Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography

Thermo Fisher Scientific Inc.

Introduction

Chromium exists in the environment in either the trivalent, Cr(III), or hexavalent, Cr(VI), form. Cr(III) is considered to be essential to mammals for the maintenance of glucose, lipid, and protein metabolism. On the other hand, Cr(VI) is known to have an adverse affect on the lungs, liver, and kidneys. The method presented here provides a sensitive and selective means of determining Cr(VI) as the chromate anion CrO_4^{2-} down to the 1 um/L level in a variety of environmental matrices.[1,2]

This method has been documented in several standard methodologies.[1,3,4,7] It has been validated over the range of 1–1000 µg/L in both wastewater and drinking water[5] with a method detection limit in the range of 1 µg/L (see Figure 1). It has also been recommended for use in determining Cr(VI) in extracts of ambient air particulates[6] as well as air particulates from waste incinerators.[7] For a more complete discussion of this method, please refer to Dionex™ (now part of Thermo Scientific™) Technical Note 26: *Determination of Cr(VI) in Water, Wastewater, and Solid Waste Extracts*.

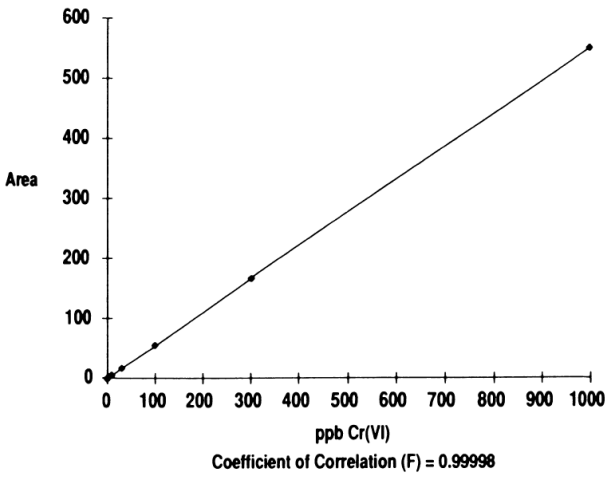

Figure 1. Area response for colorimetric chromium detection.

Equipment

Any Dionex chromatographic system* configured with:

- UV/Visible absorbance detector (VDM-2)
- Postcolumn reagent delivery system

**Equivalent or improved results can be achieved using the Thermo Scientific Dionex ICS-2100 or the Thermo Scientific Dionex ICS-5000⁺ system.*

Reagents and Standards

- Ammonium sulfate
- Ammonium hydroxide
- 1,5-Diphenylcarbazide
- Methanol, HPLC grade
- Sulfuric acid

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Conditions	
Columns:	Thermo Scientific™ Dionex™ IonPac™ AS7 analytical and Dionex IonPac NG1 guard
Eluent:	250 mM Ammonium sulfate, 100 mM Ammonium hydroxide
Eluent Flow Rate:	1.5 mL/min
Expected Backpressure:	900–1100 psi (6.2–7.6 kPa)
Postcolumn Reagent:	2.0 mM Diphenylcarbazide, 10% methanol, 1.0 N sulfuric acid
Postcolumn Reagent Flow Rate:	0.5 mL/min
Detection:	Visible, 530 nm
Sample Volume:	50 µL

Preparation of Solutions and Reagents

Eluent:

250 mM Ammonium sulfate
100 mM ammonium hydroxide

Dissolve 33 g of ammonium sulfate in 500 mL of water and add 6.5 mL of ammonium hydroxide. Dilute to 1 L with water.

Postcolumn Reagent:

2.0 mM Diphenylcarbazide
10% methanol
1.0 N sulfuric acid

Dissolve 0.50 g of 1,5-diphenylcarbazide in 100 mL of HPLC-grade methanol. Add 500 mL of water containing 28 mL of 98% sulfuric acid while stirring. Dilute with water to 1 L in a volumetric flask. This reagent is stable for four or five days, but should only be prepared in 1 L quantities as needed.

Standard:

1000 ppm Cr(VI)

Dissolve 0.283 g of potassium dichromate ($K_2Cr_2O_7$) that has been dried at 100 °C for one hour in water. Dilute to 100 mL in a volumetric flask.

Working standards are prepared by appropriate dilutions of the stock solutions. As an example, for a 1 ppm Cr(VI) standard, pipet 1.00 mL of the chromium stock solution into a 1 L volumetric flask. Dilute to 1 L with water.

Discussion

An aqueous sample is injected onto a high capacity anion exchange column where Cr(VI) as CrO_4^{2-} is retained and then eluted with an alkaline sulfate eluent. After this separation, a diphenylcarbazide color reagent is added to the eluent stream, which then flows through a photometric detector. The regent forms a color complex with Cr(VI), which is detected by absorbance at 530 nm. An example of hexavalent chromium in a aqueous sample is shown in Figure 2.

This method is not applicable to the determination of total chromium in environmental samples. For regulatory purposes, total chromium is traditionally defined as the sum of the free and combined chromium from both the liquid and solid portions of the sample. Accordingly, the sample must undergo a strong acid digestion to release the chromium for analysis. This step converts all chromium to the Cr(III) form. Other methods can be used to determine the total chromium in the digest solution.

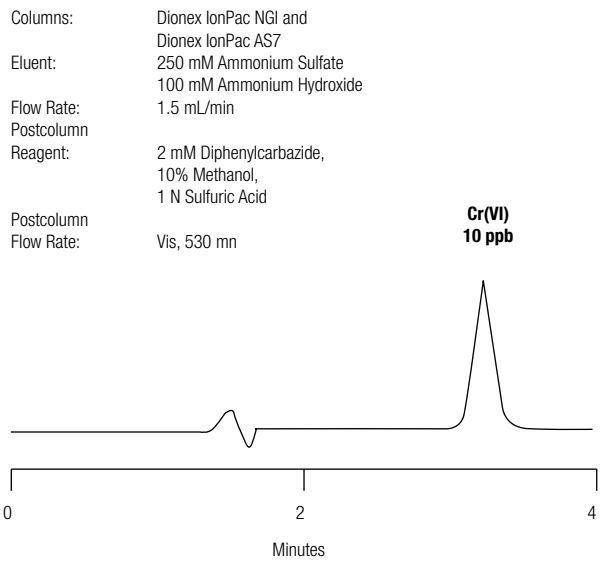

Figure 2. Determination of Cr(VI) in water, wastewater, and solid waste extracts.

References

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