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Oorganiska ytbeläggningar – Kromateringsskikt på zink, kadmium, aluminium-zinklegeringar och zink-aluminiumlegeringar – Provningsmetoder (ISO 3613:2010)

Metallic and other inorganic coatings – Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys – Test methods (ISO 3613:2010)

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Europastandarden EN ISO 3613:2010 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 3613:2010.

Denna standard ersätter SS-EN ISO 3613, utgåva 2.

The European Standard EN ISO 3613:2010 has the status of a Swedish Standard. This document contains the official version of EN ISO 3613:2010.

This standard supersedes the Swedish Standard SS-EN ISO 3613, edition 2.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Oorganiska ytbeläggningar, SIS/TK 116.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 3613

December 2010

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Supersedes EN ISO 3613:2001

English Version

Metallic and other inorganic coatings - Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys - Test methods (ISO 3613:2010)

Revêtements métalliques et autres revêtements inorganiques - Couches de conversion au chromate sur zinc, cadmium et alliages d'aluminium-zinc et de zinc-aluminium - Méthodes d'essai (ISO 3613:2010)

Metallische und andere anorganische Überzüge - Chromatierüberzüge auf Zink, Cadmium, Aluminium-Zink- und Zink-Aluminium-Legierungen - Prüfverfahren (ISO 3613:2010)

This European Standard was approved by CEN on 3 December 2010.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN ISO 3613:2010) has been prepared by Technical Committee ISO/TC 107 “Metallic and other inorganic coatings” in collaboration with 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 June 2011, and conflicting national standards shall be withdrawn at the latest by June 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 3613:2001.

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Endorsement notice

The text of ISO 3613:2010 has been approved by CEN as a EN ISO 3613:2010 without any modification.

Introduction

This International Standard specifies methods for the qualitative determination of the presence of chromate conversion coatings as well as the total chromium content of chromate conversion coatings.

The application of very thin, colourless, practically invisible chromate conversion coatings is frequently called “passivation”, while the application of thicker, coloured chromate conversion coatings is called “chromating”. The term “passivation” is not correct, does not comply with the ISO 2080 designation and is therefore deprecated.

Metallic and other inorganic coatings — Chromate conversion coatings on zinc, cadmium, aluminium-zinc alloys and zinc-aluminium alloys — Test methods

WARNING — This International Standard calls for the use of substances and/or procedures that may be injurious to health if adequate safety measures are not taken. This International Standard does not address any health hazards, safety or environmental matters associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health, safety and environmentally acceptable practices and take suitable actions for any national and international regulations. Compliance with this International Standard does not in itself confer immunity from legal obligations.

1 Scope

This International Standard specifies methods for the determination of

— the presence of colourless chromate conversion coatings,

— the presence of hexavalent chromium in colourless and coloured coatings on zinc or cadmium or aluminium-zinc (mass fraction of aluminium: 55 %, within a range of 54 % to 56 % mass fraction) and zinc-aluminium (mass fraction of aluminium: 5 %) alloys,

— the total chromium content per unit area on zinc and cadmium,

— the mass per unit area of both colourless and coloured coatings,

— the satisfactory adhesion of chromate conversion coatings, and

— the quality of chromate coatings.

These methods are applicable

— to colourless and coloured chromate conversion coatings containing trivalent and hexavalent chromium in varying proportions and produced by either chemical or electrochemical processes, and

— only to chromate coatings that are free from any supplementary coatings, such as oil, water or solvent-based polymers or wax.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 4520, *Chromate conversion coatings on electroplated zinc and cadmium coatings*

IEC 60068-2-30, *Environmental testing — Part 2-30: Tests — Test Db: Damp heat, cyclic (12 h + 12 h cycle)*

3 Reagents

Use only reagents of recognized analytical grade and distilled water or water of equivalent purity, unless otherwise specified, for analysis.

3.1 Test solution A (see 5.2).

Dissolve 1 g of diphenylcarbazide in a mixture of 20 ml of acetone, 60 ml of glacial acetic acid and 40 ml of distilled water contained in a beaker. Add 15 ml of concentrated hydrochloric acid ($\rho = 1{,}18$ g/cm^3), stir and add slowly 30 ml of sodium hypochlorite solution (10 % to 15 % available chlorine). Add 5 ml of hydrogen peroxide (30 % volume fraction) slowly with continuous stirring. Leave the solution in the open beaker for 24 h in a fume cupboard, to allow excess chlorine to escape, before use.

The solution does not deteriorate with age and can be kept in a bottle with a loosely fitted stopper. However, there may be losses due to evaporation and the concentration may alter so it is discarded after 6 months.

3.2 Test solution B (see 5.3).

Dissolve 50 g of lead acetate trihydrate [$(CH_3COO)_2Pb{\cdot}3H_2O$] in 1 l of distilled water or water of equivalent purity. Ensure that the pH of the solution is between 5,5 and 6,8 as prepared. If the pH of the solution is outside this range, discard the solution and obtain a new supply of lead acetate.

Any white precipitate formed during the initial preparation of the solution may be dissolved by small additions of glacial acetic acid, provided that the pH is not reduced to a value below 5,5. Discard the stock solution if the white precipitate does not disappear.

3.3 Test solution C (see 5.5)

3.3.1 Test solution C 1

Dissolve 0,4 g of diphenylcarbazide in a mixture of 20 ml of acetone and 20 ml of ethanol (96 %). After dissolution, add 20 ml of 75 % orthophosphoric acid solution and 20 ml of distilled water. Prepare this solution not more than 8 h prior to use.

3.3.2 Test solution C 2

Add 700 ml of orthophosphoric acid, of specific gravity 1,7, to 250 ml of distilled water or water of equivalent purity and make up to 1 000 ml.

Dissolve 1,0 g of 1,5-diphenylcarbazide in 100 ml of acetone, adding one drop of glacial acetic acid to help dissolution. Keep the solution in a dark glass bottle in the refrigerator. The solution shall be discarded after 4 weeks.

For the Cr(VI) standard solution, dissolve 0,113 g of $K_2Cr_2O_7$ in distilled water or water of equivalent purity and make up to the mark in a 1 000 ml volumetric flask. Measure 2,5 ml of this solution into a second 1 000 ml volumetric flask and make up to the mark. 1 ml of this standard solution contains 0,1 µg of Cr(VI). The solution shall be discarded after 9 months.

For the preparation of comparison solution, add 1 ml of phosphoric acid and 1 ml of diphenylcarbazide solution to 50 ml of standard Cr(VI) solution, and mix thoroughly. Allow the solution to stand for 10 min for the colour reaction to be completed.

3.4 Test solution D (see 5.6 and 5.7).

Dissolve 0,50 g of diphenylcarbazide in 50 ml of acetone. Dilute slowly, while stirring, with 50 ml of water (rapid mixing can result in precipitation of diphenylcarbazide).

For maximum stability, store the solution under refrigeration in an amber-coloured glass bottle.

3.5 Sulfuric acid, diluted 1 + 3.

Slowly add 1 volume of concentrated sulfuric acid (ρ = 1,84 g/cm^3) to 3 volumes of water.

3.6 Ammonium persulfate [$(NH_4)_2S_2O_8$].

3.7 Sodium hydroxide (NaOH), 240 g/l solution.

3.8 Silver nitrate ($AgNO_3$), 17 g/l solution.

3.9 Potassium dichromate ($K_2Cr_2O_7$), standard solution.

Dilute 2 ml of standard volumetric potassium dichromate solution (4,9 g/l) to 1 000 ml.

3.10 Phosphate buffer solution.

Dissolve 55 g of sodium dihydrogen orthophosphate monohydrate ($NaH_2PO_4 \cdot H_2O$) in 100 ml of water.

4 Apparatus

Use normal laboratory apparatus and the following.

Clean all glassware thoroughly. It is recommended that cleaning be done by boiling the glassware with nitric acid (HNO_3) of 30 % mass fraction and then rinsing thoroughly with distilled water or water of equivalent purity.

4.1 Photoelectric absorptiometer (colorimeter), with a filter having a mean transmission of 520 nm. The absorptiometer shall be used with an absorption cell having an optical path length of 10 mm.

4.2 Spectrophotometer, with wavelength set at 540 nm. The spectrophotometer shall be used with an absorption cell having an optical path length of 10 mm.

4.3 Analytical balance, capable of weighing to the nearest 0,1 mg.

5 Test methods

5.1 General

The test surface, prior to the following tests, shall be free of all contaminants, finger prints and other extraneous stains. If the surface is coated with a thin oil film, this shall be removed prior to the test by degreasing using a suitable solvent at room temperature (not exceeding 35 °C). If it is necessary to store samples, they shall be stored at temperatures not exceeding 40 °C and at a relative humidity below 70 %. For test purposes, the samples shall not be subjected to forced drying at temperatures in excess of 35 °C. Treatment in alkaline solutions shall not be performed as chromate conversion coatings are broken down by alkalis. Spot tests are not always a precise means of determining the presence of chromate coatings.

The test methods for

a) the presence of a colourless chromate coating on zinc, cadmium and aluminium-zinc alloys,

b) the presence of hexavalent chromium in both coloured and colourless chromate coatings,

c) the determination of hexavalent chromium content, and

d) the determination of total chromium content