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**Določevanje šestvalentnega kroma v plasteh antikorozijske zaščite –  
Kvalitativne analize**

Determination of hexavalent chromium in corrosion protection layers - Qualitative  
analysis

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March 2005

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ICS

English version

## Determination of hexavalent chromium in corrosion protection layers - Qualitative analysis

Bestimmung von sechswertigem Chrom in  
Korrosionsschutzschichten - Qualitative Bestimmung

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## Foreword

This document (prEN 15205:2005) has been prepared by Technical Committee CEN/TC 262 “Refractory products and materials”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

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## 1 Scope

This document describes the testing method for the qualitative analysis of hexavalent chrome in corrosion protection layers.

## 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-diphenylcarbazine. Cr(VI) oxidises 1,5-Diphenylcarbazine to diphenylcarbazone, which forms a red-violet coloured complex with the developed Cr(III). The evaluation is made by visual inspection of the coloured solution.

## 3 Apparatus

Normal laboratory apparatus and the following:

NOTE Clean all glassware thoroughly. It is recommended that cleaning is done by boiling with 5 M HNO<sub>3</sub> and then rinsing thoroughly with deionised water.

**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 Ortho phosphoric acid

Make 700 ml of 87% ortho-phosphoric acid solution up to 1000 ml with deionised water.

### 4.2 Diphenylcarbazine solution

Dissolve 1,0 g of 1,5-diphenylcarbazine in 100 ml acetone with the addition of 1 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.

### 4.3 Cr(VI) Standard solution

Dissolve 0,113 g of K<sub>2</sub>CrO<sub>7</sub> in deionised water and made up to the mark of 1000 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 1000 ml volumetric flask and make up to the mark. 1 ml of this standard solution contains 0,1 mg Cr(VI).

### 4.4 Comparison solution

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

## 5 Procedure

### 5.1 Preparation for extraction

Use a chrome-plated sample with a surface area of  $(50 \pm 5) \text{ cm}^2$ .

For small test pieces, take several to achieve a total surface area of  $(50 \pm 5) \text{ cm}^2$ . For larger test pieces, remove a section with a surface area of  $(50 \pm 5) \text{ cm}^2$ .

NOTE If it is necessary to deviate from the standard sample surface area, maintain the ratio of indicator solution used. For example, for every  $\text{cm}^2$  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. Anti-bumping granules shall be added. Immerse the sample in the water, cover with a watch glass and boil for exactly 10 minutes. 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. Make the solution up to the mark (e.g. 50 ml) with deionised water or reduce it to the mark boiling. Add 1 ml ortho phosphoric acid (4.1) per 50 ml volume and 1 ml diphenylcarbazide solution (4.2) and mix well.

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

Judge the colour of the solution obtained visually against the comparison solution (4.4).

## 6 Expression of test results

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

Observation	Cr(VI)-concentration	Result
The colour intensity of the sample solution is lower than that of the comparison solution.	$< 0,1 \mu\text{g}/\text{cm}^2$	Sample is free from Cr(VI).
The colour intensity of the sample solution is higher than that of the comparison solution.	$> 0,1 \mu\text{g}/\text{cm}^2$	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.