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**Adornment - Determination of Chromium (VI) - 1,5-
Diphenylcarbohydrazide Spectrophotometric Method**

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Adornment - Determination of Chromium (VI) - 1,5-Diphenylcarbohydrazide Spectrophotometric Method

1 Scope

This standard specifies the 1,5-Diphenylcarbohydrazide spectrophotometric method for Chromium (VI).

This Standard applies to the determination of Chromium (VI) in adornment.

2 Method and Principle

Use alkaline leaching solution to leach the water-soluble and water-insoluble hexavalent chromium compounds from the sample. In acidic solution, the hexavalent chromium from the leachate reacts with the diphenylcarbazide and the reaction forms the purple-red complex. At the wavelength of 540 nm, perform spectrophotometric determination.

3 Reagents and Materials

Unless otherwise stated, in analysis, it can only use the reagents, which are confirmed as the analytic reagents and distilled water, or deionized water, or the water with the equal purity.

3.1 Nitric acid - guarantee reagent.

3.2 Sulfuric acid - guarantee reagent.

3.3 Sodium hydroxide.

3.4 Anhydrous sodium carbonate.

3.5 Dipotassium phosphate.

3.6 Potassium dihydrogen phosphate.

3.7 Potassium dichromate - reference substance.

3.8 Diphenylcarbazide.

5 Analysis Steps

5.1 Sample Preparation

5.1.1 Metal Material

Process the sample into the crumb or strip whose diameter are no more than 1 mm and whose length are no more than 5 mm.

5.1.2 Other Materials

Use the crushing apparatus, such as electric saw and grinder, etc. to grind the sample into the powder-like, and the powder's particle size shall be not more than 1 mm.

5.2 Sample extraction

Accurately weigh and take two 0.3g~1.0g of the processed sample - accuracy: 0.1mg; place them in a conical flask; add 25.0mL of leaching solution (3.12) and 0.5mL of buffer solution (3.13). Leaching solution shall completely immerse the sample; shake it evenly; in a 90°C~95°C water bath kettle [or the oscillating water bath kettle (4.2)], continuously stir it for 1.5h; take it out; cool it to the room temperature; filter it; use water to wash the conical flask and sample; collect the filtrate and washing liquid into a beaker; dropwise-add 5mol/L nitric acid solution (3.11); use the acidity meter (4.3) to test and control solution's pH value at 7.5 ± 0.5 . If the flocculent precipitate occurs, it shall be filtered again and shall retain the filtrate. At the same time, perform reagent's blank test.

5.3 Color Rendering

Add 2.0mL of diphenylcarbazide (DPC) solution (3.16) into the filtrate (5.2); mix it evenly; add sulfuric acid solution (3.10); make filtrate's pH reach 2 ± 0.5 ; then transfer the whole filtrate into a 100mL volumetric flask; use water to dilute it until the mark; shake it evenly; place it for 0.5h.

5.4 Calibration Solution

Respectively pipette 0.0mL, 2.0mL, 4.0mL, 6.0mL, 8.0mL and 10.0mL of standard solution of hexavalent chromium (3.15) into the 100mL volumetric flask; add 50mL of water; add 2.0mL of diphenylcarbazide (DPC) solution (3.16); mix it evenly; add 2 drops of sulfuric acid solution (3.10); make filtrate's pH reach 2 ± 0.5 ; use water to dilute it until the mark; shake it evenly; place it for 0.5h.

In this calibration solution series, their hexavalent-chromium's concentration was 0.0mg/L, 0.1mg/L, 0.2mg/L, 0.3mg/L, 0.4mg/L and 0.5mg/L respectively.

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