Designation: D 6492 – 99

Standard Practice for Detection of Hexavalent Chromium On Zinc and Zinc/Aluminum Alloy Coated Steel[1]

This standard is issued under the fixed designation D 6492; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice can be used to detect the presence of hexavalent chromium on galvanized and zinc/aluminum alloy coated steel surfaces. Hexavalent chromium-bearing treatments (passivates) can be applied to coated steels to prevent storage stain. While passivated 55 % aluminum-zinc alloy coated steel is commonly painted, passivated galvanized steel is not. Chrome passivation may interfere with the successful pretreatment of galvanized steel, as well as contaminate cleaning and pretreatment baths on a coil coating line.

1.2 The amount of hexavalent chromium that will cause the indicator to produce a discernible pink color is in the range of 0.5 parts per million dissolved in the indicator solution. It is possible that a coated steel surface that produces a negative result does have chromium on the surface. If a material that yields a negative result is suspected of having chromium on the surface, instrumental methods should be used. Chrome deposits of 1 mg/ft^2 can be easily missed by analytical instruments such as the scanning electron microscope with energy dispersive x-ray analysis (EDXA) capability. Auger electron spectroscopy (AES) or electron spectroscopy for chemical analysis (ESCA) can identify chemical species present in the levels required for adequate detection. Stripping the metallic coating and analyzing for chrome by atomic absorption or inductively coupled plasma can also give reliable results in detecting the presence of chrome.

1.3 This practice is designed to be a qualitative means of screening chrome passivated coils from those which are not chrome passivated.

1.4 Some chromium-free passivates are being used commercially. Although these products will test negative for hexavalent chromium, they may interfere with cleaning and pretreating. Chromium bearing passivates that contain film forming constituents such as acrylic resins are also being commercially applied. The reaction of these products to the spot test will vary. Abrading the surface with emery paper will improve the likelihood of reliable detection. This technique is not recommended for acrylic resin containing passivation treatments.

1.5 *This standard does not purport to address all of the safety concerns, if any, 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. Referenced Documents

2.1 *ASTM Standards:*

D 2092 Guide for Treatment of Zinc-coated (Galvanized Steel Surfaces) for Painting[2]

D 5723 Practice for Determination of Chromium Treatment Weight on Metal Substrates by X-Ray Fluorescence[2]

3. Apparatus

3.1 The following materials will be required to perform the stripping procedure:

3.1.1 *Dark colored or Brown Polyethylene Wash Bottle*, (500 mL) or brown glass dropper bottle.

3.1.2 *Test Specimens*, which may be cut panels or coil stock.

4. Reagents

4.1 The following chemical reagents are required to perform this procedure:

4.1.1 *1,5-Diphenylcarbohydrazide.*

4.1.2 *Acetone.*

4.1.3 *Ethanol*, 85 %.

4.1.4 *Phosphoric Acid.*

4.1.5 *Distilled Water.*

5. Preparation of Indicator Solution

5.1 Combine 25 mL acetone with 25 mL ethanol in a suitable container. Add 0.5 g 1,5-diphenylcarbohydrazide and allow to dissolve completely. Vigorous agitation may be required to effect complete dissolution. Slowly add 25 mL 85 % phosphoric acid to 25 mL distilled water. Add the diluted phosphoric acid to the 1,5-diphenylcarbohydrazide solution and mix well.

5.2 The indicator solution has a shelf life of approximately 6 h when exposed to light. Shelf life can be extended to several

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[2] *Annual Book of ASTM Standards*, Vol 06.02.

days if the solution is kept refrigerated. Discard the solution if it becomes discolored.

6. Procedure

6.1 Remove oil and dirt from the metal surface to be tested. A thorough solvent wipe is generally sufficient. Apply several drops of the indicator to the material to be tested, or use a wash bottle to apply a small amount of solution onto outer and inner wraps of a coil. Samples of non-passivated material should be tested side-by-side as controls. Any color change perceived in the indicator on the material being tested should be compared to the controls.

7. Evaluation

7.1 A pink color will develop if hexavalent chromium is present. The color can appear within 30 s for material with a passivation less than 1 week old. For material up to 1 month old, the reaction time should be extended to 30 min. If the material is older than 1 month, the reaction time should be extended to 1 h. The color that is achieved and the time required for the color change to occur depends upon age of the metal being tested, the amount of chromium on the surface, and the age of the indicator. Heavier hexavalent chrome concentrations will give a darker pink color.

7.2 Even heavy chrome passivation (greater than 3 mg/ft^2 chrome) can yield a light pink color or no color change at all. The detection of hexavalent chrome by this practice requires hexavalent chrome ions to be dissolved by the indicator solution. It is common for chrome passivation films to decline in hexavalent chrome content over time. The temperature and humidity conditions under which the coils are stored will greatly affect the residual hexavalent chrome concentration as well as its solubility.

8. Keywords

8.1 diphenylcarbohydrazide; hexavalent chromium; passivate; passivation; steel; zinc; zinc/aluminum