BRITISH STANDARD

BS EN
15205:2006

Determination of hexavalent chromium in corrosion protection layers — Qualitative analysis

The European Standard EN 15205:2006 has the status of a British Standard

ICS 77.060

BSI
British Standards

National foreword

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A list of organizations represented on STI/33 can be obtained on request to its secretary.

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English Version

Determination of hexavalent chromium in corrosion protection layers - Qualitative analysis

Détermination du chrome hexavalent dans les revêtements anti-corrosion - Analyse qualitative

Bestimmung von sechswertigem Chrom in Korrosionsschutzschichten - Qualitative Bestimmung

This European Standard was approved by CEN on 6 October 2006.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 15205:2006) 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of an identical text or by endorsement, at the latest by May 2007, and conflicting national standards shall be withdrawn at the latest by May 2007.

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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard describes a testing method for the qualitative analysis of hexavalent chromium in corrosion protection layers.

WARNING — Use of this standard may involve the handling of hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Principle

The coated part to be examined (test sheet metal, construction unit) is extracted and the Cr(VI) content in the extracted solution is determined according to the colour reaction with 1,5-Diphenylcarbazide. Cr(VI) oxidizes 1,5-Diphenylcarbazide to 1,5-Diphenylcarbazone, which forms a red-violet coloured complex with the developed Cr(III). Evaluation is performed by visual inspection of the coloured solution or by using a spectrophotometer.

3 Apparatus

Normal laboratory apparatus and the following:

NOTE Clean all glassware thoroughly. It is recommended that cleaning is done by boiling the glassware with 5 M HNO_3 and then it rinsing thoroughly with deionised water (4.1).

3.1 Spectral or filter photometer. With a spectral photometer, the wavelength is set at 540 nm. With a filter photometer, use a filter with a middle transparency at approximately 540 nm.

3.2 Cuvettes, with a path length of 1 cm or 5 cm.

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

4 Reagents

4.1 Deionised water, having a pH value 4 to 7 and conductivity <10 µS.

4.2 Ortho phosphoric acid

Add 700 ml of orthophosphoric acid (87 %) to 250 ml of water, then make up to 1 000 ml with deionised water (4.1).

4.3 Diphenylcarbazide solution

Dissolve 1,0 g of 1,5-Diphenylcarbazide in 100 ml acetone adding one drop of glacial acetic acid to help dissolution. Keep the solution in a dark glass bottle in the refrigerator. The solution is stable for at least 4 weeks.

4.4 Cr(VI) Standard solution

Dissolve 0,113 g of $K_2Cr_2O_7$ in deionised water (4.1) and make up to the mark of 1 000 ml in a volumetric flask.

NOTE This solution has a shelf life approximately of 1 year.

Pipette 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 Cr(VI).

4.5 Comparison solution

To 50 ml of standard solution (4.4) add 1 ml phosphoric acid and 1 ml diphenylcarbazide solution (4.3) and mix thoroughly. Allow the solution to stand for 10 min for the colour reaction to be completed.

5 Procedure

5.1 Preparation for extraction

The sample shall have a surface area of (50 ± 5) cm^2.

For large test pieces, remove a section with a surface area of (50 ± 5) cm^2.

For small test pieces, take several pieces to give a total surface area of (50 ± 5) cm^2.

NOTE If it is necessary to deviate from the standard sample surface area, maintain the ratio of indicator solution used. For example, for every cm^2 of the sample, 1 ml of indicator solution should be used.

Sealed (e.g. painted) parts shall be scratched. The resulting abrasion shall also be analysed.

5.2 Extraction and comparison

Carry out the extraction in a graduated beaker using boiling deionised water (4.1). Anti-bumping granules shall be added. Immerse the sample in the water, cover with a watch glass and boil for exactly 10 min after the sample resumes boiling. The entire sample shall be covered with water throughout the boiling time.

Remove the beaker with the sample from the hot plate and remove the sample. Allow the beaker contents to cool to ambient temperature. If necessary, filter the solution using, e.g. a 0,45-µm filter and make the solution up to the mark (e.g. 50 ml) with deionised water (4.1) or reduce it to the mark by boiling. Add 1 ml ortho phosphoric acid (4.2) per 50 ml volume and 1 ml diphenylcarbazide solution (4.3) and mix well.

Allow the solution to stand for 10 min for the colour reaction to be completed.

Judge the colour of the solution obtained visually against the comparison solution (4.5).

6 Expression of test results

Express the results of the comparison of test and comparison solution as described in Table 1.

Table 1 — Comparison of solutions

Observation	Cr(VI)-concentration	Result
The colour intensity of the sample solution is lower than that of the comparison solution	< 0,1 µg/cm^2	Sample is free from Cr(VI)
The colour intensity of the sample solution is higher than that of the comparison solution	> 0,1 µg/cm^2	Sample contains Cr(VI)

If a clear visual estimation is not possible with the comparison solution, or if before making the indicator addition a disturbing self-colouring of the solution occurs, carry out a photometric measurement at a wavelength of 540 nm against the comparison solution.

7 Test report

The test report shall contain as a minimum the following information:

a) all information necessary for identification of the sample tested;

b) reference to this European Standard (EN 15205);

c) results of the test, including the results of the individual determinations and their mean as described in clause 6;

d) any deviations from the procedure specified;

e) any unusual features (anomalies) observed during the test;

f) date of the test or the date of test report.

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