

Low-Pressure Chromium Speciation in Drinking Water using the SC-DX chromFAST System with ICPMS Detection

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Introduction

Chromium exists in the environment in several forms which differ in their effects upon organisms. Chromium enters the air, water and soil as Cr(III) and Cr(VI) through natural processes and human activities. At certain levels, Cr(III) is an essential nutrient for humans. On the other hand, Cr(VI) is detrimental to human health and is considered carcinogenic.

For this reason, there is an interest in knowing the level of Cr(VI) in drinking water. Elemental Scientific's chromFAST chromium speciation kit, coupled with the SC-DX FAST automated sampling system, is an economical, robust, and easy-to-use system that uses a peristaltic pump and a low-pressure anion exchange column to separate, detect, and quantify Cr(III) and Cr(VI) species in water samples.



ICPMS hardware for fully-automated Cr(III) and Cr(VI) speciation

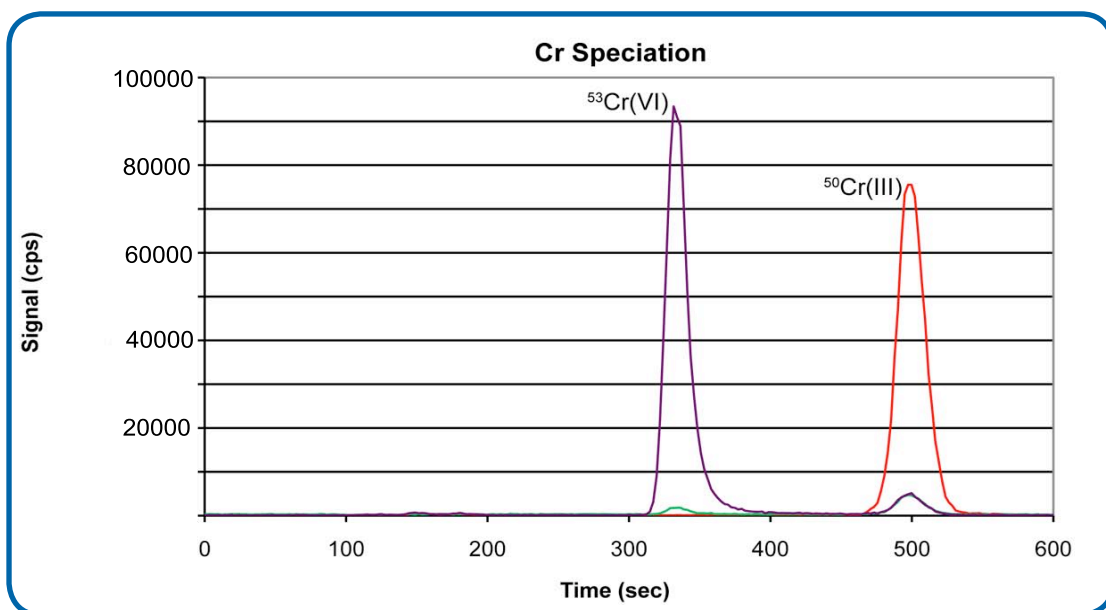


Table 1. Detection Limits	
Species	Detection Limit (ppt)
Cr(III)	3
Cr(VI)	4

Figure 1. Low-pressure separation and detection of isotopically-enriched Cr standards at 0.5 ppb concentration with SC-DX FAST and the ELEMENT2 ICPMS.

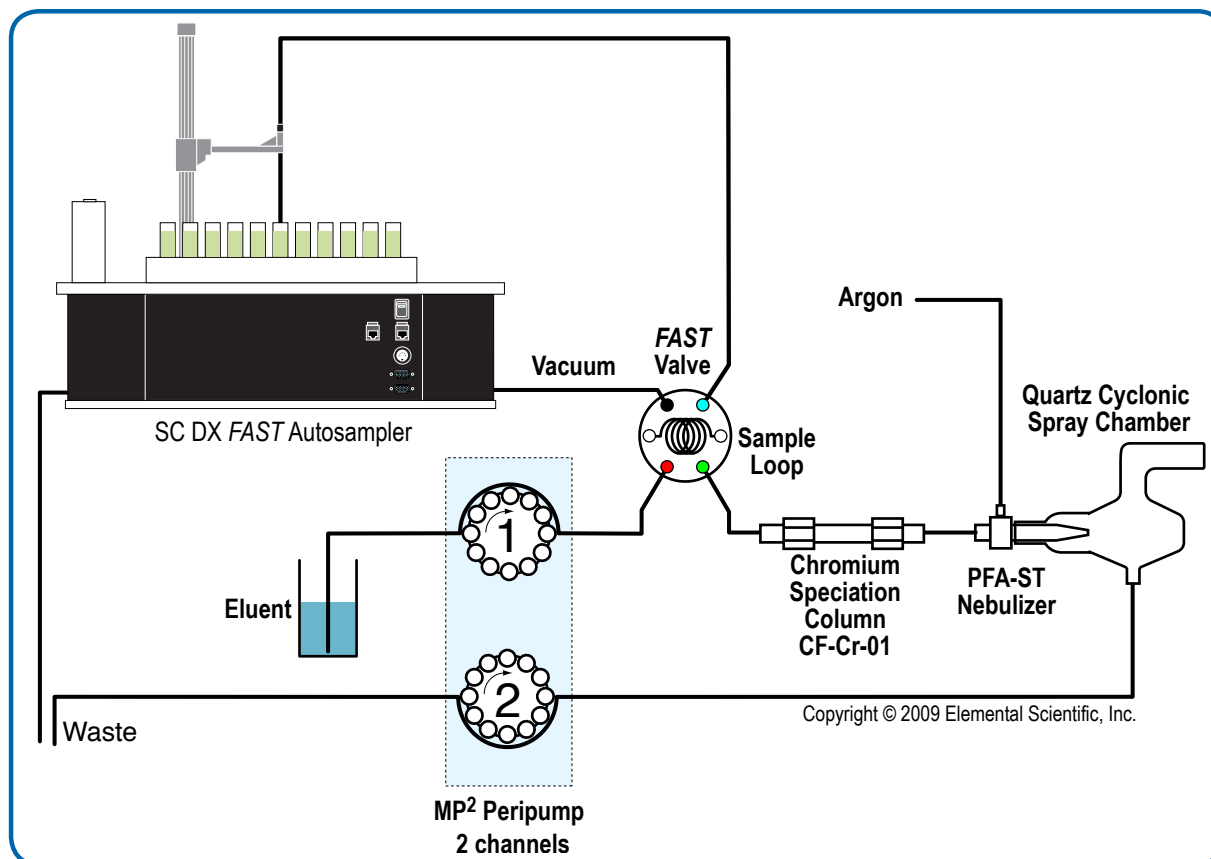


Figure 2. Diagram of the Cr speciation system. The speciation column is added to an existing SC-DX FAST system for separation of Cr species by isocratic elution anion exchange chromatography.

Instrumentation, Sample Introduction, and ICPMS Parameters

- ESI chromFAST speciation kit (CF-KIT-Cr36) with the Cr anion exchange column (CF-Cr-01)
- ESI MP² micro peripump (MP2-4-52)
- periSPEC software (ES-2999-4001)
- Thermo Scientific ELEMENT2 ICPMS

Procedure

Samples and standards were automatically loaded into the FAST valve sample loop and injected onto the Cr column. The Cr species were separated on the column and nebulized using a low dead volume PFA-LC nebulizer. The peaks for Cr(III) and Cr(VI) were measured using the time-resolved software built into the ELEMENT2 and were exported to a spreadsheet for data analysis.

Data Analysis

The collected data were analyzed with the ESI periSPEC Peak Area Finder software. This simple and relatively-inexpensive software utilizes an Excel-based spreadsheet to integrate the chromatographic peak areas. The resulting data can then be used for calibration and determination of Cr species.

Table 2. Cr Isotopes and Interferences

Cr Isotope	Natural Abundance	Spectral Interferences
50	4.3 %	$^{36}\text{Ar}^{14}\text{N}^+$
52	83.8 %	$^{40}\text{Ar}^{12}\text{C}^+$
53	9.5 %	$^{37}\text{Cl}^{16}\text{O}^+$
54	2.4 %	$^{40}\text{Ar}^{14}\text{N}^+$

Separation of Mass Spectral Interference

If a high level of Cl⁻ is present in the sample then $^{37}\text{Cl}^{16}\text{O}^+$, a mass spectral interference on $^{53}\text{Cr}^+$, can form (Table 2). The chromFAST Cr speciation kit separates Cl⁻ from Cr(III) and Cr(VI), resolving this interference (Fig. 3).

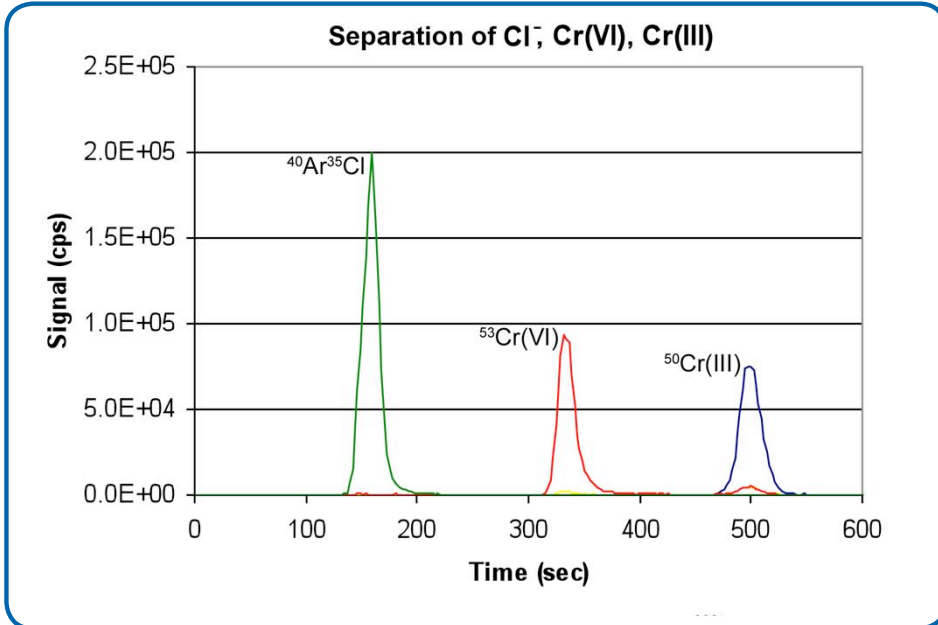


Figure 3. Low-pressure separation of $^{40}\text{Ar}^{35}\text{Cl}^+$, Cr(VI), and Cr(III). $^{40}\text{Ar}^{35}\text{Cl}^+$ was monitored as a proxy for the ClO⁺ interference.

Calibration and Separation Studies

Isotopically-enriched species-specific standards of Cr(III) and Cr(VI) were used to both demonstrate the separation and species interconversion. Species separation was obtained by using an isocratic elution. With this single-phase elution, elements are eluted in the following order: $^{40}\text{Ar}^{35}\text{Cl}^+$, Cr(VI), and Cr(III). $^{40}\text{Ar}^{35}\text{Cl}^+$ monitored as a proxy for the ClO⁺ interference. (Fig. 3).

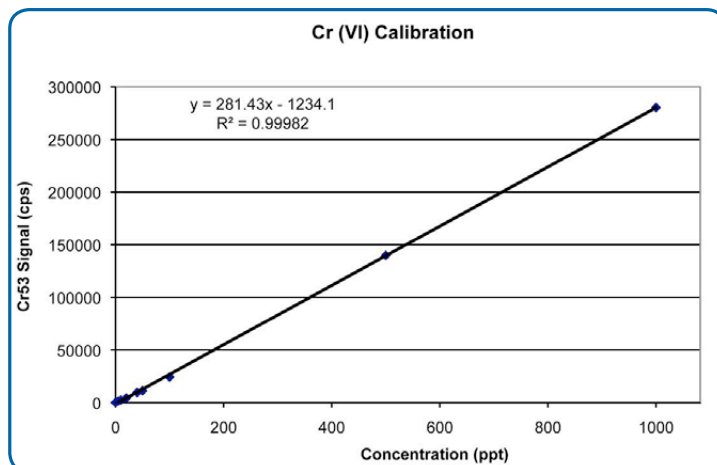
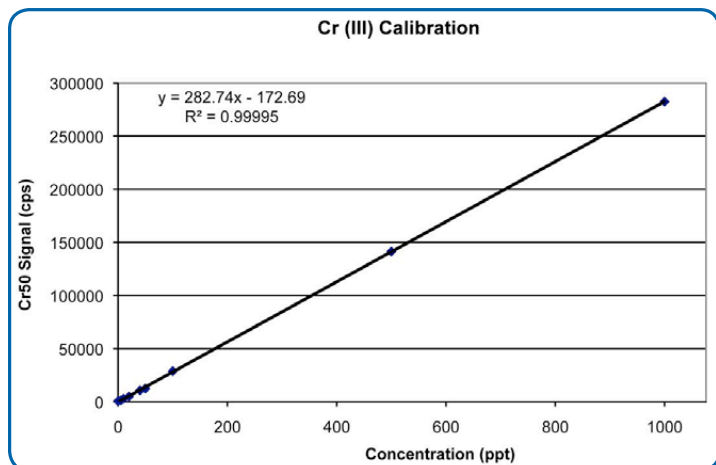


Figure 4. Calibration curve for inorganic Cr(III) and Cr(VI) species.

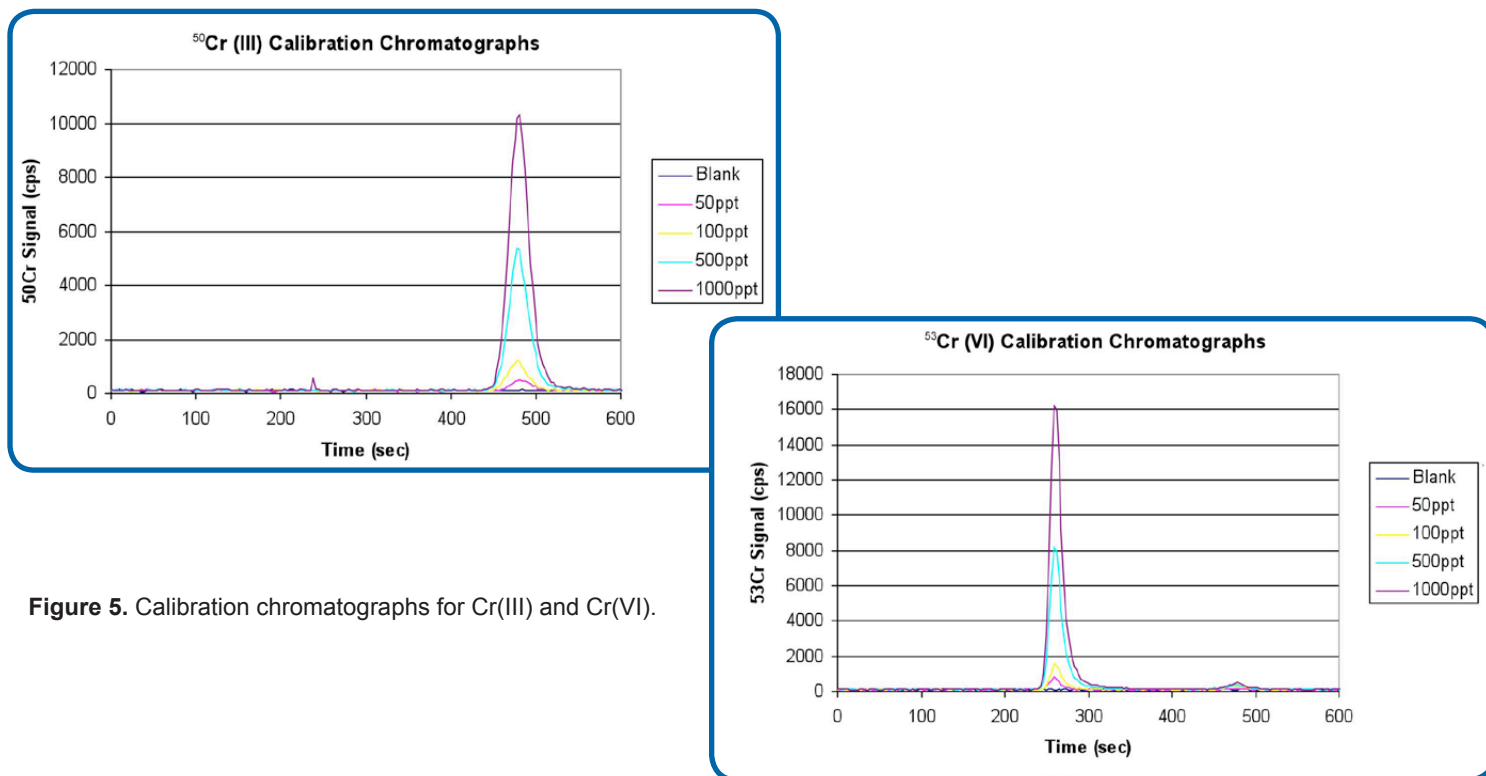


Figure 5. Calibration chromatographs for Cr(III) and Cr(VI).

Spike Recovery Studies

Spike recoveries were measured by spiking 0.5 ppb of each Cr(III) and Cr(VI) into various water samples including tap water in Omaha, NE (Table 3). Spike recoveries ranged from 96% to 101%.

Table 3. Detection of Cr(III) and Cr(VI) and spike recoveries in three types of water	Cr(III)		Cr(VI)	
	Concentration (ppt)	% Recovery	Concentration (ppt)	% Recovery
Distilled Water	< 3		5	
Distilled Water (Spiked)	490	98	508	100
Bottled Water	6		313	
Bottled Water (Spiked)	514	101	817	101
Omaha Tap Water	4		1068	
Omaha Tap Water (Spiked)	498	99	1548	96

Conclusion

Obtaining accurate measures of trace amounts of Cr species can be costly and time consuming. chromFAST low-pressure anion exchange chromium speciation kit (CF-KIT-Cr36) and the SC-DX FAST system for ICPMS is an economical alternative to HPLC-ICPMS systems. The fully-automated and easy-to-use system can switch between multi-element ICPMS sample analysis to elemental speciation detection modes in minutes. The unique periSPEC peak finder software provides a straightforward method for data analysis. The robust anion exchange column can be used for multiple samples and efficiently separates Cr species providing a cost-effective solution for speciation needs.