Determination of Hexavalent Chromium Cr(VI) in Drinking Water by Suppressed Conductivity Detection

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Key Words

Dionex IonPac AS11-HC and AS11-HC-4µm Columns, Chromate, High-Ionic-Strength Water (HIW), LCMRL, Dionex ICS-5000+ HPIC System

Goal

To develop an ion chromatography (IC) method that uses suppressed conductivity detection for the determination of Cr(VI) in drinking water

Introduction

Two ionic forms of chromium—trivalent chromium, Cr(III), and hexavalent chromium, Cr(VI)—are present in various forms in soil, water, and the biota. Cr(III) and Cr(VI) occur naturally in the environment, being found in water from the erosion of chromium deposits in rocks and soils. Cr(VI) is also produced by industrial processes and manufacturing activities, including discharges from steel and pulp mills.[1] Cr(III) is a nutritionally essential element in humans and is often added to vitamins as a dietary supplement. Cr(III) has relatively low toxicity and is a concern in drinking water only at very high levels of contamination, whereas Cr(VI) is more toxic and poses potential health risks.[2,3]

Chromates are oxyanions (e.g., CrO_4^{2-}, $Cr_2O_7^{2-}$) of chromium in the oxidation state of +6. All Cr(VI) compounds are strong oxidizing agents that are considered toxic and potentially carcinogenic.[4] Hence, chromates are regulated in the environment and are a primary drinking water contaminant in the U.S.[5,6] For example, in 1999, the state of California established a public health goal (PHG) of 0.2 µg/L (ppb) for Cr(VI) and 2.5 µg/L for total chromium.[7] The PHG is based on an estimated one-in-one-million lifetime cancer risk level. In July 2011, the Office of Environmental Health Hazard Assessment (OEHHA) finalized the PHG for Cr(VI) at 0.020 µg/L.[8]

Drinking water standards are regularly re-evaluated by the U.S. Environmental Protection Agency (EPA). Currently, dissolved Cr(VI) is measured as chromate according to U.S. EPA Method 218.7.[9] This method is a modified version of U.S. EPA Method 218.6 and is based on anion-exchange chromatography.[10–12] The approach uses a Thermo Scientific™ Dionex™ IonPac™ AS7 column (4 × 250 mm) and detection after postcolumn reaction with diphenylcarbazide (DPC), which yields a compound with visible absorbance at 530 nm. Using EPA Method 218.7, detection limits for Cr(VI) fortified into reagent water ranged from 0.0044 to 0.015 µg/L.[9]

Although the PHG for Cr(VI) in drinking water in California is 0.020 µg/L, the current regulatory maximum contaminant level (MCL) is 10 µg/L.[13] Application Update (AU) 179 describes a method that uses postcolumn reaction with DPC and has a method detection limit (MDL) of 0.001 µg/L. The AU 179 method allows a minimum quantitation limit of 0.003 µg/L, which is more than sufficient for the proposed California PHG of 0.02 µg/L.[14] Determination of Cr(VI) at the California PHG cannot be accomplished with a direct injection of drinking water, separation on a single anion-exchange column, and detection by suppressed conductivity. However, it may be possible to design such a method to determine Cr(VI) at the 10 µg/L MCL regulatory limit.

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The method described here uses suppressed conductivity detection for the determination of Cr(VI) in drinking water, thus eliminating the postcolumn derivitization used in EPA Methods 218.6 and 218.7. This new approach uses either the Dionex IonPac AS11-HC or AS11-HC-4µm column to separate chromate from other anions in HIW. The eluent is automatically generated by a Reagent-Free™ IC (RFIC™) system for added convenience. Even in the challenging HIW sample, the method can easily detect and quantify 2 µg/L Cr(VI). Using RFIC systems and hydroxide eluents provides lower detection limits since the suppression product is water, which has a lower background conductance than a carbonate based eluent approach. The suppression product using a carbonate based eluent is carbonic acid, which has a higher background conductance making lower detection limits much more challenging.

Equipment

- A Thermo Scientific™ Dionex™ ICS-5000⁺ HPIC™ system,* capable of performing high-pressure IC, including:
  - DP Dual pump
  - EG Eluent Generator
  - DC Detector/Chromatography Compartment
- Thermo Scientific Dionex AS-AP Autosampler with a 5 mL syringe (P/N 074308) and 8500 µL buffer line assembly (P/N 075520)
- Thermo Scientific Dionex EGC 500 Potassium Hydroxide (KOH) Eluent Generator Cartridge (P/N 075778)
- Thermo Scientific™ Dionex™ CR-ATC 500 Continuously Regenerated Anion Trap Column (P/N 075550)
- Vial Kit, polystyrene with caps and blue septa, 10 mL (P/N 074228)
- Thermo Scientific™ Dionex™ AERS™ 500 Anion Electrolytically Regenerated Suppressor (P/N 082541)
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software version 7.2

* This method can be run on any system capable of eluent generation.

Reagents and Standards

- Deionized (DI) water, 18 MΩ-cm or better
- Ammonium Sulfate (Fisher Scientific P/N A701-3)
- Ammonium Hydroxide (Fisher Scientific P/N 08-901-802)
- Potassium Dichromate, Fine Granules (Fisher Scientific P/N P186-3)
- Sodium and Potassium salts, ACS grade, for preparing high ionic strength water and anion standards
- Dionex™ Fluoride Standard, 1000 mg/L (P/N 37158)
- Dionex™ Chloride Standard, 1000 mg/L (P/N 37159)
- Dionex™ Sulfate Standard, 1000 mg/L (P/N 37160)
- Nitrate Standard, 1000 mg/L (Fisher Scientific P/N AS-NO39)
- Phosphate Standard, 1000 mg/L (Fisher Scientific P/N AS-NO49)
- Bromide Standard, 1000 mg/L (Fisher Scientific P/N US-ICC-001)

Conditions	
Column Set 1:	Dionex IonPac AG11-HC, Guard, 2 × 50 mm (P/N 052963) Dionex IonPac AS11-HC, Analytical, 2 × 250 mm (P/N 052961)
Column Set 2:	Dionex IonPac AG11-HC-4µm, Guard, 2 × 50 mm (P/N 078036) Dionex IonPac AS11-HC-4µm, Analytical, 2 × 250 mm (P/N 078035)
Eluent:	45 mM KOH, isocratic
Eluent Source:	Dionex EGC 500 KOH cartridge with Dionex CR-ATC trap column
Flow Rate:	0.38 mL/min
Injection Volume/ Loop Size:	300 µL
Inject Mode:	Push Full
Temperature:	30 °C
Loop Overfill Factor:	5
Sample Adjustment Buffer:	Ammonium hydroxide/ammonium sulfate
Detection:	Suppressed conductivity, Dionex AERS 500 suppressor in recycle mode, 186 mA
System Backpressure:	Column Set 1, ~2050 psi Column Set 2, ~4650 psi
Background Conductance:	~1.1–1.2 µS
Noise:	Column Set 1, 0.7–1.8 nS/min, peak-to-peak Column Set 2, ~0.5–1.3 nS/min, peak-to-peak
Run Time:	10 min

Preparation of Solutions and Reagents

Sample Adjustment Buffer

- 250 mM Ammonium Sulfate
- 1000 mM Ammonium Hydroxide

Dissolve 3.3 g of ammonium sulfate in ~75 mL of DI water and add 6.5 mL of 29% ammonium hydroxide. Dilute to 100 mL with DI water.

Standard Solutions

Add 0.283 g of potassium dichromate (dried at 100 °C to a constant weight) to ~50 mL of DI water in a 100 mL volumetric flask to make 1000 mg/L of stock standard. Dissolve and bring to volume with DI water. Store the stock standard at 4 °C. Adjust the pH to 9.0–9.5 by adding 1 mL of sample adjustment buffer per 100 mL of final volume before bringing to final volume. Prepare working standards fresh daily.

High-Ionic-Strength Water (HIW)

HIW is defined in EPA Method 300.1 as simulated drinking water prepared from DI water fortified with chloride (100 mg/L), nitrate (10 mg/L as N), phosphate (10 mg/L as P), sulfate (100 mg/L), and carbonate (100 mg/L). For this study, prepare HIW from DI water fortified with fluoride (1 mg/L), nitrite (0.1 mg/L), and bromide (0.02 mg/L) in addition to the ions specified in EPA Method 300.1. Prepare HIW by diluting appropriate volumes of the 1000 mg/L stock standards with DI water. For several of the analytes of interest, 1000 mg/L standard solutions are available from Fisher Scientific and other commercial sources. When commercial standards are not available, 1000 mg/L standards can be prepared by dissolving the appropriate amounts of the corresponding mass in 1000 mL of DI water, as described in Application Note 133.[15]

System Preparation and Configuration

Dionex ICS-5000+ HPIC System

Install and configure the Dionex AS-AP Autosampler in Push Full mode. Follow the instructions in the Dionex AS-AP Autosampler Operator's Manual (Document No. 065361) to calibrate the sample transfer line to ensure accurate and precise sample injections.

Prepare the Dionex AERS 500 suppressor for use by hydrating the internal membrane. Push 3 mL of DI water through the Eluent Out port and 5 mL of DI water through the Regen In port. Allow the suppressor to sit for 20 min to ensure complete hydration before installing it in the system.

Condition the Dionex EGC 500 KOH cartridge before first use by running 50 mM KOH at 1 mL/min for 45 min. For more information on installation and operation of a Dionex EGC 500 KOH cartridge, consult the product manual (Document No. 065018-04).

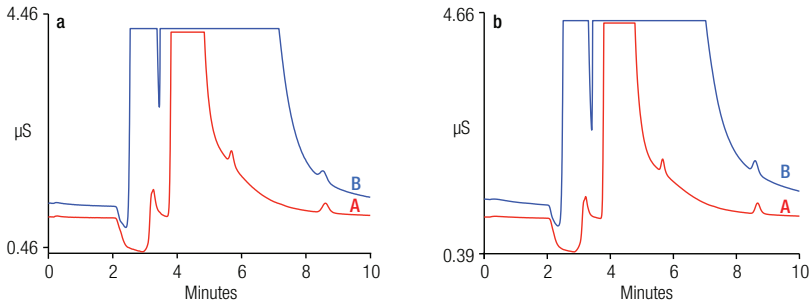
Install the Dionex IonPac AG11-HC-4µm (2 × 50 mm) and the Dionex IonPac AS11-HC-4µm (2 × 250 mm) columns in the lower compartment of the DC. After connecting the inlet of the column, pump 50 mM KOH through the column with the outlet directed to waste for at least 30 min before connecting the column outlet to the suppressor using 0.005 in. i.d. PEEK tubing.

Equilibrate the column with eluent for 60 min and run a system blank. The system will display the background conductance and noise listed under Conditions. Inject the Dionex IonPac AS11-HC-4µm column Quality Assurance Report (QAR) standard mix. The column is equilibrated when three consecutive injections of the standard produce the same retention times for all analytes. Confirm that the resulting chromatogram resembles the chromatogram shown in the QAR that comes with the column. Note that the chromatogram shown in the QAR is generated without the guard column; thus, analyte retention times will be longer than those shown in the QAR.

Results and Discussion

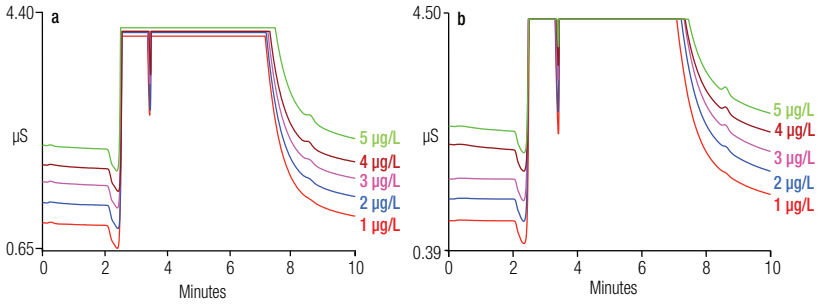
A number of high-capacity Dionex IonPac columns were evaluated for this analysis. The Dionex IonPac AS11-HC column was chosen because it achieved the best separation of chromate from other late-eluting anions, such as sulfate and phosphate, especially with the HIW sample. After completing method development using the Dionex IonPac AS11-HC column, the method was adapted to the Dionex IonPac AS11-HC-4µm column.

Figure 1 shows chromatograms of a 10 µg/L Cr(VI) as CrO_4^{2-} standard in DI water and in HIW using the Dionex IonPac AS11-HC and AS11-HC-4µm columns, respectively. The elution time for chromate was 8.60 and 8.65 min on the Dionex IonPac AS11-HC and AS11-HC-4µm columns, respectively. A slight shift (0.09 min) in the retention time for chromate in the HIW matrix was observed on both columns. Figure 2 shows the chromatograms obtained for HIW spiked with Cr(VI) at 1, 2, 3, 4, and 5 µg/L on the Dionex IonPac AS11-HC and AS11-HC-4µm columns, respectively.



A 5% signal offset has been applied.

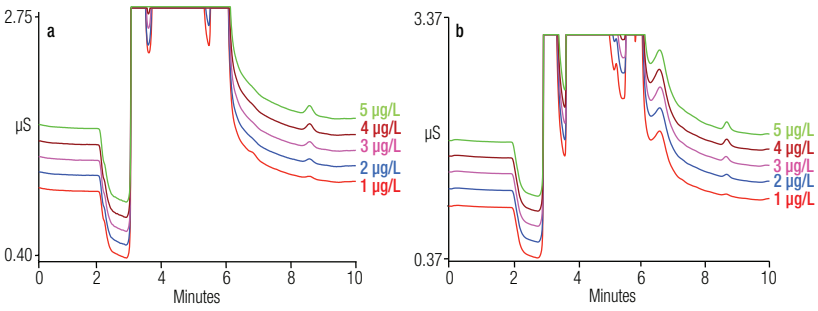
Figure 1. 10 µg/L Cr(VI) spiked in (A) DI water and (B) HIW using (a) the Dionex IonPac AS11-HC column and (b) the Dionex IonPac AS11-HC-4µm column.



A 5% signal offset has been applied.

Figure 2. Cr(VI) spiked in HIW at 1, 2 , 3 , 4, and 5 µg/L using (a) the Dionex IonPac AS11-HC column and (b) the Dionex IonPac AS11-HC-4µm column.

Figure 3 shows the same analysis as shown in Figure 2, but with Sunnyvale, CA drinking water substituted for the HIW sample. This sample has fewer early eluting anions (2–6 min) than the HIW sample and they did not impact the chromate peak eluting at ~8.6 min. The calibration curves (Figure 4) are linear over the calibration range 1–30 µg/L for Cr(VI) as CrO_4^{2-}, with coefficients of determination greater than 0.9994 and 0.9993, respectively, for the Dionex IonPac AS11-HC and AS11-HC-4µm columns.



A 5% signal offset has been applied.

Figure 3. Cr(VI) spiked in Sunnyvale drinking water at 1, 2 , 3 , 4, and 5 µg/L using (a) the Dionex IonPac AS11-HC column and (b) the Dionex IonPac AS11-HC-4µm column.

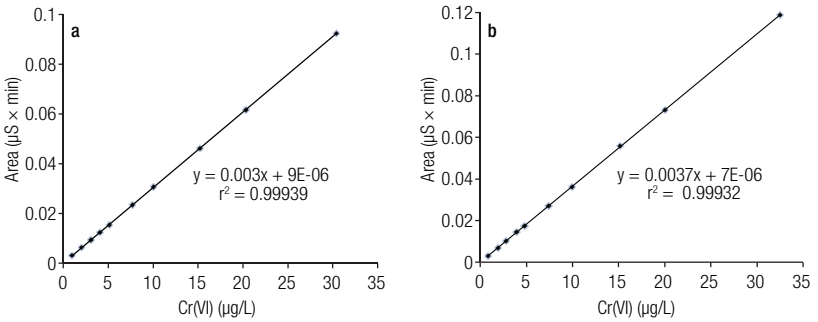


Figure 4. Calibration curves for Cr(VI) using (a) the Dionex IonPac AS11-HC column and (b) the Dionex IonPac AS11-HC-4µm column.

Table 1. Results of spike recoveries for Cr(VI) in water samples.

Sample	Dionex IonPac AS11-HC Column (2 × 250 mm)			Dionex IonPac AS11-HC-4µm Column (2 × 250 mm)		
	Spiked (µg/L)	Found (µg/L)	Recovery (%)	Spiked (µg/L)	Found (µg/L)	Recovery (%)
10 µg/L Cr(VI) in HIW	9.96	9.02	90.5	9.94	10.37	105
5 µg/L Cr(VI) in HIW	5.01	4.02	80.1	5.03	5.07	101
2 µg/L Cr(VI) in HIW	2.07	1.17	56.8	1.99	1.60	80.5
10 µg/L Cr(VI) in Sunnyvale Tap Water	10.4	11.4	110	10.7	10.9	102
5 µg/L Cr(VI) in Sunnyvale Tap Water	5.04	5.41	107	5.02	5.06	101
2 µg/L Cr(VI) in Sunnyvale Tap Water	2.14	2.05	96.2	2.01	1.95	96.8

For recovery studies, known amounts of chromate standard were spiked into the Sunnyvale drinking water and HIW samples. In the concentration range 1–10 µg/L, the peak response recoveries ranged from 80 to 110%, with the exception of only an approximate 57% recovery of the 2 µg/L spike in the HIW sample (Table 1) on the Dionex IonPac AS11-HC column. This illustrates the necessity to check recovery when determining low concentrations of an analyte in an HIW sample.

To determine the limit of detection (LOD) and limit of quantification (LOQ), baseline noise was first determined by measuring peak-to-peak noise in a representative 1-min segment of the baseline where no peaks elute, but close to the peak of interest. The signal was determined from the average peak height of seven injections of 1 µg/L Cr(VI) spiked in DI water. The LOD and LOQ were then calculated by multiplying the signal-to-noise (S/N) ratio 3× and 10×, respectively.

Table 2 summarizes the results of the calibration, LOD, and LOQ for the two columns. Comparable results were obtained for seven replicate injections of HIW fortified with 2 or 3 µg/L Cr(VI) as CrO_4^{2-}. The LOD and LOQ obtained were 0.18 µg/L and 0.58 µg/L, respectively, for the Dionex IonPac AS11-HC-4µm column. These values are in DI water, and Figure 2 shows that values below 2 µg/L will be difficult to determine in HIW samples using the Dionex IonPac AS11-HC column. The detection limits of this method were also evaluated using the following three other calculations.

Table 2. Results of calibration, LOD, and, LOQ for Cr(VI).

Analyte	Range (µg/L)	Dionex IonPac AS11-HC Column (2 × 250 mm)			Dionex IonPac AS11-HC-4µm Column (2 × 250 mm)		
		$(r^2)^a$	LOD[b] (µg/L)	LOQ[c] (µg/L)	$(r^2)^a$	LOD[b] (µg/L)	LOQ[c] (µg/L)
Cr(VI)	1–30	0.9994	0.34	1.14	0.9993	0.17	0.58

[a] Coefficient of determination (linear with offset)
[b] LOD = 3 × S/N
[c] LOQ = 10 × S/N

Method Detection Limit (MDL)

MDL is a measure of the precision of low-level standard replicate injections and is defined as the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. In this application, the MDL for chromate Cr(VI) as CrO_4^{2-} was determined by analyzing seven replicate injections of HIW fortified with Cr(VI) as CrO_4^{2-} at two concentration levels of 2 and 3 µg/L (i.e., approximately 3–5× the estimated instrument detection limit). Both levels produced a calculated MDL value of ~1 µg/L. This enables a minimum quantitation limit of ~3 µg/L for Cr(VI) as CrO_4^{2-}, which is adequate for routine analysis at the California MCL of 10 µg/L.

Lowest Concentration Minimum Reporting Limit (LCMRL)

LCMRL is defined as the lowest spiking concentration such that the probability of spike recovery in the 50–150% range is at least 99%. For the LCMRL calculations, Cr(VI) was spiked in HIW at six levels (1–10 µg/L) for the Dionex IonPac AS11-HC column, and at seven levels (1–10 µg/L) for the Dionex IonPac AS11-HC-4µm column. Figure 5 shows the LCMRL plots of Cr(VI) spiked in HIW using the Dionex IonPac AS11-HC and AS11-HC-4µm columns. The LCMRL calculated using the EPA's LCMRL calculator for Cr(VI) spiked in HIW was 2.2 and 1.2 µg/L, respectively, when using these two columns (Figure 5).[16]

Hubaux-Vos LOD (H-V LOD)

H-V LOD is a value representing the minimum amount of an analyte that can be detected by a method with a specified level of certainty, given a particular set of calibration data. The H-V LOD is derived from selected values of α and β, where α represents the probability of false positives (reporting detection when no analyte is present) and β represents the probability of false negatives (reporting nothing detected when the analyte is actually present). The selected α and β values are used to calculate upper and lower prediction intervals, respectively.

Here, the α = β = 0.5 value was used to calculate the upper and lower prediction intervals. In other words, the probabilities of false positives and negatives were each 0.5%. From the prediction intervals, the H-V LOD was determined by constructing a horizontal line through the intersection of the upper prediction interval (defined by α) and the response axis, then finding the amount that corresponds to the point where the constructed horizontal line intersects the lower prediction interval (defined by β). Figure 6 shows the H-V LOD graphs of Cr(VI) spiked in HIW on the Dionex IonPac AS11-HC and AS11-HC-4µm columns, respectively. The H-V LODs (graphed and calculated in Chromeleon CDS software, version 7.2) for Cr(VI) in HIW were 1.4 and 0.75 µg/L when using the Dionex IonPac AS11-HC and AS11-HC-4µm columns, respectively.

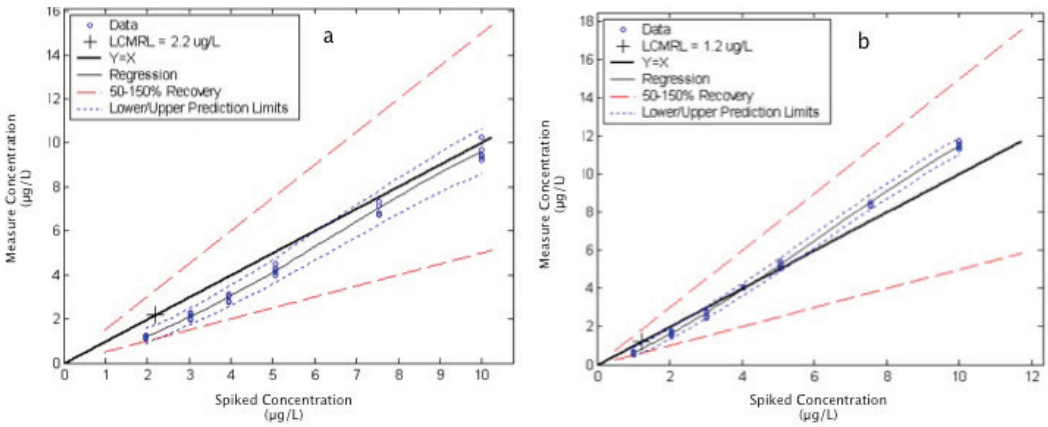


Figure 5. Determination of the LCMRL using the LCMRL calculator. The LCMRL values for chromate equal 2.20 µg/L using (a) the Dionex IonPac AS11-HC column and equal 1.20 µg/L using (b) the Dionex IonPac AS11-HC-4µm column.

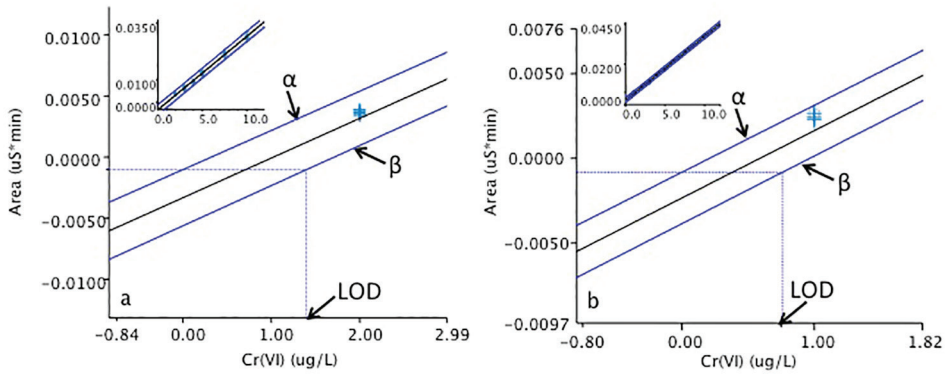


Figure 6. Determination of the H-V LOD using a H-V plot. The H-V LOD values for chromate equal 1.40 µg/L using (a) the Dionex IonPac AS11-HC column and equal 0.75 µg/L using (b) the Dionex IonPac AS11-HC-4µm column.

Conclusion

This study demonstrates the determination of hexavalent chromium (i.e., Cr(VI) as CrO_4^{2-}) in drinking water at the California MCL level of 10 µg/L. This IC method uses suppressed conductivity detection and can be performed using either a Dionex IonPac AS11-HC or AS11-HC-4µm column. The detection limits calculated by different methods are summarized in Table 3 and indicate this method is more than sufficient to determine Cr(VI) at the California drinking water regulatory limit of 10 µg/L. For example, the resulting MDL for Cr(VI) as CrO_4^{2-} at 1 µg/L allows a minimum quantitation limit of 3 µg/L.

Table 3. Detection limits of Cr(VI) calculated by different methods.

Method	Dionex IonPac AS11-HC Column	Dionex IonPac AS11- HC-4µm Column	Comments
	Value (µg/L)	Value (µg/L)	
LOD	0.65	0.23	Seven injections of HIW fortified with 2 or 3 µg/L Cr(VI)
LOQ	2.18	0.77	Seven injections of HIW fortified with 2 or 3 µg/L Cr(VI)
MDL	0.53	0.35	Seven injections of HIW fortified with 2 or 3 µg/L Cr(VI)
LCMRL	2.20	1.20	Five injections of Cr(VI) in HIW at seven levels (1, 2, 3, 4, 5, 7.5, and 10 µg/L)
H-V LOD	1.40	0.75	Five injections of Cr(VI) in HIW at seven levels (1, 2, 3, 4, 5, 7.5, and 10 µg/L)

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AN71491-EN 08/16S