

Paints and varnishes — Coating of aluminium and aluminium alloys for architectural purposes —

Part 1: Coatings prepared from coating powder

The European Standard EN 12206-1:2004 has the status of a
British Standard

ICS 87.040

National foreword

This British Standard is the official English language version of EN 12206-1:2004.

The UK participation in its preparation was entrusted by Technical Committee STI/27, Paint systems for metallic substrates, to Subcommittee STI/27/1, Organic coatings on aluminium and galvanized steel for architectural purposes, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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Foreword

This document (EN 12206-1:2004) has been prepared by Technical Committee CEN/TC 139 “Paints and varnishes”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by December 2004, and conflicting national standards shall be withdrawn at the latest by December 2004.

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

Annexes A, B, C, D and F are normative, annex E is for information only.

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Introduction

This is the first Part of EN 12206. The present intention is to develop another Part dealing with the organic coating of aluminium and aluminium alloy extrusions, sheet and preformed sections for architectural purposes, prepared from liquid coating materials.

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1 Scope

This Part of EN 12206 specifies requirements and the corresponding methods of test relating to the organic coating of aluminium and aluminium alloy extrusions, sheet and preformed sections for architectural purposes, using coating powders. It also describes:

- a) the pretreatment of the substrate prior to the coating process;
- b) the coating powder;
- c) the coating process;
- d) the final product.

Each item is dealt with separately in this Part of EN 12206 so that any interested party can ensure compliance appropriate to its area of responsibility.

CAUTION — The procedures described in this standard are intended to be carried out by suitably trained and/or supervised personnel. The substances and procedures used in this method may be injurious to health if adequate precautions are not taken. Attention is drawn in the text to specific hazards. This standard refers only to technical suitability and does not absolve the user from statutory obligations relating to health and safety.

2 Normative references

This European Standard incorporates, by dated or 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 573-3, *Aluminium and aluminium alloys – Chemical composition and form of wrought products – Part 3: Chemical composition*.

EN ISO 1519, *Paints and varnishes – Bend test (cylindrical mandrel) (ISO 1519:2002)*.

EN ISO 1520, *Paints and varnishes – Cupping test (ISO 1520:1999)*.

EN ISO 2409, *Paints and varnishes – Cross-cut test (ISO 2409:1992)*.

EN ISO 2813, *Paints and varnishes – Determination of specular gloss of non-metallic paint films at 20°, 60° and 85° (ISO 2813:1994, including Technical Corrigendum 1:1997)*.

EN ISO 3231, *Paints and varnishes – Determination of resistance to humid atmospheres containing sulfur dioxide (ISO 3231:1993)*.

EN ISO 3668, *Paints and varnishes – Visual comparison of the colour of paints (ISO 3668:1998)*.

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)*.

EN ISO 6270-1, *Paints and varnishes – Determination of resistance to humidity – Part 1: Continuous condensation (ISO 6270-1:1998)*.

EN ISO 8130-9, *Coating powders – Part 9: Sampling (ISO 8130-9:1992)*.

EN ISO 8565, *Metals and alloys – Atmospheric corrosion testing – General requirements for field tests (ISO 8565:1992)*.

EN 12206-1:2004 (E)

EN ISO 11341:1997, *Paints and varnishes – Artificial weathering and exposure to artificial radiation – Exposure to filtered xenon-arc radiation (ISO 11341:1994)*.

ISO 2810, *Paints and varnishes – Notes for guidance on the conduct of natural weathering tests*.

ISO 2859-1, *Sampling procedures for inspection by attributes – Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*.

ISO 2859-2, *Sampling procedure for inspection by attributes – Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection*.

ISO 7724-3, *Paints and varnishes – Colorimetry – Part 3: Calculation of colour differences*.

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

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1

powder coating

a continuous layer of a coating powder which has been applied to the aluminium or aluminium alloy substrate and which is protective or decorative, or both

3.2

conversion layer

a layer produced on an aluminium surface by chemical pretreatment with or without the use of an applied electric current

3.3

finish

the surface of the coated article which determines its appearance

3.4

coating powder

a solvent-free coating material in powder form which, after fusing and possible curing, gives a continuous film

[EN 971-1:1996]

3.5

significant surface

that area of the article on which the coating is essential for serviceability and/or appearance

3.6

specifier

the person specifying the performance requirements for the coating and significant surfaces of the article

3.7

test specimen

a single sample of the final product to be used for testing

4 Requirements

4.1 Material (substrate)

Aluminium and aluminium alloys are classified in accordance with EN 573-3. Suitable materials are those listed as series 1000, 3000, 5000 and 6000.

4.2 Pretreatment of the substrate

4.2.1 General

Before application of the coating powder, a pretreatment layer shall be applied. This pretreatment may be either

- a) chemical treatment with aqueous solutions containing either chromate ions or chromate and phosphate ions, without applying an electric current, or
- b) an alternative pretreatment (for instance chromium-free systems, "no-rinse-dry-in-place pretreatment" or electrolytic conversion, or other processes).

After the conversion process, the substrate is normally rinsed with deionized water, and dried.

4.2.2 Conventional pretreatment

4.2.2.1 General

The aluminium or aluminium alloy shall be free from corrosion prior to pretreatment.

Any item previously coated or anodized shall be stripped down to the aluminium substrate prior to the pretreatment.

The sequence of the pretreatment stages is shown in annex E.

The pretreatment stages shall take place one after the other with the minimum delay in between to ensure that the surface to be pretreated does not become dry between successive stages.

The pretreatment solutions shall be applied either:

- a) **by dipping** in baths of sufficient size to allow efficient operation of the chemical process on all significant surfaces ; or
- b) **by spraying**, in tanks or cabinets made so that all significant surfaces are in contact with the spraying solution for the specified period of time.

The solution used for the conversion process is normally used for aluminium and aluminium alloys only. Pretreatment of other metals is not authorized unless removal of metallic-ion contamination is ensured.

4.2.2.2 Cleaning, pickling and rinsing

All surface contaminants such as oils, greases, lubricants and other residues shall be removed using alkaline or acidic solutions and/or organic solvents in appropriate combinations. Before the conversion stage, the substrate shall be thoroughly cleaned and/or pickled.

After cleaning and/or pickling, the substrate may be rinsed (see annex E).

NOTE It is advisable that the rinse water prior to the conversion stage should comply to the following requirements :

- electrical conductivity : max. 1 000 $\mu\text{S}/\text{cm}$;
- chloride content : less than 100 mg/l ;
- sulfate content : less than 50 mg/l ;
- phosphate content : less than 5 mg/l (for chromate conversion only).

4.2.2.3 Conversion and drying of chromate and phosphochromate layers

A conversion layer is produced by application of the appropriate solution.

EN 12206-1:2004 (E)

Chromate conversion layers vary in colour from an iridescent yellow to a golden tan. The layers are characterized by the presence of chromium and the absence of phosphate (see annex A).

The mass per unit surface area of the conversion layer, often referred to as "conversion coating weight", as measured in accordance with the method described in annex A, shall be between 0,4 g/m² and 1 g/m².

Phosphochromate conversion layers vary in colour from iridescent to various shades of green. The layers are characterized by the presence of both chromate and phosphate (see annex A).

The mass per unit surface area of the conversion layer, as measured in accordance with the method described in annex A, shall be between 0,4 g/m² and 1,2 g/m².

NOTE The colours given above are only indicative. They depend on alloys used and pretreatment and do not characterize the quality of the conversion layer.

The conversion layer can be rinsed with water and shall finally be rinsed with deionized water. The electrical conductivity of the water draining off from significant surfaces at the final rinse shall be below 30 µS/cm at 20 °C.

If the final rinse is carried out with hot water, the temperature shall not exceed 60 °C and the rinsing time shall be as short as possible in order to avoid dissolving of hexavalent chromium from the conversion layer.

The layer shall be as uniform as possible, adhering to the substrate and free from any powdering.

The drying temperature after pretreatment, as measured on the metal surface, shall not exceed 100 °C if the coating powder is applied without delay or interruption after pretreatment. If it is applied with delay or interruption after pretreatment (less than 16 h but not more), the maximum temperature on the metal surface shall be as follows:

- 65 °C for chromate layers;
- 85 °C for phosphochromate layers.

The coating powder shall not be applied more than 16 h after pretreatment.

4.2.3 Alternative pretreatments

Alternative pretreatments, e.g. chromium-free systems, "no-rinse-dry-in-place pretreatment" or electrolytic conversion or other processes may be specified but shall comply with the requirements of annex B.

4.3 Coating powder

4.3.1 General

The tests shall be carried out in accordance with clause 5. The panels shall be prepared in accordance with 5.1.

NOTE A coating powder that has been found to be of suitable quality to pass the requirements of this Part of EN 12206 may then be used to confirm the suitability of other pretreatment processes.

4.3.1.1 Coating powders based on conventional technology

Coating powders based on conventional technology shall meet all the criteria in 4.3. Products based on future technology may exhibit different characteristics.

4.3.1.2 Enhanced durability coating powders

Enhanced durability coating powders can be specified. See annex G.

4.3.2 Colour

When tested in accordance with 5.3, the colour of the coating shall match the colour previously specified with agreed limits.

4.3.3 Gloss

When tested in accordance with 5.4, the gloss level of the coating shall be in the range of ± 10 units of the specified value for coatings of gloss greater than 50 units, and in the range of ± 7 units for coatings of gloss equal to or less than 50 units.

4.3.4 Adhesion

When tested in accordance with 5.5, the result of the test shall be classification 0.

4.3.5 Cupping test

When tested in accordance with 5.6, there shall be neither cracking in the coating nor any detachment of the coating.

4.3.6 Falling weight test

When tested in accordance with 5.7, there shall be neither cracking in the coating nor any detachment of the coating.

4.3.7 Bend test

When tested in accordance with 5.8, there shall be neither cracking in the coating nor any detachment of the coating.

4.3.8 Resistance to mortar

When tested in accordance with 5.9, the mortar shall be easily removed and there shall be no detachment of the coating nor any staining.

4.3.9 Pressure cooker test

When tested in accordance with 5.10, there shall be neither blistering nor detachment of the coating.

4.3.10 Resistance to humidity

When tested in accordance with 5.11, there shall be no blistering, softening or detachment of the coating. Loss of adhesion of the coating from the scratches shall be no more than 1 mm.

4.3.11 Resistance to acetic acid salt spray

When tested in accordance with 5.12, the coating shall show neither blistering nor more than 3 mm of corrosion from the scratches.

4.3.12 Resistance to sulfur dioxide

When tested in accordance with 5.13, the coating shall show no more than 1 mm of corrosion from the scratches, and no visual colour change.

4.3.13 Resistance to filiform corrosion

No requirements for resistance to filiform corrosion are laid down in this Part of EN 12206 as yet.

A test method is described in 5.14.

4.3.14 Resistance to weathering

For testing resistance to weathering, two test methods are provided, artificial weathering (5.15) and natural weathering (5.16).

Natural weathering (5.16) provides the best assessment and is the referee method. However, it takes a relatively long time and until the results from natural weathering are available, results from artificial weathering can provide useful guidance.

After weathering in accordance with 5.15 or 5.16, the residual gloss of the coating shall be not less than 50 % of the original gloss. Acceptable limits for colour change (e.g. in terms of ΔE and/or ΔC and/or ΔL) shall be agreed between the interested parties.

4.4 Coating process

All the workers handling the pretreated but uncoated items shall wear clean gloves in order to avoid contamination of the surfaces. If not immediately coated, the pretreated parts shall be stocked and handled in covered, dry and clean areas.

The application of the coating powder shall take place in a suitable booth. During the coating process, continued care shall be exercised to ensure that contamination of the pretreated metal surface by dust, grease, electrolytic residues etc. are excluded. Touching of the significant surface with unprotected hands shall be avoided. After coating, the articles shall be transferred to an oven and stoved at the conditions (metal temperature and time) recommended by the supplier of the coating powder.

After stoving, the coated parts shall be allowed to cool to room temperature before handling.

4.5 Final product

Tests on final products are performed on test specimens prepared in accordance with 5.2.

4.5.1 Significant surface

The significant surface (3.6) shall be indicated by the specifier by the use of reference samples or technical drawings.

4.5.2 Surface appearance

When the coating on significant surfaces is illuminated and examined at an oblique angle with normal or corrected vision, no blisters, craters, pinholes or scratches shall be visible from a distance of 3 m (products for inside use) or 5 m (products for outside use). The coating shall be free from defects extending down to the substrate.

4.5.3 Colour

When tested in accordance with 5.3, the coating on each significant surface shall match the colour previously specified.

4.5.4 Gloss

When tested in accordance with 5.4, the gloss of the coating shall be within 10 units of the specified value for coatings of gloss greater than 50 units and within 7 units for coatings of gloss equal to or less than 50 units.

4.5.5 Film thickness of the coating

When tested in accordance with annex C, the average thickness shall be at least 50 μm . No value below 40 μm is allowed.

NOTE The control of the film thickness is technically difficult and is largely dependent on the geometry of the part to be coated. Therefore, it may not always be possible to ensure any given maximum film thickness whilst maintaining a minimum film thickness of 40 μm .

4.5.6 Adhesion

When tested in accordance with 5.5, the result of the test shall be classification 0.

4.5.7 Falling weight test

When tested in accordance with 5.7, there shall be neither cracks in the coating nor any detachment of the coating.

4.5.8 Pressure cooker test

When tested in accordance with 5.10, there shall be neither blistering nor detachment of the coating.

4.5.9 Further relevant requirements

Further relevant requirements may be specified. In this case, test methods to be used, acceptance criteria and frequency of testing shall be agreed between the interested parties.

5 Test methods

Carry out all tests in duplicate, unless otherwise specified. Prepare own test reports for tests for which no reference is made to relevant standards, comprising all essential information.

5.1 Preparation of test panels for testing coating powders

5.1.1 Take a representative sample of the coating powder to be tested, as described in EN ISO 8130-9.

5.1.2 Use one of the following aluminium alloys complying with the requirements specified in EN 573-3 as the substrate for the test panels:

EN AW-1050A – H14 (halfhard)

EN AW-5050A – H14 (halfhard)

EN AW-6060

EN AW-6063

EN AW-6063A

Unless otherwise specified, the dimensions shall be 150 mm x 100 mm x (0,8 mm to 1,0 mm). For referee tests, panels of thickness 0,8 mm shall be used.

5.1.3 Pretreat the test panels as described in 4.2 and coat each panel with the coating to be tested in accordance with the recommendations of the supplier of the coating powder. For mechanical tests, the film thickness of the coating shall be $(55 \pm 5) \mu\text{m}$.

5.1.4 Condition the coated test panels at $(23 \pm 2) ^\circ\text{C}$ and $(50 \pm 5) \%$ relative humidity for at least 1 h prior to testing, or as specified in the relevant test method.

5.2 Sampling of the final product and preparation of test specimens

5.2.1 Unless otherwise agreed, take samples in accordance with ISO 2859-1 and ISO 2859-2.

5.2.2 Prepare test specimens consisting, where possible, of the coated aluminium alloy extrusions, sheets or preformed sections comprising the lot under inspection. For some tests, test panels similar to those specified in 5.1 are required. Prepare these test panels at the same time as the parts comprising the lot and treat them precisely as the parts to be coated.

5.3 Colour

Compare the colour visually in accordance with EN ISO 3668 or colorimetrically in accordance with ISO 7724-3. Carry out the comparison at a film thickness that ensures sufficient hiding power.

5.4 Gloss

Measure the specular gloss in accordance with EN ISO 2813, using an angle of incidence of 60°.

5.5 Adhesion

Carry out the cross-cut test in accordance with EN ISO 2409, using a spacing of the cuts of 2 mm.

5.6 Cupping test

Carry out the cupping test in accordance with EN ISO 1520, using a depth of indentation of 5 mm.

5.7 Falling weight test

This test shall be carried out within 1 h to 3 h after application of the coating powder.

A suitable test apparatus is shown in annex D. A steel cylinder of mass 0,908 kg is allowed to fall freely from a height of 250 mm or another agreed height. The impact energy of the falling cylinder is transmitted by a ground-steel hemisphere of diameter 15,9 mm to the test panel which is placed over a die of 16,3 mm diameter. A vertical guide tube is used to direct the falling cylinder so that the centre lines of the cylinder and of the hemisphere coincide with that of the die.

Place the test panel on the die with the coating faced downwards. Raise the steel cylinder to a height of 250 mm or the agreed value and allow it to fall freely on to the test panel. Examine the coated surface of the indented test panel, using normal or corrected vision.

NOTE Performance in falling weight test may decrease during long-term ageing.

The test conditions for the falling weight test may be different depending on the coating powder formulation. Falling heights other than 250 mm may be specified by the supplier of the coating powder as a result of thermal analysis and shall be indicated in the test report.

5.8 Bend test

Carry out the bend test in accordance with EN ISO 1519, using a mandrel of diameter 5 mm.

5.9 Resistance to mortar

Prepare a mortar by mixing 15 g of hydrated lime, 41 g of cement and 244 g of sand with sufficient tap water to make a soft paste. Apply four portions of this mortar, about 15 mm in diameter and about 6 mm thick to the coating on the test panel.

Place the panel in an environment of (38 ± 3) °C and (95 ± 5) % relative humidity for 24 h.

At the end of this period, dislodge the mortar by hand from the surface of the coating and remove any residue with a damp cloth. Allow to dry and examine the coating with normal or corrected vision.

5.10 Pressure cooker test

This test requires a suitable pressure cooker equipped with a weighted needle valve and is carried out using one test panel.

Pour into the pressure cooker deionized water to a depth of (25 ± 3) mm. Stand the panel vertically into the pressure cooker and secure the lid of the cooker.

Heat the pressure cooker until steam is emitted from the valve, insert the weighted needle valve to give an internal pressure of (100 ± 10) kPa and continue heating for 2 h from the time when the steam has first emitted. Cool the pressure cooker with care. Remove the test panel and allow it to cool to ambient temperature. Examine it immediately but ignore the area within 3 mm of the edges.

5.11 Resistance to humidity

Determine the resistance to humidity in accordance with EN ISO 6270, using a test period of 1 000 h.

5.12 Resistance to acetic acid salt spray

Determine the resistance to acetic acid salt spray in accordance with ISO 9227 (AASS test), using a test period of 1 000 h.

5.13 Resistance to sulfur dioxide

Determine the resistance to sulfur dioxide in accordance with EN ISO 3231, using 0,2 l of sulfur dioxide and a test period of 24 cycles.

The test panel shall have scratches 1 mm wide in the form of a St. Andrews cross through the coating down to the substrate.

5.14 Resistance to filiform corrosion

5.14.1 Test panels

Use an agreed aluminium alloy representative of the production to be tested for the test panels which shall have dimensions of 110 mm x 90 mm.

Using a suitable tool, make two scratches through the coating down to the substrate, each 50 mm long and 1 mm wide. The distance of the scratches from each other and from the edges of the test panel shall be 20 mm (see Figure 1).

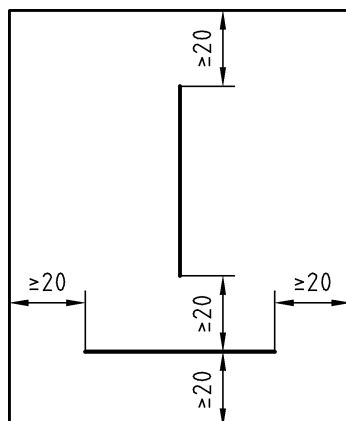


Figure 1 — Scratches on test panels

5.14.2 Testing final products

Test specimens shall be taken from normal production and have a minimum length of 110 mm.

5.14.3 Procedure

Introduce into a plastic tank sufficient hydrochloric acid, 30 % (by mass), to cover the bottom of the tank to a depth of at least 2 mm. Close the tank and maintain the tank at a temperature of (23 ± 1) °C for a minimum of 1 h to obtain in it an atmosphere saturated with hydrochloric acid vapour.

Place the test panels horizontally with the scratches faced downwards into the tank on racks at a height of (100 ± 10) mm above the liquid level. Close the tank. After 1 h remove the panels and immediately place them in a cabinet, maintained at (40 ± 2) °C and (80 ± 5) % relative humidity. After 1 000 h, examine visually for filiform corrosion. Express the result as the number of filaments per 10 cm of scratch, multiplied by the average length of the filaments.

5.15 Resistance to artificial weathering

Determine the resistance to artificial weathering in accordance with EN ISO 11341, using a test period of 1 000 h and an irradiance of 550 W/m^2 in the spectral range $290 \text{ nm} < \lambda < 800 \text{ nm}$, with the spectral energy distribution as given in Table 1 of EN ISO 11341:1997, a black standard temperature of (65 ± 2) °C and a wetting cycle of 102/18 min.

5.16 Resistance to natural weathering (referee test)

Determine the resistance to natural weathering in accordance with ISO 2810.

Expose coated test panels for a period of 12 months at an angle of 5° to the horizontal, facing south, starting in April, at a natural weathering site in Florida which shall be at least 5 km from the sea.

The test panels shall be washed prior to inspection with water containing 1 % (by mass) of a detergent, using a sponge and taking care to avoid polishing. Afterwards, the panels shall be rinsed with deionized water, having a maximum electrical conductivity of $1 \mu\text{S/cm}$.

Annex A (normative)

Methods for the determination of the mass per unit area of conversion layers and for the identification of chromium, phosphorus and zinc

A.1 Determination of mass per unit area

A.1.1 Scope

This method can be used for the determination of the mass per unit area of conversion layers. It is applicable to chromate and phosphochromate layers on aluminium and is based on the determination of mass differences.

A.1.2 Principle

A test specimen of a defined area is weighed. The conversion layer is then removed by an appropriate method. The specimen is then rinsed with deionized water, dried and reweighed. From the mass difference prior and after removal of the conversion layer and the area of the test specimen the mass per unit area is calculated.

For the removal of the coating two procedures are provided.

A.1.3 Apparatus

Analytical balance, capable of weighing to 0,1 mg.

A.1.4 Procedure

Take a test specimen of defined area (A) of approximately 400 cm² and weigh it to the nearest 1 mg (m_1). Remove the conversion layer by one of the procedures given in Table A.1, rinse the specimen with deionized water, dry it and reweigh it to the nearest 1 mg (m_2).

WARNING Attention is drawn to the potential hazard associated with the use of the compounds indicated in Table A.1.

Table A.1 — Procedures for removal of conversion layers

Medium for removal		Conditions for removal	
		min	°C
Nitric acid, 65 % (by mass), 1,4 g/ml	Freshly prepared layers cured below 40 °C and less than 8 h before testing	5 to 15	25
Molten-salt bath : 98 % (by mass) sodium nitrate ; 2 % (by mass) sodium hydroxide.	Freshly prepared layers cured above 40 °C, or aged layers (more than 8 h)	2 to 5	385 to 415

A.1.5 Expression of results

Calculate the mass per unit surface area, m_A , expressed in grams per square metre, using the following equation:

$$m_A = \frac{m_1 - m_2}{A} \times 10000$$

where :

- A is the area, in square centimetres, of the coated surface of the test specimen;
- m_1 is the mass in grams, of the test specimen before removal of the conversion layer ;
- m_2 is the mass, in grams, of the test specimen after removal of the conversion layer.

A.2 Non-destructive analytical methods for the identification of chromium, phosphorus and zinc

The following two methods can be used for chromate and phosphochromate coatings on aluminium.

A.2.1 Microprobe (SEM/EDS)

By means of a microprobe (SEM/EDS), the surface of the test specimen is checked for the presence or absence of chromium, phosphorus and zinc.

A.2.2 X-ray fluorescence technique

By means of the X-ray fluorescence technique the surface of the test specimen is checked for the presence or absence of chromium, phosphorus and zinc. The intensity of the fluorescent radiation is proportional to the content of the respective element in the conversion layer, and is compared to the fluorescent radiation of standards. The sensitivity limit is about 10 mg/m² to 20 mg/m² for chromium. The precision is about 5 % (relative).

A.3 Destructive analytical methods for the identification of chromium, phosphorus and zinc

A.3.1 Reagents

Use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in EN ISO 3696.

- A.3.1.1 Sodium hydroxide solution, 5 % (by mass).**
- A.3.1.2 Sodium hydroxide solution, 20 % (by mass).**
- A.3.1.3 Hydrogen peroxide solution, 30 % (by mass).**
- A.3.1.4 Nitric acid, concentrated, about 65 % (by mass).**
- A.3.1.5 Nitric acid, 1:1.**

Dilute 1 part by volume of concentrated nitric acid, approximately 65 % (by mass), by adding it to 1 part by volume of water.

- A.3.1.6 Acetic acid, 10 % (by mass).**
- A.3.1.7 Lead nitrate solution, 10 % (by mass).**
- A.3.1.8 Ammonium molybdate reagent.**

Dissolve 88,3 g of ammonium heptamolybdate tetrahydrate, (NH₄)₆Mo₇O₂₄ · 4H₂O, and 240 g of ammonium nitrate, NH₄NO₃, in water. Add 30 ml of ammonium hydroxide solution, 25 % (by mass), and make up to 1 litre with water.

- A.3.1.9 Ammonium nitrate.**

- A.3.1.10 Hydrochloric acid**, 25 % (by mass).
- A.3.1.11 Potassium hexacyanoferrate solution**, about 5 % (by mass).
- A.3.1.12 Sulfuric acid**, 25 % (by mass).
- A.3.1.13 Phenolphthaleine indicator**, 0,5 % (by mass) in ethanol (96 % by volume).
- A.3.1.14 Diphenylcarbazide solution**, about 0,1 % (by mass) in acetone.

Prepare this solution freshly before use.

A.3.2 Identification of chromium in the conversion layer

Treat a test specimen of about 300 cm² surface with a mixture of 50 ml of sodium hydroxide solution (A.3.1.1) and 5 ml of hydrogen peroxide solution (A.3.1.3) at 50 °C to 60 °C. Choose a suitable container appropriate to the shape of the test specimen. If it is not possible to immerse the test specimen completely, flood the solution repeatedly until the conversion layer is completely dissolved.

Transfer the solution to a beaker, and boil for about 10 min until the hydrogen peroxide is completely eliminated. Cool down to room temperature and filter the solution. For the identification of chromium, use one of the procedures given under a) and b).

- a) **using diphenylcarbazide** : Add carefully 10 ml to 25 ml of hydrochloric acid (A.3.1.10) to the solution. Cool down to room temperature and add 2 ml of diphenylcarbazide solution (A.3.1.14). A purple precipitate indicates the presence of hexavalent chromium. Residual hydrogen peroxide will interfere. The minimum quantity of total chromium detectable corresponds to about 3 mg/m² ;
- b) **using lead nitrate** : Add lead nitrate solution (A.3.1.7) to the solution. A yellow precipitate indicates the presence of hexavalent chromium. The minimum quantity of total chromium detectable corresponds to about 5 mg/m².

A.3.3 Identification of phosphorus in the conversion layer

Treat a test specimen of an area of about 100 cm² surface with 100 ml of sodium hydroxide solution (A.3.1.1) at 80 °C to 90 °C until the conversion layer is completely dissolved or until the surface has been clearly attacked. Filter the resulting solution and acidify with an excess of nitric acid 1 + 1 (A.3.1.5).

Add 10 ml of ammoniummolybdate reagent (A.3.1.8) and 5 g of ammonium nitrate (A.3.1.9). If phosphorus is present, a yellow precipitate will be formed within 15 min. The minimum quantity of phosphorus detectable corresponds to about 40 mg/m² of phosphorus pentoxide.

A.3.4 Identification of zinc in the conversion layer

Treat a test specimen of about 100 cm² surface with 50 ml of concentrated nitric acid (A.3.1.4) at room temperature until the conversion layer is completely dissolved or until the surface has been clearly attacked. Filter the resulting solution through glass wool. Neutralize 25 ml of the solution with sodium hydroxide solution (A.3.1.2) until, after addition of phenolphthaleine indicator (A.3.1.13), the solution shows a permanent pink colour.

Add 10 drops of sulfuric acid (A.3.1.12), followed by 5 ml of potassium hexacyanoferrate solution (A.3.1.11). In the presence of zinc, a greenish-white precipitate will be formed. The minimum quantity of zinc detectable corresponds to about 20 mg/m².

A.3.5 Spectrometric methods

By means of atomic absorption spectrometry or inductively-coupled-plasma spectroscopy it is possible and usually convenient to check for the presence or absence of chromium and zinc in the test solutions.

The test solutions, prepared as described in A.3.2 and A.3.4 respectively, are analyzed after filtration. In parallel, blank solutions are prepared from aluminium alloy substrate which has not been surface-treated.

Annex B (normative)

Requirements for alternative pretreatment

B.1 All alternate pretreatment systems for subsequent coating using coating powders shall lead to a finished product meeting the performance requirements of 4.3 of this Part of EN 12206.

B.2 All alternate pretreatment systems for subsequent coating using coating powders shall have completed a minimum of two years for interim approval and five years for full approval, natural exposure in both marine and industrial conditions in accordance with EN ISO 8565.

At the end of this period, the total area of the blistering, softening, detachments of the coatings and the signs of corrosion shall not be greater than 16 mm² by 10 cm scratch length and no attack shall be longer than 4 mm.

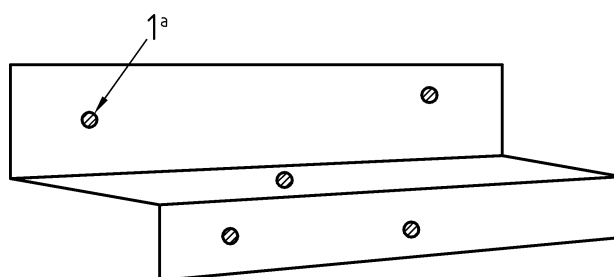
Annex C (normative)

Measurement of film thickness

C.1 The film thickness on each significant surface of the coating on each part to be tested shall be measured at not less than five measuring points distributed around the significant surface. 3 to 5 single readings shall be taken at each measuring point which shall be about 1 cm² in area. The average of the single readings taken at one measuring point gives the measurement value to be recorded in the inspection report. If any measurement value is less than 80 % of the required film thickness, the piece in question is unsatisfactory and is to be considered as a reject sample in accordance with Table C.1, column 3. For further details see EN ISO 2360.

Measuring point 1 cm ²

The average of the 3 to 5 single readings gives the measurement value of a measuring point



- a At least 5 measuring points per piece, distributed around the upper significant surface

Key

1 Single reading

Figure C.1 – Measurement of film thickness

Measurements shall not be made close to an edge, hole, inside corner, etc.

C.2 Inspection of finished products shall follow the statistical control indicated in Table C.1.

Table C.1 — Statistical control of finished products

Lot size ^a	Number of samples (random selection)	Acceptance limit for substandard samples
1 to 10	all	0
11 to 200	10	1
201 to 300	15	1
301 to 500	20	2
501 to 800	30	3
801 to 1 300	40	3
1 301 to 3 200	55	4
3 201 to 8 000	75	6
8 001 to 22 000	115	8
22 001 to 110 000	150	11

^a A lot comprises the complete order of a customer or the part of it which is in the works.

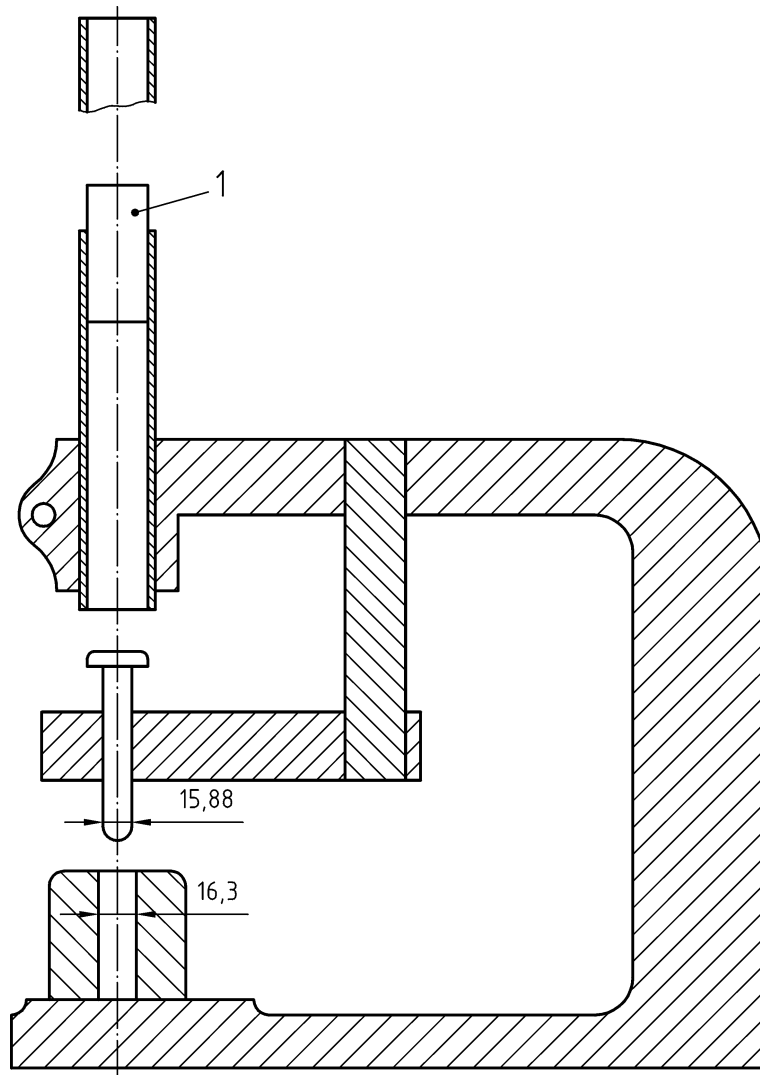
C.3 The average thickness proposed is 50 µm with an absolute minimum of 80 % = 40 µm verified as described in C.1 and C.2.

The film thickness shall be no less than 40 µm and on average no less than 50 µm. Any component with a measuring value at any measuring point of less than 40 µm is a sub-standard and cannot be accepted.

Annex D (normative)

Falling weight test apparatus

Dimensions in millimetres



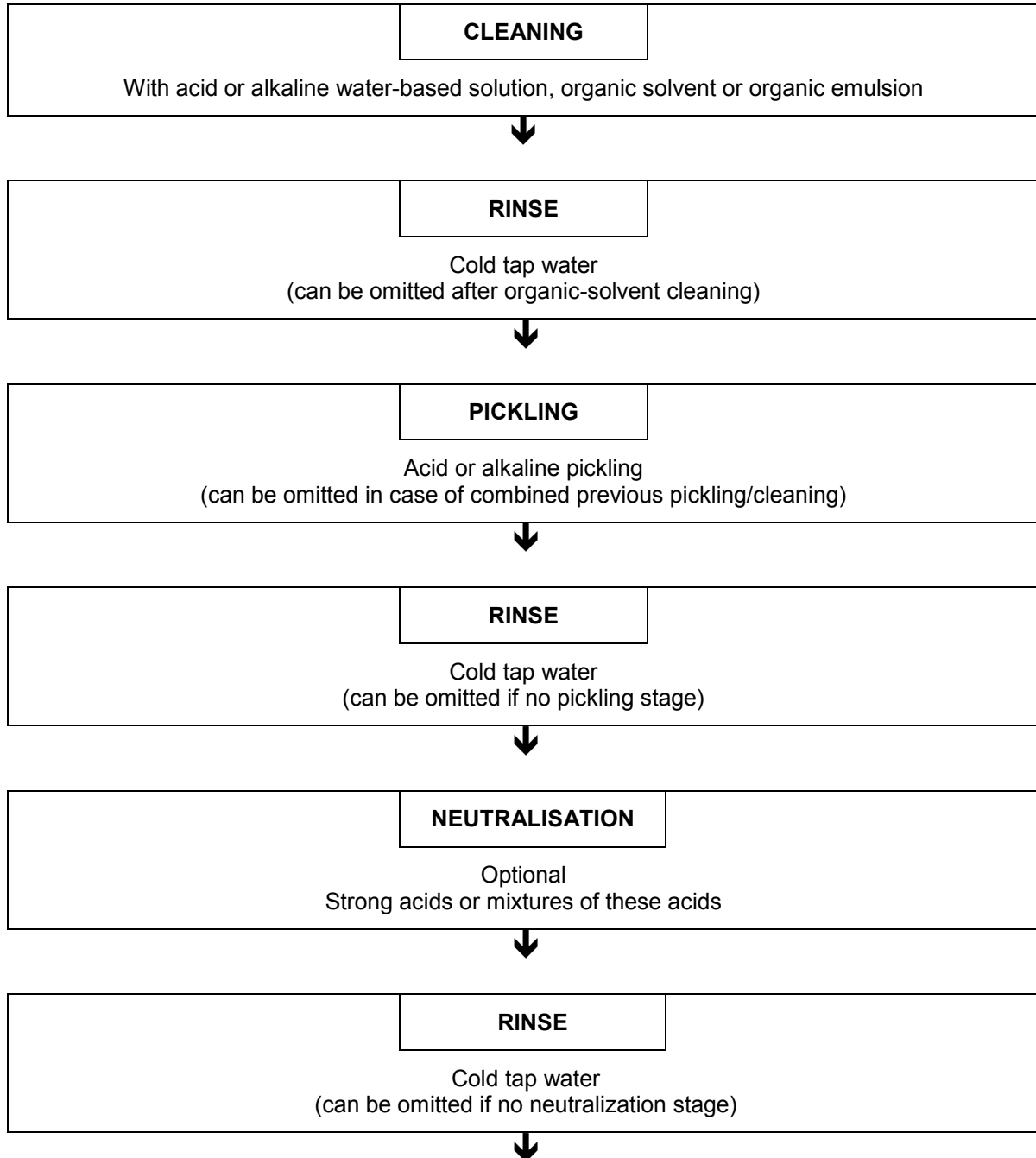
Key

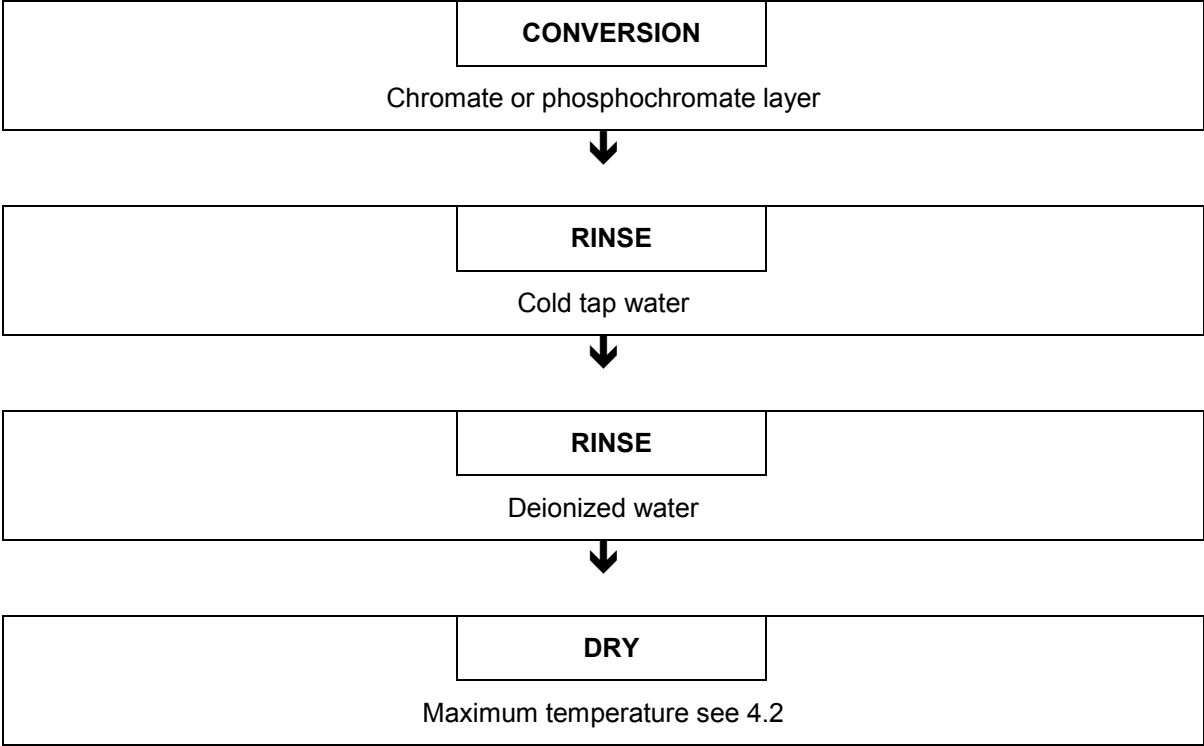
- 1 Mass of falling cylinder = 0,908 kg

Figure D.1 — Falling weight test apparatus

Annex E
(informative)

Sequence of pretreatment stages





NOTE All rinsing operations are to be performed carefully in order to avoid pollution of the successive solutions.

Annex F
(normative)

Enhanced durability coating powders

Recent advances in technology have led to the development of coating powder systems which have improved gloss retention under natural weathering conditions, e.g. typically 5 years exposure at the Florida test site referenced in 5.16 with less than 50 % drop in gloss.

These systems are, however, less flexible and may not meet the criteria of 4.3.5, 4.3.6 and 4.3.7.

Where these systems are to be used, it is suggested that the interested parties agree modifications of these test methods and/or criteria, e.g. the inclusion of a tape test with no disbondment in place of cracking, together with suitable adjustment of the exposure period specified in 5.16.

All the remaining criteria of 4.3 shall be met.

Bibliography

EN 971-1:1996, *Paints and varnishes – terms and definitions for coating materials – Part 1: General terms.*

EN ISO 2360, *Non-conductive coatings on non-magnetic electrically conductive basis metals – Measurement of coating thickness – Amplitude-sensitive eddy current method (ISO 2360:2003).*

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