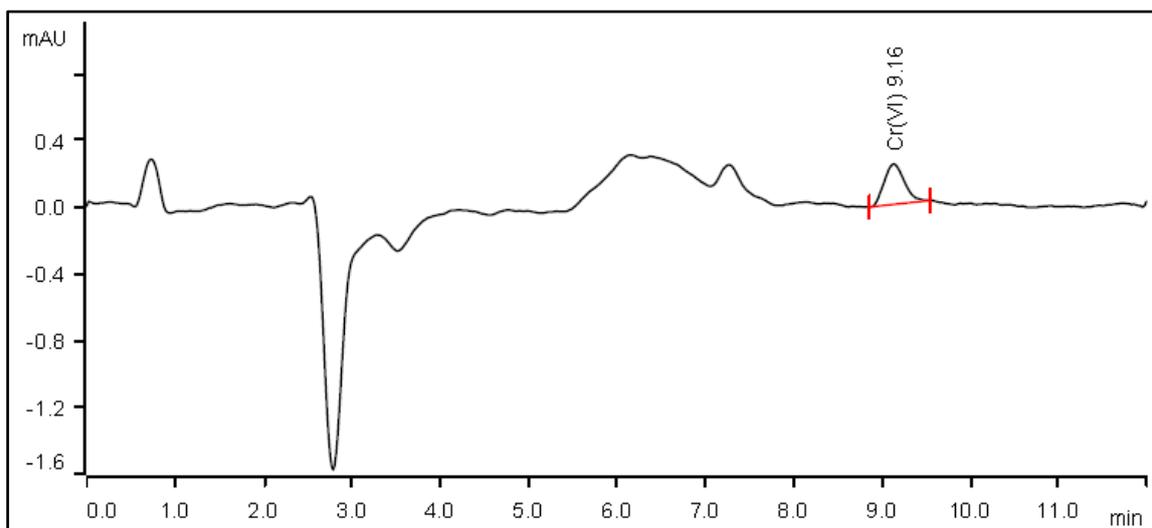


Determination of hexavalent chromium (Cr⁺⁶) by USEPA 218.7



Chromium exists in the environment in two basic forms including trivalent chromium (Cr⁺³) and hexavalent chromium (Cr⁺⁶). While Cr⁺³ is a beneficial nutrient to humans, significant study into the detrimental effects of Cr⁺⁶ in humans has labeled hexavalent chromium as carcinogenic. The EPA's new federal mandate, in addition to growing public concern over Cr⁺⁶, is now persuading municipal water systems to monitor Cr⁺⁶ down to sub-parts-per-billion (ppb) levels. Use of ion chromatography (IC) coupled with post column reaction and UV detection is demonstrated according to EPA 218.7.

Results

Sample	Average injections n=60	Standard deviation n=60	Relative standard deviation n=60
0.020 ppb Cr ⁺⁶ in water	0.023 ppb	0.007 ppb	28.7 %

Sample	Cr+6 Results	Spike	Spike Recovered
Filtered fountain water	0.032 ppb	0.020 ppb	105 %
Tap Water	0.044 ppb	0.020 ppb	109 %

Sample

Stock standard of 0.020 ppb Cr⁶⁺, drinking fountain filtered water, and tap water.

Measurement and data

Separation was performed using the Metrosep A Supp 10 - 250/2.0 using an eluent consisting of 100 mmol/L ammonium hydroxide and 100 mmol/L ammonium sulfate prepared from high purity ammonium sulfate salt and reagent grade ammonium hydroxide (28-30% ammonia basis). A post column reagent containing 10% methanol, 500 mmol/L sulfuric acid, and 2 mmol/L 1,5-diphenylcarbazide was used for post-column derivatization of Cr⁶⁺. A peristaltic pump was used to deliver PCR reagent and was mixed with eluent flow downstream of the separation column. Detection of derivatized Cr⁶⁺ was accomplished using the 944 UV-Vis spectrophotometric detector set to monitor absorbance at 530 nm. Standards were prepared to calibrate the instrument over a concentration range of 0.020 ppb to 5 ppb Cr⁶⁺. Detection limits of 0.020 ppb were achieved and instrument stability was tested by analyzing 60 replicate injections of 0.020 ppb Cr⁶⁺. Samples of water from a filtered drinking fountain and from tap water were spiked with 0.020 ppb Cr⁶⁺ to determine recovery; recoveries of 105% and 109% of the spikes samples were determined.

Sample preparation

Direct injection

Columns

Metrosep A Supp 10 - 250/2.0	6.1020.230
Metrosep A Supp 10 Guard/2.0	6.1020.600

IC solutions

Eluent	10 mmol/L ammonium hydroxide + 10 mmol/L ammonium sulfate
PCR Reagent	2 mmol/L 1,5-diphenylcarbazide + 500 mmol/L sulfuric acid + 10% methanol

Parameters

Flow rate	0.3 mL/min
Sample loop	1 mL
Column temperature	50 °C
Recording time	12 min
UV/Vis Wavelength	530 +/- 15 nm
Flow rate (PCR Reagent)	0.2 mL/min

Instrumentation

930 Compact IC Flex Oven/SeS/Deg	2.930.2460
944 Professional UV/VIS Detector Vario	2.944.0010
919 IC Autosampler plus	2.919.0020
Sample rack 148 x 11 mL + 3 x 300 mL	6.2041.440
Kit for hexavalent chromium system with low pressure delivery	KIT-IC0040

Author

Jay Sheffer
Applications Specialist
Metrohm USA
October 2016

www.metrohmusa.com

