



CATALYST FOR SUCCESS

► HIGHLY SENSITIVE ANALYSIS OF CHROMIUM (VI) IN DRINKING WATER BY ION CHROMATOGRAPHY (IC) WITH POST-COLUMN DERIVATIZATION AND UV/VIS DETECTION

Chromium is widely used in the manufacturing of metal alloys, surface coatings, pigments and other products. It is also present naturally in soil. Chromium (VI) is the most toxic form of Chromium and is a possible human carcinogen. Due to its high solubility, Hexavalent Chromium can easily contaminate drinking and ground water sources and its levels must be monitored.

In July 2011, the Office of Environmental Health Hazard Assessment (OEHHA) established a Public Health Goal (PHG) for Chromium (VI) at a concentration of 0.02 ug/L. Based on the PHG, the California Department of Public Health is developing a primary drinking water standard (Maximum Contaminant Level, MCL) that is specific for Chromium (VI).

EPA Method 218.7 uses post-column derivatization with diphenylcarbazide under highly acidic conditions and UV/VIS detection to analyze Chromium (VI) in drinking water. Injection volumes of 1000 uL and 1250 uL were used in the method but Pickering Laboratories was able to meet method requirements with as low as 100 uL injections. Modern IC systems and columns can successfully separate Chromium (VI) from other ions with short run times. Pickering Laboratories' post-column system is uniquely suitable for delivering aggressive derivatizing reagents with unmatched flow precision and low noise level in order to consistently provide highly sensitive detection of Hexavalent Chromium.

METHOD

Sample Preparation

Clean all glassware and containers with 1:1 H₂O/HNO₃. Rinse well with deionized water.

Adjust all samples and standards to pH 9.0-9.5 using Sample Adjustment Buffer (250 mM Ammonium Sulfate, 500 mM Ammonium Hydroxide in water). For most samples, no more than 1 mL of Adjustment Buffer is used per 100 mL of water sample. Correct final results for dilution. Store all samples and standards in the refrigerator and analyze within 24 hours.

An injection volume of 100 uL is sufficient to detect levels as low as 0.01 ppb. Injection volume can be increased up to 1000 uL if needed.

Analytical Conditions

IC System: ICS 900 or equivalent IC system (Thermo Scientific)

Analytical Column: IonPac AS22, 4x250 mm (Thermo Scientific)

Guard Column: IonPac AG22, 4x50 mm (Thermo Scientific)

Flow Rate: 1 mL/min

Column Temperature: 30 °C

Mobile Phase: 250 mM Ammonium Sulfate, 50 mM Ammonium Hydroxide in water

Injection Volume: 100 uL

Post-Column Conditions

Post-column System: Pinnacle PCX for Chromium (VI) Analysis

Reactor Volume: 1.0 mL

Reactor Temperature: 30 °C

Reagent: 2 mM Diphenylcarbazide, 1 N Sulfuric Acid, 10% Methanol

Reagent Flow Rate: 0.33 mL/min

Detection: UV/VIS, 530 nm

TABLE 1. RECOVERIES DATA FOR TAP WATER SPIKED WITH CHROMIUM (VI).

| SPIKE CONCENTRATION | RECOVERIES | RSD, % N=3 |
|---------------------|------------|---------------|
| 0.1 ppb | 104% | 10.6 |
| 0.5 ppb | 96% | 4.3 |

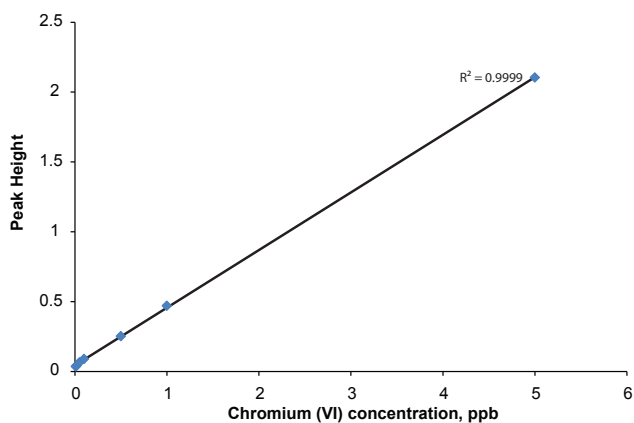


Fig. 1 Calibration curve for Chromium (VI) 0.01 – 5 ppb range

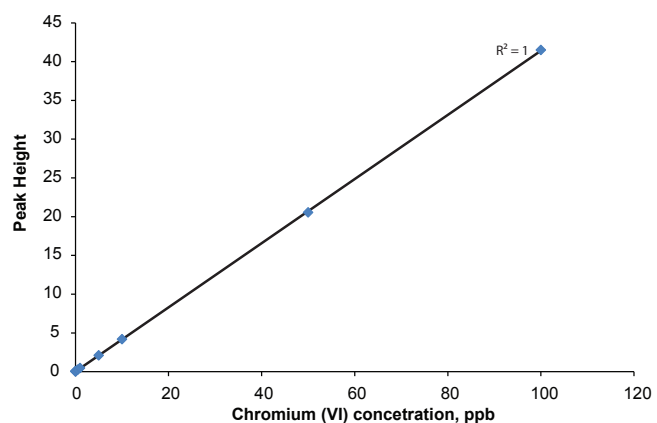


Fig. 2 Calibration curve for Chromium (VI) 0.01 – 100 ppb range

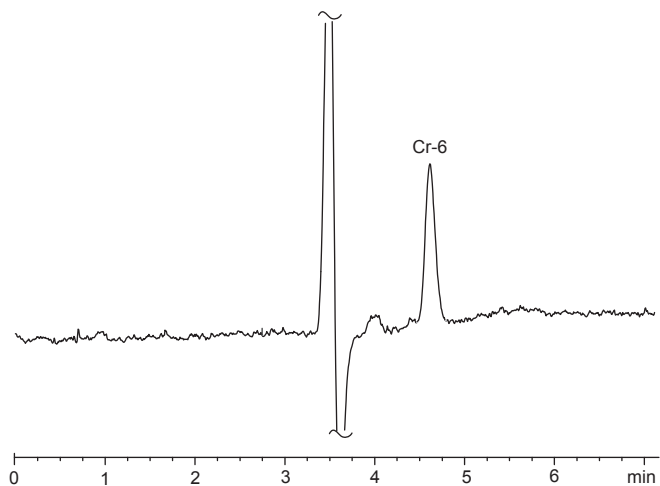


Fig. 3 Chromatogram of tap water spiked with 0.5 ppb of Chromium (VI)

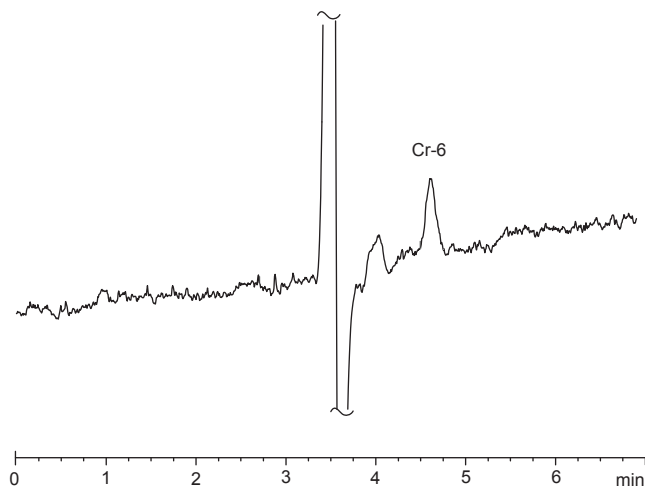


Fig. 4 Chromatogram of 0.02 ppb standard solution of Chromium (VI)

