Determination of hexavalent chromium in drinking water according to EPA Method 218.7

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Ion Chromatography

Summary

Chromium(VI) compounds are highly water-soluble and oxidizing and are considered toxic and potentially carcinogenic. Chromatographic determinations apply anion-exchange chromatography followed by 1,5-diphenylcarbazide post-column chemistry according to U.S. EPA Method 218.7.

This poster looks at the possibility to modify the existing EPA Method to meet California`s rigorous public health goal (PHG) of 0.02 µg/L. After optimizing instrument settings and method parameters, a method detection limit (MDL) of 0.01 µg/L has been obtained. The microbore column used guaranteed precise and accurate results within less than six minutes. The applied flow-through reactor and the UV/VIS detector allowed to significantly enhance the efficiency of the post-column reaction.

Introduction

Have you seen the movie “Erin Brockovich”? It is based on a true story and tells the eponymous protagonist`s fight against a U.S. energy corporation that had poisoned the local water supply with toxic chromium(VI).

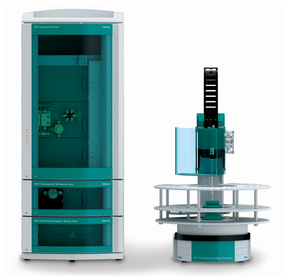
Chromium is one of the essential micronutrients required by living cells to convert fat into energy. Chromium required by living cells is supposed to be in its +3 oxidation state; chromium in its +6 oxidation state, however, is regarded as highly toxic and carcinogenic and significantly increases risks for liver and kidney diseases.

With ion chromatography, chromium(VI) is detected with high sensitivity as a magenta-colored chromium-diphenylcarbazone complex at 530 nm after separation on an anion-exchange column and subsequent post-column derivatization with 1,5-diphenylcarbazide.

The environmental working group (EWG) used California`s «safe limit» of 0.06 µg/L when they conducted their 35-city test of municipal drinking water. Their findings showed 31 of those cities had contamination readings above this limit, which prompted the EPA to re-evaluate Method 218.6. U.S. EPA and Metrohm are currently working on improving chromium(VI) analysis. Without compromising the core principle of existing EPA Method 218.6, the margins for using different eluents, columns, and injection volumes are widened. This paper presents the latest results on chromium(VI) testing.

Instrumentation

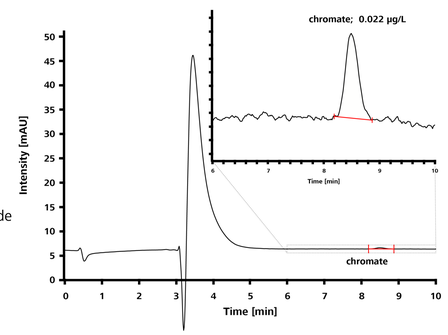
- 940 Professional IC Vario
- 943 Professional Reactor Vario
- 944 Professional UV/VIS Detector Vario
- 858 Professional Sample Processor



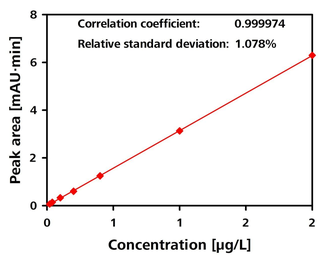
Post-column reaction

After separation on an anion-exchange column, chromium(VI) is derivatized with 1,5-diphenylcarbazide in a post-column reaction. The generated magenta-colored chromium-diphenylcarbazone complex is detected by a UV/VIS detector at 530 nm. Instrument settings and method parameters have been optimized in terms of separation column, column oven temperature, wavelength, and flow rates.

0.022 µg/L chromium standard	
Column:	Metrosep A Supp 5 - 250/4.0
Column temp.:	45 °C
Eluent:	12.8 mmol/L Na_2CO_3 4.0 mmol/L $NaHCO_3$ 2.5 g/L $(NH_4)_2SO_4$
Flow:	0.8 mL/min
Loop:	2 mL
PCR volume:	0.39 cm³, 0.5 mm i.d. × 2 m
PCR reagent:	2 mmol/L 1,5-diphenylcarbazide (acidified with 98% H_2SO_4)
PCR flow:	0.2 mL/min
PCR temp.:	45 °C
Wavelength:	538 ± 21 nm

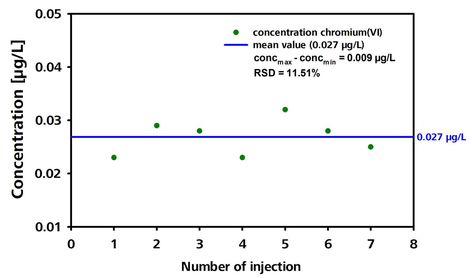


Calibration



Precision

Precision was determined by sevenfold injection of a 0.027 µg/L chromium(VI) standard.



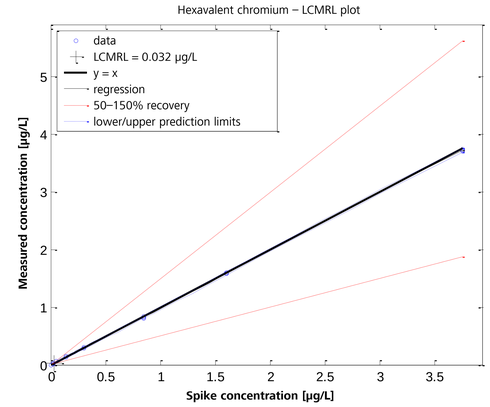
Method detection limit (MDL)

The method detection limit was calculated from the resulting standard deviation of seven replicates of reagent water fortified with two chromate(VI) concentration levels in the ppt range. For 0.025 and 0.05 µg/L chromium(VI) spikes, the determined MDLs were 0.008 and 0.01 µg/L, respectively, which are both below the current California public health goal (PHG) of 0.02 µg/L.

EPA`s lowest concentration minimum reporting level (LCMRL)

EPA has developed a user-friendly and freely available downloadable software for determining single-laboratory lowest concentration minimum reporting levels (LCMRLs). The idea behind involves a much better understanding and control of measurement data quality to facilitate decision-making.

The LCMRL is defined as the lowest spiking concentration at which a laboratory can reliably achieve the measurement quality objective of 50 to 150% recovery. Unlike the MDL, which only considers the standard deviation of repeated measurements of low-level spikes, the MRL additionally includes the accuracy of the measurement.



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References

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(2) Determination of hexavalent chromium in drinking water by ion chromatography with post-column derivatization and UV-visible spectroscopic detection, U.S. Environmental Protection Agency, Method 218.7, Version 1.0, Cincinnati, Ohio, USA (2011).

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