two parts by mass of solid sodium hydroxide.

**9.2.2** *Procedure* — Place the reagent 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.

**9.2.2.1** 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 two to five minutes 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 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 nitric acid solution ( *see* **9.1.1**) for 15 to 30 seconds 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 ).

## 10. EXPRESSION OF RESULTS

**10.1 Calculation** — The mass per unit 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

$m_1$ = mass in mg, of the coated test piece;

$m_2$ = mass in mg, of the test piece after the coating has been dissolved; and

$A$ = area, in cm$^2$, of the coated surface of the test piece.

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

**10.2 Precision** — The precision of the methods depends on the accuracy in measuring the total surface area and in weighing the test pieces, that is, on the possibility of carrying out the determination 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 five percent.