Chromium, hexavalent, colorimetric, diphenylcarbazide

Parameter and Code:

Chromium, hexavalent, dissolved, I-1230-85 (μ g/L as Cr⁺⁶): 01032

1. Application

This method may be used to analyze most natural water containing from 50 to 4,000 μ g/L hexavalent chromium. Samples containing higher concentrations must first be diluted.

2. Summary of method

2.1 This method determines only hexavalent chromium in solution.

2.2 In acid solution, diphenylcarbazide and hexavalent chromium form a soluble red-violet product that absorbs light at 540 nm. The pH of the reaction is not particularly critical; solutions differing in pH from 0.7 to 1.3 give identical colors. The color of the chromium-diphenylcarbazide product changes slightly with time, but for practical purposes it can be considered stable.

2.3 Additional information on the principle of the determination is given by Sandell (1950).

3. Interferences

For all practical purposes the reaction is specific for chromium; metallic interference almost never occurs. Iron, mercury, and molybdenum in concentrations as high as $100,000 \ \mu g/L$ show only a small effect. Vanadium should not be present in concentrations exceeding $4,000 \ \mu g/L$. The effect of water color is small, and color as much as 50 color units (Hazen scale) is tolerable. The chromium color develops almost instantly and is stable; whereas, vanadium color develops instantly and then fades rapidly. If the original vanadium concentration is less than $4,000 \ \mu g/L$, no vanadium color persists after 10 min.

4. Apparatus

4.1 Spectrometer for use at 540 nm.

4.2 Refer to the manufacturer's manual for optimizing instrument.

5. Reagents

5.1 Chromium standard solution, 1.00 mL= 100 μ g Cr⁺⁶: Dissolve 0.2829 g primary standard K₂Cr₂O₇, dried for 1 h at 180 °C, in demineralized water and dilute to 1,000 mL.

5.2 Diphenylcarbazide reagent: Dissolve 0.2 g diphenylcarbazide and 1.0 g phthalic anhydride in 200 mL ethanol. This reagent is stable for several weeks; a slight discoloration will not impair the usefulness of the reagent.

5.3 Sulfuric acid, 1.2M: CAUTIOUSLY, add 6.5 mL concentrated H_2SO_4 (sp gr 1.84) to demineralized water and dilute to 100 mL.

6. Procedure

6.1 Pipet a volume of sample containing less than 40 μ g Cr⁺⁶ (10.0 mL max) into a 50-mL beaker, and adjust the volume to 10.0 mL with demineralized water.

6.2 Prepare a blank of demineralized water and sufficient standards, and adjust the volume of each to 10.0 mL with demineralized water.

6.3 Add 1.0 mL 1.2M H₂SO₄ and mix.

6.4 Add 0.5 mL diphenylcarbazide reagent and mix.

6.5 Allow to stand 10 min.

6.6 Determine the absorbance of the sample and standards against the blank, and when necessary make a correction for water color.

7. Calculations

7.1 Determine micrograms hexavalent chromium in the sample from a plot of absorbances of standards.

7.2 Determine the hexavalent chromium concentration in micrograms per liter as follows:

$$Cr^{+6} (\mu g/L) = \frac{1,000 \times \mu g Cr^{+6} \text{ in sample}}{mL \text{ sample}}$$

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8. Report

Report chromium, dissolved hexavalent (01032), concentrations as follows: 50 to 100 μ g/L, nearest 10 μ g/L; 100 μ g/L and above, two significant figures.

9. Precision

Precision expressed in terms of the percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
5	810	5
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Sandell, E. B., 1950, Colorimetric determination of traces of metals (2d ed.): New York, Interscience Publishers, p. 260.