

Chromium, hexavalent, atomic absorption spectrometric, chelation extraction

Parameter and Code:

Chromium, hexavalent, dissolved, I-1232-85 ($\mu\text{g/L}$ as Cr): 01032

1. Application

1.1 This method may be used to analyze water and brines containing from 1 to 25 $\mu\text{g/L}$ of chromium. Samples containing more than 25 $\mu\text{g/L}$ need to be diluted prior to chelation-extraction.

1.2 If the iron concentration of the sample exceeds 5,000 $\mu\text{g/L}$, determine hexavalent chromium by the colorimetric diphenylcarbazide method (I-1230).

2. Summary of method

Hexavalent chromium is determined by atomic absorption spectrometry. The element is chelated with ammonium pyrrolidine dithiocarbamate (APDC) and extracted with methyl isobutyl ketone (MIBK). The extract is aspirated into the air-acetylene flame of the spectrometer (Midgett and Fishman, 1967).

3. Interferences

Concentrations of iron greater than 5,000 $\mu\text{g/L}$ interfere by suppressing the chromium absorption.

4. Apparatus

4.1 *Atomic absorption spectrometer* equipped with electronic digital readout and automatic zero and concentration controls.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

Grating -----	Ultraviolet
Wavelength -----	357.9 nm
Source (hollow-cathode lamp) -----	Chromium
Oxidant -----	Air
Fuel -----	Acetylene
Type of flame -----	Reducing

4.3 Different burners may be used according to manufacturers' instructions.

5. Reagents

5.1 *Ammonium pyrrolidine dithiocarbamate (APDC) solution*, 1.0 g/100 mL: Dissolve 1.0 g APDC in demineralized water and dilute to 100 mL. Prepare fresh daily.

5.2 *Chromium standard solution I*, 1.00 mL=100 $\mu\text{g Cr}^{+6}$: Dissolve 0.2829 g primary standard $\text{K}_2\text{Cr}_2\text{O}_7$, dried for 1 h at 180°C, in demineralized water and dilute to 1,000 mL.

5.3 *Chromium standard solution II*, 1.00 mL=10.0 $\mu\text{g Cr}^{+6}$: Dilute 100 mL chromium standard solution I to 1,000 mL with demineralized water.

5.4 *Chromium standard solution III*, 1.00 mL=0.10 $\mu\text{g Cr}^{+6}$: Dilute 10.0 mL chromium standard solution II to 1,000 mL with demineralized water.

5.5 *Methyl isobutyl ketone (MIBK)*.

5.6 *Sodium hydroxide solution*, 2.5M: Dissolve 100 g NaOH in demineralized water and dilute to 1 L. Alternately a 2.5M NH_4OH solution may be used. Add 167 mL concentrated NH_4OH (sp gr 0.90) to 600 mL demineralized water. Mix, cool, and dilute to 1 L with demineralized water.

6. Procedure

6.1 Clean all glassware used in this determination with warm, dilute HNO_3 (1+9) and rinse with demineralized water immediately before use.

6.2 Pipet a volume of sample containing less than 2.5 $\mu\text{g Cr}^{+6}$ (100 mL max) into a 200-mL volumetric flask, and adjust the volume to approx. 100 mL.

6.3 Acidify a liter of demineralized water with 1.5 mL concentrated HNO_3 (sp gr 1.41). Prepare a blank and at least six standards containing from 1 to 25 $\mu\text{g/L}$ of Cr^{+6} , and adjust the volume of each to approx. 100 mL with the acidified demineralized water.

6.4 With a pH meter, adjust the pH of each solution to 2.4 by dropwise addition of 2.5M NaOH or NH_4OH .

6.5 Add 5.0 mL APDC solution and mix.

6.6 Add 10.0 mL MIBK and shake vigorously for 3 min.

6.7 Allow the layers to separate and add demineralized water until the ketone layer is completely in the neck of the flask. The Cr^{+6} -APDC complex is stable for at least 36 h.

6.8 Aspirate the ketone layer of the blank to set the automatic zero control. Use the automatic concentration control to set the concentrations of standards. Use at least six standards. Calibrate the instrument each time a set of samples is analyzed and check calibration at reasonable intervals.

7. Calculations

Determine the micrograms per liter of hexavalent chromium in each sample from the

digital display or printer while aspirating each sample. Dilute those samples containing chromium concentrations that exceed the working range of the method; repeat the chelation-extraction and multiply by the proper dilution factor.

8. Report

Report chromium, dissolved, hexavalent (01032), concentrations as follows: less than 10 $\mu\text{g/L}$, nearest microgram per liter; 10 $\mu\text{g/L}$ and above, two significant figures.

9. Precision

It is estimated that the precision of this method for Cr^{+6} is equal to total chromium by the atomic absorption spectrometric chelation-extraction method.

Reference

Midgett, M.R., and Fishman, M. J., 1967, Determination of total chromium in fresh waters by atomic absorption: Atomic Absorption Newsletter, v. 6, p. 128-131.