

Corrosion protection of metals — Rinsed and non-rinsed chromate conversion coatings on aluminium and aluminium alloys

The European Standard EN 12487:2000 has the status of a
British Standard

ICS 25.220.20

National foreword

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The UK participation in its preparation was entrusted to Technical Committee STI/38, Chemical conversion coatings, which has the responsibility to:

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- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 16, an inside back cover and a back cover.

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**Corrosion protection of metals - Rinsed and non-rinsed
chromate conversion coatings on aluminium and aluminium
alloys**

Protection contre la corrosion des métaux - Couches de
conversion au chromate rincées et non rincées sur
l'aluminium et les alliages d'aluminium

Korrosionsschutz von Metallen - Gespülte und no-rinse
Chromatierüberzüge auf Aluminium und
Aluminiumlegierungen

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 262 "Metallic and other inorganic coatings", the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by October 2000, and conflicting national standards shall be withdrawn at the latest by October 2000.

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

1 Scope

This European Standard specifies requirements for 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

This European Standard incorporates by dated and undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

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

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

IEC 60130-1, *Connectors for frequencies below 3 MHz — Part 1: General requirements and measuring methods*.

3 Terms and definitions

For the purposes of this standard, the following terms and definitions apply:

3.1

rinsed chromate coating

chromate coating that is rinsed in water prior to drying

NOTE This type of coating is typically applied to extruded and rolled 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

NOTE This 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 Information to be supplied by the purchaser

The following information shall be supplied by the purchaser:

- a) the coating designation (see clause 5);
- b) sampling methods, acceptance levels or any other inspection requirements, if different from those given in ISO 4519 (see clause 6);
- c) the surface preparation prior to chromate conversion coating (see annex A for guidance);
- d) the requirements for adhesion (see 7.3) and corrosion resistance (see 7.6);
- e) the nature, condition and finish of the basis metal, if any of these could affect the serviceability and/or the appearance of the coating.

5 Coating types and designation

5.1 Coating types

A conversion coating shall be referred to as type A, C1, C2, E1 or E2 in accordance with table 1.

NOTE Annex A provides guidance on coating type, purpose, end use and mass per unit area.

Table 1 — Types of chromate conversion coating

Type	Appearance	Coating mass per unit area (g/m ²)	Corrosion protection	Other properties
A	Colourless	0,05 to 0,2	Slight	Decorative, low insulation resistance
C1	Yellow (light to iridescent)	0,4 to 1,0	Moderate	Used as a paint base and for bonding to rubber
C2	Yellow (to brown)	1,0 to 3	Maximum	Generally used as final finish
E1	Light green	0,4 to 1,2	Moderate	Used as a paint base and for bonding to rubber
E2	Green	2 to 5	Moderate	Generally used as final finish

5.2 Conversion coating designation

The conversion coating designation shall comprise the following:

- a) the number of this European Standard;
- b) a hyphen;
- c) the basis metal code, e.g. its chemical symbol (or that of the principal constituent of an alloy) (see note 1);
- d) a solidus;
- e) a symbol designating the type of coating (see table 1 and note 1).

If the chromate conversion coating has to be after-treated, the designation shall also comprise the following:

- a) a solidus;
- b) a symbol indicating any after-treatment of the conversion coating, as specified in table B.1, annex B (see note 2).

NOTE 1 It is recommended that the chemical symbol is followed by the standard designation of the basis metal.

NOTE 2 This can be repeated if more after-treatments are required.

EXAMPLE

Designation of a chromate conversion coating of coating mass 0,4 g/m² to 1,2 g/m² (E1) on aluminium (Al) that has been after-treated with the application of inorganic or organic sealant (T2)

Chromate conversion coating EN 12487-Al/E1/T2

6 Sampling

Sampling shall either be in accordance with ISO 4519 or as specified by the purchaser (see 4,b)).

7 Coating requirements

7.1 General

Any tests (including corrosion resistance tests) shall be deferred until the expiry of a period of 24 h after treatment.

NOTE Conversion coatings harden with age by gradual dehydration, and they should therefore be handled carefully for the first 24 h after the last treatment.

7.2 Electrical insulation

When measured at an open circuit voltage of 9 V and a current of 2 A in accordance with IEC 60130-1, the resistance between an electrical contact and the aluminium shall be less than 0,1 Ω for type A, C1 and E1 coatings.

NOTE Highly coloured type C2 and E2 coatings show a marked increase in electrical resistance with increasing mass per unit area of the chromate layer.

7.3 Adhesion

7.3.1 The coatings shall be adherent and non-powdery. The conversion coating shall pass the test described in 7.3.2 or 7.3.3.

7.3.2 Rub the dried surface with a white soft tissue paper using normal hand pressure (approximately 10 moves). This treatment shall not leave any trace of staining on the paper.

7.3.3 If the test described in 7.3.2 is not sufficiently adequate to measure the adhesion of a conversion coating on aluminium, another practical evaluation of the adhesion can be made by measuring the adhesion of a secondary organic film applied to the chromated aluminium.

7.4 Coating mass per unit area

For all but thin (type A) coatings, the coating mass per unit area of chromate and chromate-phosphate coatings on aluminium shall be determined in accordance with EN ISO 3892 or with the methods described in C.2 to C.4.

For thin (type A) chromate coatings, the coating mass per unit area shall be determined in accordance with C.3 and C.4.

NOTE For thin chromate coatings, the errors associated with the methods described in EN ISO 3892 are unacceptably high.

7.5 Coating identification

7.5.1 General

The test methods specified in annex D shall be used to determine the presence or the absence of relevant chemical elements in the conversion coating.

7.5.2 Chromate conversion coatings (yellow)

The presence of chromium and the absence of phosphate in the coating shall identify the layer as a chromate conversion coating.

7.5.3 Chromate phosphate coatings (green)

The presence of phosphate and chromium and the absence of zinc shall identify the layer as a chromate-phosphate conversion coating.

7.6 Corrosion resistance

The corrosion resistance of a conversion coating shall be determined in accordance with ISO 9227.

NOTE Requirements for corrosion resistance of conversion coatings vary widely depending on the aluminium alloys to which they are applied. Annex E gives typical figures of corrosion resistance attained under the best possible conditions.

Annex A **(informative)**

Process systems

A.1 Surface preparation

The surfaces of the parts to be chromated should be clean and substantially free from any oxidation, scale or soils such as metal turnings, grinding dust, oil, grease, lubricants, hand-sweat or any other contamination that is detrimental to the final finish. The parts should therefore be cleaned before chromating and if necessary be pickled.

Prior to the non-rinsed chromating process, surfaces should be free from non-reactive soluble salts.

Care should be taken when rinsing in order to avoid splattering of chemicals and corrosion initiation.

Figure A.1 shows processing step options available depending on the preparation and intended end use required.

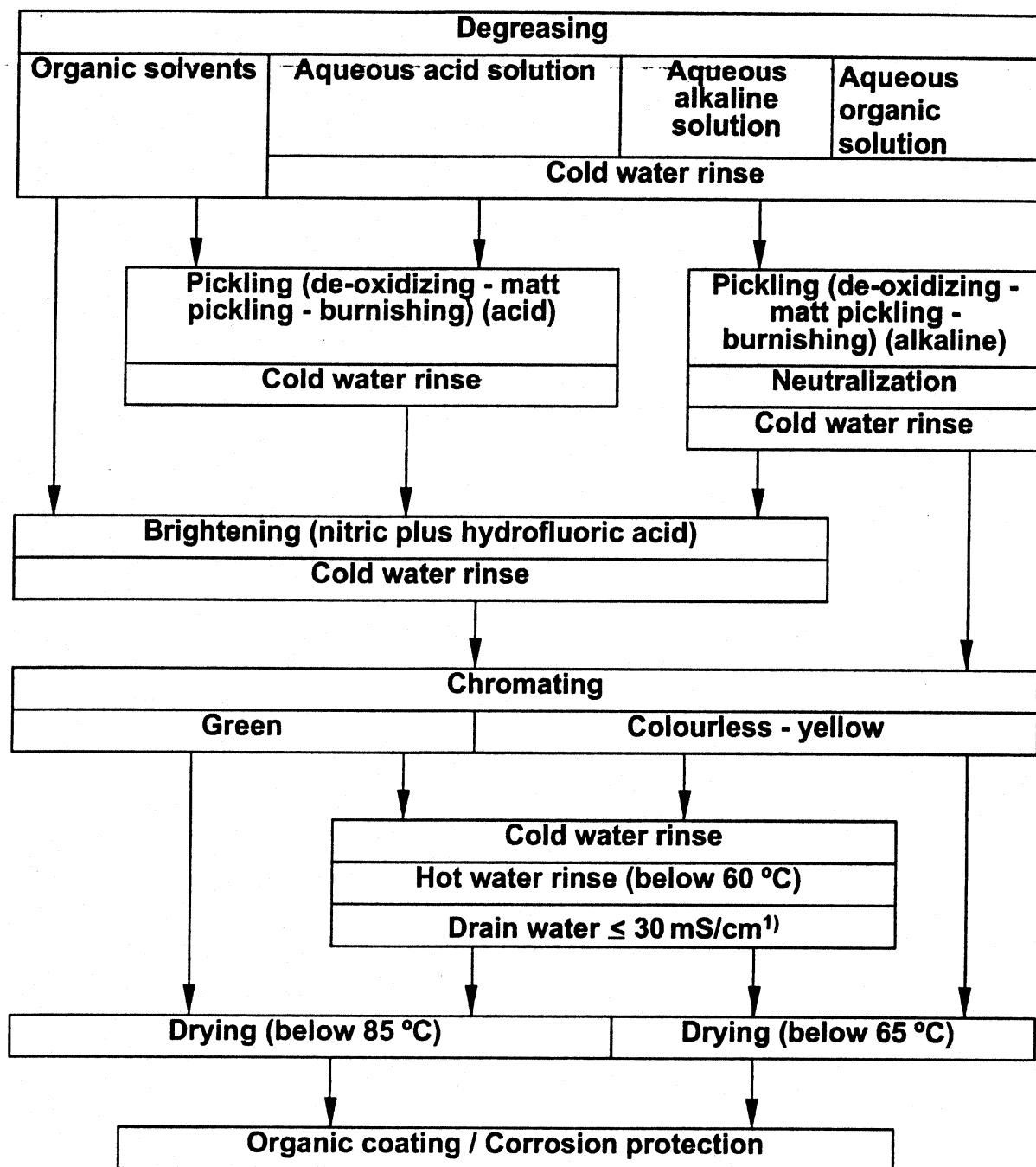


Figure A.1 — Treatment stages of the chromating of aluminium and aluminium alloys

¹⁾ For coatings a final rinse is necessary; for the conductivity of the drain water, see A.2.

A.2 Method of application

Different base metals 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, flooding, spraying, roller coating or by wipe-on techniques. The application method should be in accordance 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, depends on the composition of the chromating solution, but it is also affected by the pH and the 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 rinse is necessary. If the rinsed chromate coating is meant to be the basis for additional coatings, the surface should be rinsed in pure water and with deionized water. The drain water conductivity as measured on the treated surfaces that will receive additional coatings should be maintained below 30 $\mu\text{S}/\text{cm}$ at 20 °C. If hot water is used as the final rinse after the chromating process, the temperature should not exceed 60 °C and the time of rinsing should be kept as short as possible in order to prevent the dissolution of the hexavalent chromium and to prevent cracking due to dehydration, causing loss of adhesion and performance of the conversion coating.

The maximum drying temperature of an after-treatment, as measured on the metal surfaces, should not exceed those temperatures indicated in table A.1.

Table A.1 — Maximum drying temperature of an after-treatment

Delay in performing after-treatment after application of the conversion coating	Maximum drying temperature ^a (°C)
Performed without delay	100
Performed with delay of up to 16 h: —chromate (yellow) conversion coatings —chromate-phosphate (green) conversion coatings	65 85
^a As measured on the metal surface.	

The drying of properly non-rinsed chromate coatings should be performed according to the manufacturers' instructions. Any additional subsequent treatments depend upon the purposes for which the coated parts are intended.

A.3 Types of coatings

Chromate finishes can be applied ranging in colour from dark yellow (brown), thick coatings providing maximum corrosion protection, to yellow, intermediate thickness coatings suitable as an organic film base, or to colourless, thin coatings suitable for lowest insulation resistance. The yellow coatings vary from golden yellow to iridescent light yellow. Chromate-phosphate finishes can be applied ranging in colour from green to iridescent light green.

Finishers can seldom guarantee to supply shades of colour with chromate conversion coatings. If it is essential to have exact shades of colour, it is possible to dye a chromate coating having a coating mass greater than 0,4 g/m², 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. Iridescence 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.

Annex B **(normative)**

After-treatments

Table B.1 gives the symbols that shall be used by the purchaser when specifying the after-treatments.

Table B.1 — After-treatments

Symbol	Type of treatment
T1	Application of paints, varnishes or similar coatings materials
T2	Application of inorganic or organic sealants

Annex C **(normative)**

Analytical methods for the determination of the coating mass and for the identification of chromium, phosphorus and zinc

C.1 Coating mass

The method for the determination of the coating mass of chromate and chromate-phosphate coatings on aluminium is described in EN ISO 3892. It is based on the measurement of mass differences.

C.2 Electron microscopical determination

A scanning electron microscope equipped with an energy dispersing detector (EDS type) makes it possible to detect chromium, phosphorus and zinc on the surface of the test specimen. The depth of the analysis depends on the energy of the incident electrons and on the angle of incidence. The detection limit depends on the apparatus used.

C.3 Photometric determination

In this method the chromate coating is dissolved with a suitable acid and the chromium content in the resulting solution is determined photometrically. For example, the coating is removed from five samples in the form of sheets measuring 100 mm x 100 mm by pickling in about 500 ml of sulfuric acid at boiling point (duration of pickling: 10 min).

After removing the coating from the sheets by pickling, the pickling solution is evaporated down to about 50 ml and the chromium in this solution is determined photometrically as total chromium. To obtain the blank reading, the samples are again pickled by the same method and the determination is repeated.

For a photometric determination, it is sufficient to determine the total chromium content, and for this purpose the trivalent chromium in the solution is oxidized into the hexavalent state, for example, with ammonium peroxodisulfate and can then be determined photometrically at a wavelength of $\lambda = 445$ nm.

Experience shows that removing fresh chromate coatings by pickling using this method presents no problems, but in the case of aged coatings the blank value measured may be relatively high. In that case the pickling process should be repeated and the blank value determined again.

C.4 X-ray fluorescence analysis

The mass of chromium per unit area can also be determined by a suitable physical analytical method such as X-ray fluorescence analysis (RFA). In this method the chromium in the chromate layer is excited by high-energy primary radiation to produce characteristic secondary X-ray fluorescence radiation. The mass of chromium per unit area can be determined from the intensity of this radiation using calibration samples. The limit of detection by the RFA method is about 10 mg/m² to 20 mg/m² chromium for yellow and green chromate conversion coatings on aluminium. The relative uncertainty of measurement of the method is ± 5 %.

Annex D (normative)

Testing of the layer composition

D.1 Reagents

Only analytical grade reagents shall be used. The water used shall be distilled or de-ionized water.

Sodium hydroxide, a solution of NaOH of approximately 5 % mass fraction

Sodium hydroxide, a solution of NaOH of approximately 20 % mass fraction

Hydrogen peroxide, a solution of H₂O₂ of approximately 30 % mass fraction

Acetic acid, a solution of CH₃COOH of approximately 10 % mass fraction

Lead nitrate solution, a solution of Pb(NO₃)₂ of approximately 10 % mass fraction

Nitric acid, a solution of HNO₃ of approximately 65 % mass fraction (density: 1,40 g/cm³ at 20 °C)

Nitric acid, a solution of HNO₃ of approximately 38 % mass fraction made up for instance by mixing 1 part by volume of nitric acid of density 1,40 g/cm³ at 20 °C, with 1 part by volume of water

Ammonium molybdate-reagent: 88,5 g ammonium molybdate (NH₄)₆Mo₇O₂₄·4H₂O, 34 ml aqueous ammonium hydroxide solution with 25 % mass fraction NH₃, and 240 g ammonium nitrate NH₄NO₃ are dissolved in water up to 1 l total.

Ammonium nitrate, NH₄NO₃

Hydrochloric acid, a solution of HCl of approximately 25 % mass fraction

Potassium hexacyanoferrate solution, a solution of K₄Fe(CN)₆ of approximately 5 % mass fraction

Sulfuric acid, a solution of H₂SO₄ of approximately 25 % mass fraction

Phenolphthalein, C₂₀H₁₄O₄, solution of 0,5 % mass fraction in ethanol

D.2 Determination of the presence of chromium in the layer

A sample of about 300 cm² surface is treated with 50 ml of 5 % sodium hydroxide solution, to which 5 ml of 30 % hydrogen peroxide are added. The solution, at 50 °C to 60 °C, is poured over the surface, repeatedly if necessary, until the layer is completely removed. The resulting solution is poured off, boiled until all hydrogen peroxide is destroyed (about 5 min to 6 min), cooled down and precipitated with an approximately 10 % lead nitrate solution. A yellow precipitate shows the presence of hexavalent chromium. The least quantity of total chromium detectable by this method corresponds to about 5 mg chromium per square metre of surface.

D.3 Determination of the presence of phosphate in the layer

A sample of about 100 cm² surface is treated with 100 ml of 5 % sodium hydroxide solution 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 approximately 38 % nitric acid, then 10 ml ammonium molybdate reagent and 5 g ammonium nitrate are added. The sample is allowed to stand for at least 15 min. A yellow precipitate shows the presence of phosphate. The least quantity of phosphate detectable by this method corresponds to about 40 mg P₂O₅ per square metre of surface.

D.4 Determination of the presence of zinc in the layer

A sample of about 100 cm² surface is treated with 50 ml of approximately 65 % nitric acid at room temperature until the layer is 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 of 20 % mass fraction until phenolphthalein indicator gives a red coloration. The solution is then weakly acidified by the addition of about 10 drops of about 25 % mass fraction sulfuric acid. On the subsequent addition of 5 ml of 5 % mass fraction potassium hexacyanoferrate solution, a greenish-white precipitate indicates the presence of zinc. The least quantity of zinc detectable by this method corresponds to about 20 mg zinc per square metre of surface.

Annex E (informative)

Corrosion resistance

The corrosion resistance of conversion coatings varies widely depending on the aluminium alloys to which a coating is applied. Table E.1 gives typical figures of corrosion resistance attained under the best possible conditions.

Table E.1 — Corrosion resistance (neutral salt spray tests)

Coating type	Exposure time		
	h		
	Non-heat-treatable wrought alloys	Heat-treatable alloys and cast alloys with a nominal silicon content < 1%	Cast alloys with a nominal silicon content > 1%
A	168	120	12
C2	168	168	48
E2	500	336	48

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