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Chemical conversion coatings — Rinsed and non-rinsed chromate conversion coatings on aluminium and aluminium alloys

Couches de conversion chimique — Couches de conversion au chromate rincées et non rincées sur aluminium et alliages d'aluminium

Reference number
ISO 10546:1993(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 10546 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Sub-Committee SC 8, *Chemical conversion coatings*.

Annexes A and B of this International Standard are for information only.

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Chemical conversion coatings — Rinsed and non-rinsed chromate conversion coatings on aluminium and aluminium alloys

1 Scope

This International Standard specifies requirements relating to rinsed and non-rinsed chromate conversion coatings on aluminium and aluminium alloys intended to give protection against corrosion and as a base for other coatings.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2409:1992, *Paints and varnishes — Cross-cut test.*

ISO 3892:1980, *Conversion coatings on metallic materials — Determination of coating mass per unit area — Gravimetric methods.*

ISO 4519:1980, *Electrodeposited metallic coatings and related finishes — Sampling procedures for inspection by attributes.*

ISO 9227:1990, *Corrosion tests in artificial atmospheres — Salt spray tests.*

IEC 68-2-3:1969, *Environmental testing — Part 2: Tests — Test Ca: Damp heat, steady state.*

IEC 130-1:1988, *Connectors for frequencies below 3 MHz — Part 1: General requirements and measuring methods.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 rinsed chromate coating: Chromate coating that is rinsed in water prior to drying. This type of coating is typically applied to extruded aluminium fabricated parts, castings and long coils.

3.2 non-rinsed chromate coating: Chromate coating that is dried immediately after the chromating step without receiving a water rinse. This special type of coating is normally used on long coils of aluminium sheet stock that receive an immediate subsequent paint or adhesive coating, sometimes referred to as coil coating.

4 Surface preparation

The surfaces of the parts to be chromated shall be clean and substantially free of any oxidation, scale or soils such as metal turnings, grinding dust, oil, grease, lubricants, hand-sweat or any other contamination that may be detrimental to the final finish. The parts must therefore be cleaned before chromating and if necessary be pickled. Figure B.1 shows the various processing step options. Prior to the non-rinsed chromating process, surfaces must be free of non-reactive soluble salts.

5 Method of application of chromate coatings

Metallic materials other than aluminium should not be treated in the same chromating solution, in order to avoid galvanic corrosion.

Chromate conversion coatings are normally applied by dipping. They may also be applied by inundation, spraying, roller coating or by wipe-on techniques. The application method used should comply with the operating instructions for the chromating process employed. Chromating solutions are usually acidic and contain hexavalent chromium and/or trivalent chromium salts, together with other components which may be varied to affect the appearance and hardness of the film. The colour of the film, and therefore the type of conversion coating, depend on the composition of the chromating solution, but they are also affected by the pH and temperature, the duration of the treatment, the agitation of the solution and the nature and surface condition of the alloy being treated.

In the case of rinsed chromate coatings, a final water rinse is necessary. If the rinsed chromate treatment is meant to serve as a base for additional coatings, the surface shall be rinsed in pure water with conductibility less than 100 µS/cm. If hot water is used as the final rinse after the chromating process, it is essential that the time of rinsing should be kept as short as possible, in order to prevent the dissolution of the hexavalent chromium. The drying temperature (measured on the surface of the parts) shall not exceed 65 °C for the chromate coating and 85 °C for the phosphochromate coating, to avoid excessive dehydration.

The drying of a non-rinsed chromate coating should be performed according to the manufacturer's instructions.

If the chromate coating is to act as a primer for a lacquer coating, drying before the application of lacquer shall be carried out in two steps; a predrying not exceeding 65 °C and a second drying at 100 °C to 110 °C, in order to achieve the best adhesion.

NOTE 1 Heat treatment at 100 °C to 110 °C interferes with the corrosion resistance on non-lacquered areas.

6 Coating requirements

6.1 General

Chromate conversion coatings harden with age by gradual dehydration. They should, therefore, be handled carefully for the first 24 h after treatment, and any tests (including corrosion tests) shall be deferred until the expiry of that period.

The green chromate-phosphate coatings achieve their maximum corrosion resistance after a longer storage period, usually 1 month to 2 months at room temperature.

6.2 Electrical insulation resistance

Colourless, light yellow or light green iridescent chromate layers, of low mass per unit area, increase the electrical resistance between an electrical contact and the aluminium to a very small extent. When measured at 9 V open circuit voltage and 2 A current according to IEC 130-1, the resistance should be less than 0,1 Ω.

Highly coloured brown, yellow or green coatings show a marked increase in electrical contact resistance, with increasing mass per unit area of the chromate layer.

6.3 Adhesion

The coatings shall be adherent and non-powdery. There are no practical tests for measuring the adhesion of a chromate conversion coating on aluminium. However, a practical evaluation of the adhesion can be made by measuring the adhesion of a secondary organic film applied to the chromated aluminium. When specified, the chromate conversion coating shall pass the organic coating adhesion test specified in ISO 2409.

6.4 Corrosion resistance

When subjected to the neutral salt spray test specified in ISO 9227, three separate test specimens of the coating shall withstand exposure for the duration shown in table 1, without giving evidence of more than a total of eight isolated spots or pits to the unaided eye. None shall be larger than 1 mm in diameter. Each individual test specimen shall have not more than five isolated spots or pits, none larger than 1 mm in diameter, on their respective surfaces. Spots within 10 mm of the edges of the test specimens are not counted. As an alternative method of testing corrosion resistance, test D of IEC 68-2-3 is applicable.

6.5 Coating mass per unit area

The mass of the coating per unit area of coated surface shall conform to the values given in table 2.

The procedure specified in ISO 3892 shall be used.

6.5.1 Coating mass significance

It should be recognized that heavier coating masses do not always provide better performance, particularly when using the coating for an organic film or adhesive base.

6.6 Coating identification

The presence of chromium in conversion coatings and the identification of the appropriate class can be confirmed by their visual appearance and by the tests described in annex A.

Table 1 — Relative corrosion resistance

Coating class[1)]	Exposure time[2)], h		Cast alloys with a nominal silicon content greater than 1 %
	Non-heat-treatable wrought alloys	Heat-treatable alloys and cast alloys with a nominal silicon content less than or equal to 1 %	
1	500	336	48
2	250	168	24
3	168	120	12
4	500	336	48

1) Class numbers are assigned on the basis of traditional usage and do not reflect a ranking of corrosion resistance. The specified exposure time is only effective for a chromated surface without additional coatings. The test gives no information on the performance of a surface which is additionally coated with organic film.

2) The exposure times are indicative of the relative corrosion resistance of the various coating classes on the different alloys, but no direct relationship exists between the performance in the neutral salt spray test and performance in service.

Table 2 — Classification of chromate coatings

Class	Appearance	Coating mass per unit area g/m^2	Corrosion protection
1	Yellow to brown	1,3 to 3	Maximum corrosion resistance, generally used as final finish
2	Yellow (light to iridescent)	0,2 to 1,3	Moderate corrosion resistance, used as a paint base and for bonding to rubber
3	Colourless	0,05 to 0,2	Decorative, slight corrosion resistance, low insulation resistance
4	Green	2 to 5	Moderate corrosion resistance, used as a paint base and for bonding to rubber
5	Light green	0,2 to 2	
6	Colourless	0,05 to 0,2	

NOTE — No relationship exists between class number and degree of corrosion resistance. Class numbers have been assigned on the basis of traditional usage and for compatibility with national standards. The coating mass should be measured as described in ISO 3892.

6.6.1 Chromate coatings (classes 1 to 3)

The determination of the presence of chromium and the absence of phosphate in the coating will identify the layer as a chromate conversion coating. The test described in A.2 and A.3 shall be used.

6.6.2 Chromate-phosphate coatings (classes 4 to 6)

It is necessary to determine the presence of phosphate and chromium and the absence of zinc, in order to distinguish chromate-phosphate coatings from anodized coatings or zinc-phosphate coatings. The test described in A.2 and A.3 shall be used.

7 Sampling and test specimens

Unless otherwise specified, the sampling plans of ISO 4519 shall be used to test the coatings.

The test specimens should be of the same alloy and surface condition as the article they represent. They should measure 100 mm × 150 mm.

8 Classification

The chromate finishes applied can range from brown, thick coatings providing maximum corrosion protection, to yellow, intermediate thickness coatings suitable for organic film bases, or to colourless, thin coatings suitable for lowest insulation resistance. The yellow coatings vary from golden yellow to iridescent light yellow. The chromate-phosphate finishes applied can range in colour from green to iridescent light green.

Finishers can seldom guarantee to supply exact shades of colour with chromate conversion coatings. If it is necessary to have exact shades of colour, it is possible to dye chromate coatings, having a coating mass greater than 0,4 g/m^2, to obtain a wide range of colours, but they can only be expected to give an order of added corrosion resistance similar to that provided by the undyed coatings. It should be noted that colour and colour uniformity will vary somewhat between one alloy and another, and from a polished surface to an etched surface. Iridescense and variations in colour density from one area of the surface to another are normal, and should not be considered a sign of poor quality.

The finishes are divided into six classes; their most important characteristics are listed in table 2.

Annex A
(informative)

Qualitative testing of the layer composition

A.1 Reagents

During the test, use only reagents of analytical grade and distilled water or de-ionized water.

A.1.1 Sodium hydroxide (NaOH), approximately 5 % (*m/m*) solution.

A.1.2 Sodium hydroxide (NaOH), approximately 20 % (*m/m*) solution.

A.1.3 Hydrogen peroxide (H_2O_2), approximately 30 % (*m/m*) solution.

A.1.4 Acetic acid (CH_3COOH), approximately 10 % (*m/m*) solution.

A.1.5 Lead nitrate [$Pb(NO_3)_2$], approximately 10 % (*m/m*) solution.

A.1.6 Concentrated nitric acid (HNO_3), approximately 65 % (*m/m*) solution ($\rho_{20} \approx 1,40$ g/cm^3).

A.1.7 Nitric acid (HNO_3), approximately 38 % (*m/m*) solution, made up, for example, by mixing 1 part by volume of concentrated nitric acid (A.1.6) with 1 part by volume of water.

A.1.8 Ammonium molybdate reagent.

Mix 88,5 g of ammonium molybdate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$], 34 ml of aqueous ammonium hydroxide solution with 25 % (*m/m*) of NH_3 and 240 g of ammonium nitrate (A.1.9). Dissolve in water by shaking and dilute to 1 litre.

A.1.9 Ammonium nitrate (NH_4NO_3).

A.1.10 Hydrochloric acid (HCl), approximately 25 % (*m/m*) solution.

A.1.11 Potassium hexacyanoferrate(II) [$K_4Fe(CN)_6$], approximately 5 % (*m/m*) solution.

A.1.12 Sulfuric acid (H_2SO_4), approximately 25 % (*m/m*) solution.

A.1.13 Phenolphthalein ($C_{20}H_{14}O_4$), approximately 0,5 % (*m/m*) solution in ethanol.

A.2 Testing for chromium

A sample of about 300 cm^2 surface is treated with 50 ml of sodium hydroxide solution (A.1.1), to which 5 ml of hydrogen peroxide solution (A.1.3) was added. The solution, at 50 °C to 60 °C, is poured over the sample, repeatedly if necessary, until the layer is completely removed. The resulting solution is poured off, boiled until all the hydrogen peroxide is destroyed (about 5 min to 6 min), cooled and precipitated with the lead nitrate solution (A.1.5).

A yellow precipitate indicates the presence of hexavalent chromium. The smallest quantity of total chromium detectable by this method corresponds to about 5 mg of chromium per square metre of surface.

A.3 Testing for phosphate

To test for the presence of phosphate in the chromated layer, a sample is taken with a surface to be tested of about 100 cm^2. This is treated with 100 ml of sodium hydroxide solution (A.1.1) at 80 °C to 90 °C until the layer is completely dissolved, or at least until the surface has been clearly attacked. The resulting solution is filtered, and 25 ml of the filtrate are acidified with nitric acid solution (A.1.7), then 10 ml of ammonium molybdate reagent (A.1.8) and 5 g of ammonium nitrate (A.1.9) are added. The sample is allowed to stand for at least 15 min.

A yellow precipitate indicates the presence of phosphate. The smallest quantity of phosphate detectable by this process corresponds to about 40 mg of P_2O_5 per square metre of surface.

A.4 Testing for zinc

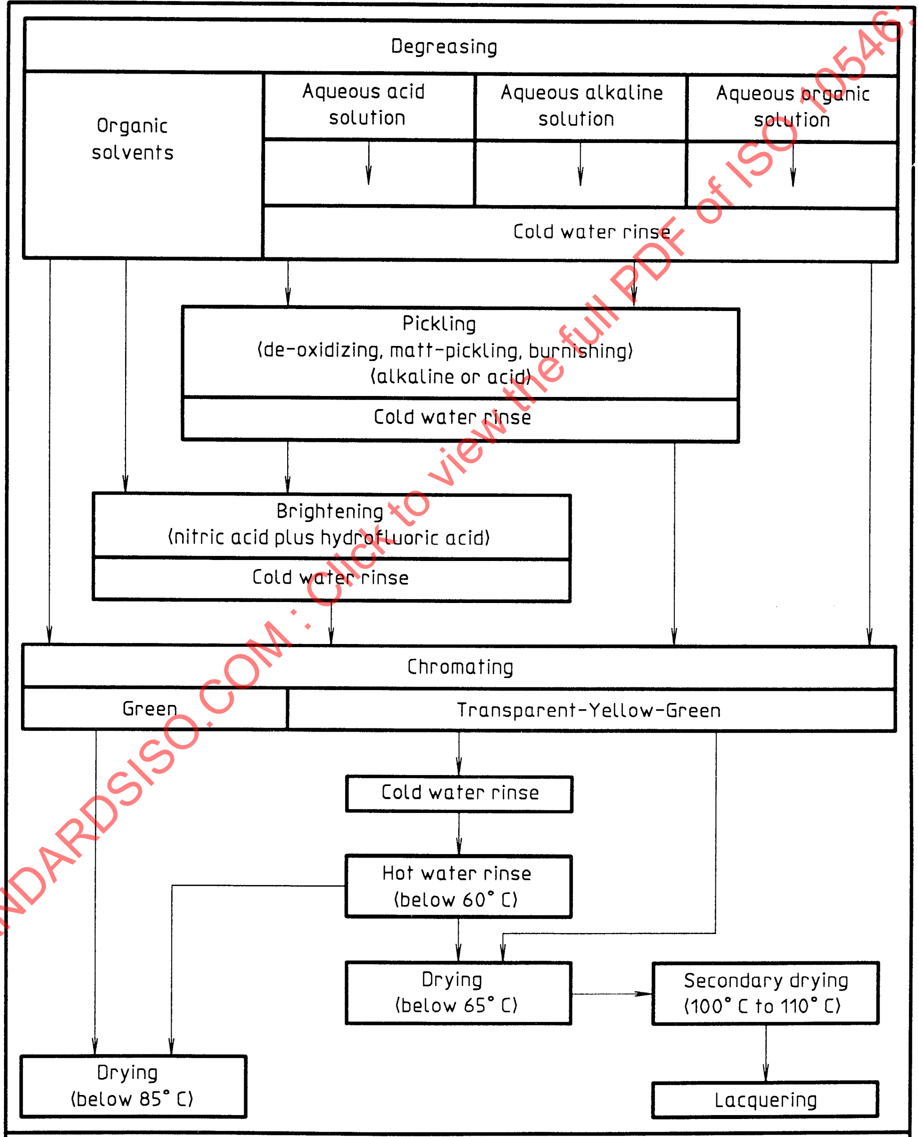
To test for the presence of zinc in the layer, a sample is taken with a surface to be tested of about 100 cm^2. This is treated with 50 ml of nitric acid (A.1.6) at room temperature until the layer is either completely dissolved, or at least until the surface has clearly been attacked. The resulting solution is filtered through glass wool, and 25 ml of the filtrate are neutralized with sodium hydroxide solution (A.1.2) until the phenolphthalein indicator (A.1.13) gives a red colouration. The solution is then weakly acidified by addition of about 10 drops of sulfuric acid solution (A.1.12).

After addition of 5 ml of the potassium hexacyanoferrate(II) solution (A.1.11), a greenish-white precipitate indicates the presence of zinc. The smallest quantity of zinc detectable by this process corresponds to about 20 mg of zinc per square metre of surface.

Annex B
(informative)

Treatment sequence options

The chromate process necessitates a specified sequence of treatment which depends upon the surface condition of the parts to be treated, the aluminium alloy used and the desired appearance of the parts. (See figure B.1.)



Rinsing must be performed carefully in order to avoid splattering of chemicals and corrosion damage.

Figure B.1 — Treatment stages of the chromating of aluminium and aluminium alloys