



Absence of Hexavalent Chrome, Cr VI, in Coatings

1 Introduction

Note: Nothing in this standard supercedes applicable laws and regulations.

Note: In the event of conflict between the English and domestic language, the English language shall take precedence.

1.1 Purpose. This test is used to prove the absence of Cr VI in coatings.

1.2 Applicability. This Test Procedure is applicable to materials and components, that may contain Cr VI in their coatings.

1.3 Remarks This test method is derived from ISO 3613 – Test Fluid C and is equivalent to EN 15205.

2 References

Note: Only the latest approved standards are applicable unless otherwise specified.

2.1 External Standards/Specifications.

EN 15205 ISO 3613

2.2 GM Standards/Specifications.

None

3 Resources

3.1 Facilities.

3.1.1 Calibration. The test facilities and equipment shall be in good working order and shall have a valid calibration label.

3.1.2 Alternatives. Alternative test facilities and equipment may also be used. However, all measuring variables as specified in this standard shall be determined correctly with respect to their physical definition.

3.2 Equipment.

3.2.1 Ordinary Laboratory Apparatus.

3.2.2 Colorimeter or Spectrophotometer. With a colorimeter, use a filter having a mean transmission of 540 nm. A wavelength of 540 nm shall be set for the spectrophotometric measurement. Use an absorption cell with an optical path length of 1 cm in both cases.

3.3 Test Vehicle/Test Piece.

3.3.1 Coating materials.

3.3.2 Reagents. Use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

3.3.2.1 Sulfuric Acid, 4.5 M. Mix three (3) volumes of water with one (1) volume of concentrated sulfuric acid (1.84 g/ml).

Note: When preparing sulfuric acid solution wear safety glasses. Take a vessel of heat resistant glass and fill it with three (3) volumes of water. Add one (1) volume of concentrated sulfuric acid slowly or in small portions, while stirring the water. Never pour water into concentrated sulfuric acid.

3.3.2.2 Diphenylcarbazide Solution. Dissolve 0.5 g of diphenylcarbazide in 50 ml of acetone. Dilute slowly, while stirring, with 50 ml of water (rapid mixing may result in precipitation of diphenylcarbazide). For maximum stability store under refrigeration in an amber colored glass bottle.

Note: Be aware that diphenylcarbazide is a cancer producing material. When handling it, make sure that you do not breathe in the dust or get otherwise in contact with it.

3.3.2.3 Cr VI Standard Solution. Dissolve 0.113 g $K_2Cr_2O_7$ in water and dilute it in a volumetric flask to 100 ml. Pipette 0.25 ml of this solution into a second 100 ml volumetric flask and dilute to the mark with water. The second solution contains 1 μg Cr VI per ml (5 ml = 5 μg Cr VI).

3.4 Test Time.

Calendar time: 1 day
Test time: 24 hours
Coordination time: 0 hours

3.5 Test Required Information. Not applicable.

3.6 Personnel/Skills. Well trained laboratory skilled workers or persons with a chemical education.

4 Procedure

4.1 Preparation. The test sample shall have a surface area of (50 ± 5) cm². From small parts take the suitable number of samples. From larger parts, use only a (50 ± 5) cm² segment.

4.2 Conditions.

4.2.1 Environmental Conditions. See 4.3.

4.2.2 Test Conditions. Deviations from the requirements of this standard shall have been agreed upon. Such requirements shall be specified on component drawings, test certificates, reports, etc.

4.3 Instructions. The test sample is leached with boiling water for 5 minutes. Hexavalent chrome compounds will react with diphenylcarbazide and form a red colored complex that can be observed visually, and, or detected colorimetrically or spectrophotometrically.

4.3.1 Place the sample in a 100 ml beaker with volume graduation, or other suitable beaker with volume graduation. Add 50 ml water. Heat the water to boiling and keep it boiling for exactly 5 minutes.

4.3.2 Remove the test piece and cool the beaker and its contents to $(+23 \pm 5)$ °C. Fill up with water to 50 ml if some water evaporated. If the solution is milky or has a precipitate, filter through a paper filter into a dry beaker. Add 1.5 ml of dilute sulfuric acid solution (4.5 M) and 1 ml of diphenylcarbazide solution; then mix well. Evaluate the color against the comparison solution (see "Comparison Solution Preparation"). A red color indicates Cr VI.

4.3.3 If a clear evaluation against the comparison solution is not possible visually, the sample is poured into an absorption cell and after a reaction time of 2 minutes, it is measured with a spectrophotometer against the comparison solution.

4.3.4 Colorimetric or spectrophotometric measurement shall be performed when water leaching produces a colored solution which interferes with the visual evaluation.

4.3.5 Comparison Solution Preparation.

4.3.5.1 For comparison, dilute 5 ml of the Cr VI-standard solution (5 ml = 5 μg Cr VI) to 50 ml with water. This solution is treated in the same way as the sample solution (see 4.3).

4.3.5.2 If according to this method, the coating is found to be Cr VI free, it still may be that a Cr VI containing prime coat is covered by a Cr VI free top coat. In this case, scrape the coating in thin layers with a suitable instrument and test against the test sample together with the coating particles.

5 Data

5.1 Calculations. Not applicable.

5.2 Interpretation of Results. The coating is accepted to be free of Cr VI ($<0.1 \mu g/cm^2$) if the red color of the test sample solution is less than the comparative solution visually, or the absorption of the sample solution at 540 nm is less than the comparative solution absorption at 540 nm.

5.3 Test Documentation. With reference to this test procedure, the test results shall be presented in a test report.