



The Determination of Hexavalent Chromium in Water by Ion Exchange Chromatography Inductively Coupled Plasma Mass Spectrometry (IC-ICP-MS)

Introduction

This method utilizes a hyphenated technique, Ion Exchange Chromatography (IC) coupled to an Inductively Coupled Plasma Mass Spectrometry (ICP-MS) to determine Cr(VI) in treated drinking water, surface water, and ground water. Samples are collected and preserved at a pH > 9 condition, and then injected directly into an anion exchange column. Cr(VI) is separated from other possible Cr species and other metals by the anion exchange functioning group inside the column. The column eluent is introduced directly into the sample introduction interface and the ionization source of the ICP-MS. Chromium chromatographic peak is identified and quantified by the mass spectrometry with external calibration.

Labware Cleaning Procedure

It is critical to pre-clean and dry labware in a clean flow bench in order to minimize contamination.

- Place tubes and caps into 10% Nitric Acid (made from reagent grade) acid bath for at least 24 hours.
- Transfer tubes and caps into a DI Water bath to soak for at least 24 hours.
- Remove tubes and caps, rinse with DI Water at least three times.
- Remove as much water as possible and place inside a Class 10 Vertical Laminar Flow Metal Free Hood and let dry.

Sample Requirements

- Sample must be preserved to achieve pH > 9 with Ultra Pure Concentrated Ammonium Hydroxide.
- Sample is collected in a 15mL amber high density polyethylene (HDPE) bottle with a plastic cap.
- Samples are stored at < 8 °C for up to 30 days, provided that the sample containers are sealed properly and stored in an acid fume free environment. However, it is recommended that samples be analyzed as soon as possible upon receipt.

Sample Preparation

Check sample pH using a pH testing strip by transferring a small volume of sample to prevent cross contamination. If the pH is > 9, sample is ready for IC-ICP-MS analysis | Note: r2 > 0.995 for the calibration curve

- Label the Thomson 0.45µm PTFE Filter Vials (P/N 34440).
- Pipette 0.5mL of the sample into the filter vial shell.
- Partially insert the filter vial plunger into the filter vial shell.
- Place filter vials in the Thomson Toggle Press (P/N 35005) and press the lever to filter the samples (can press up to 5 vials each time).
- Load the filter vials into the Varian autosampler.
- Include Calibration Standards (0.05µg/L, 0.1µg/L, 0.5µg/L, 1.0µg/L)

and QC Standards (DI Water Blank, Tap Water Blank, Tap Water Spiked) for every 20 samples analyzed.

Equipment

- LC-MS:
- Varian ProStar 210 HPLC
- Varian 820MS ICP-MS
- Pump Rate (rpm): 20
- Stablization delay(s): 0
- Skimmer Gas Source: H2
- Skimmer Flow: 30

Column

Hamilton PRP-X100 Anion Exchange Column & Guard Column

Mobile Phase

- A: 100mM/L Ammonium Nitrate, $pH \ge 9$. pH adjust with 16N Nitric Acid
- B: DI Water, $pH \ge 9$, pH adjust with Ultra Pure Ammonium Hydroxide

Time	Flow (mL/min)	% A	%B
Pre-run	1.0	80	20
9.0	1.0	80	20



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