

## Application News

Nexera™ lite inert

### Analysis of Hexavalent Chromium in Drinking Water According to EPA Method 218.7

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#### User Benefits

- ◆ Analysis of hexavalent chromium by EPA Method 218.7 can be successfully achieved in less than 6 min with Shimadzu Nexera lite inert.
- ◆ Performance achieved is suitable for the selective and accurate quantification of Hexavalent Chromium in the low µg/L range.
- ◆ Hexavalent chromium can be chromatographically separated from potential interferences and selectively detected using post-column derivatization.

#### Introduction

Chromium is a widely used metal in steel making, paints, etc. The most common forms of chromium are trivalent chromium, hexavalent chromium and metallic chromium. Total chromium, which includes hexavalent and trivalent forms is regulated by the United States Environmental Protection Agency (EPA) in wastewater and drinking water. However, in July 2011, the Office of Environmental Health Hazard Assessment (OEHA) at the California issued a new Public Health Goal (PHG) value of 0.02 µg/L for hexavalent chromium in drinking water. Therefore, the new EPA Method 218.7<sup>1)</sup> is necessary.

In EPA Method 218.7, the hexavalent chromium is separated by an anion exchange column. The colored complex formed between hexavalent chromium and 1,5-diphenylcarbazide in the post-column derivatization is then detected at 530nm. Compared to EPA Method 218.6, the sample injection volume is larger, and a lower detection limit is obtained.

This application news introduces an example of hexavalent chromium analysis of drinking water in accordance with EPA Method 218.7 by Shimadzu Nexera lite inert system.

#### Analytical Conditions

The flow path diagram according to EPA Method 218.7 is shown in Fig.1. The analytical conditions are shown in Table 1. The standard sample was prepared by diluting sodium chromate tetrahydrate with ultra pure water. In this experiment, the elution time for hexavalent chromium was about 6 min.

To minimize the reduction of hexavalent chromium to trivalent chromium which can occur in the presence of reducing species in an acidic medium, the sample pH was adjusted to greater than 8 with an aqueous solution of ammonium sulphate-ammonia prior to final dilution.

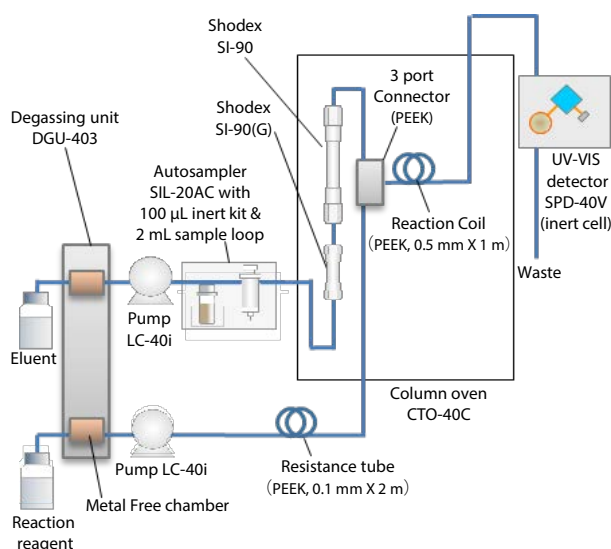


Fig. 1 Flow path diagram of hexavalent chromium analysis system

Table 1 Analytical conditions

System	: Nexera lite inert
Column	: Shodex SI-90 (250 mm × 4.0 mm I.D., 9 µm)
Guard Column	: Shodex SI-90(G) (10 mm × 4.6 mm I.D., 9 µm)
Mobile phase	: 50 mmol/L Ammonium sulfate 20 mmol/L Ammonium hydroxide
Mobile phase Flow rate	: 0.8 mL/min
Post Column Reagents	: 2 mmol/L 1,5-diphenylcarbazide 10% Methanol 1 N Sulfuric acid
Post Column Reagents Flow rate	: 0.3 mL/min
Column temp.	: 45 °C
Injection volume	: 1000 µL
Vial	: Shimadzu Vial, LC, 4 mL, Polypropylene <sup>1)</sup>
Detection	: UV-VIS (530 nm, inert cell)
Reaction Coil	: 250 µL (1 mm × 0.5 mm I.D., (PEEK))

\*1 P/N : 228-31537-91

#### Calibration and Precision and Accuracy

In this application, the calibration curve was created with 6 points between 0.02-1 µg/L in accordance with EPA requirements. The coefficient of correlation ( $r^2$ ) for the calibration curve was 0.99997 (Fig. 2). A blank sample was analyzed immediately after the highest standard sample which confirmed that no carry-over occurred (Fig. 3).

Seven replicated analyses were conducted at three points on the calibration curve: the low-level, the mid-level, and the high-level concentration to confirm precision and accuracy. Table 2 shows these results. Both the relative standard deviation (%RSD) and the mean recoveries of true values met EPA requirements.

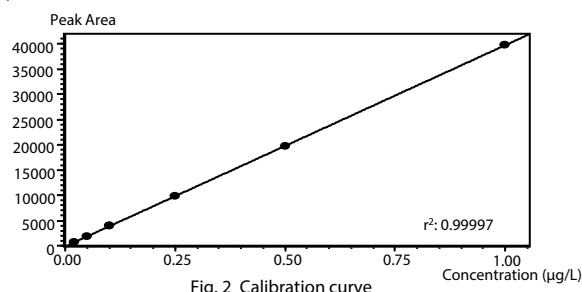


Fig. 2 Calibration curve

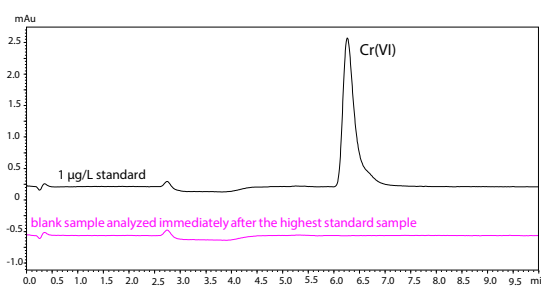


Fig. 3 Chromatogram of 1 µg/L standard and blank sample analyzed immediately after the highest standard sample

Table 2 Recoveries and precision of area

Concentration of standards	Area precision (%RSD)	Mean recoveries of true value(%)
0.02 µg/L	6.38	102.5
0.1 µg/L	2.63	102.2
1 µg/L	0.47	101.4

EPA 218.7 requirements : Percent relative standard deviation must be  $\leq 15\%$ , mean recovery within  $\pm 15\%$  of the true value

## ■ Method Detection Limit and Quantitation Limit

The method detection limit (MDL) was calculated as  $(t) \times (S)$  after seven replicated analyses of 0.02 µg/L hexavalent chromium standard. Fig. 4 shows the analysis results. The calculated MDL and calculation formula is shown in Table 3.

The MDL value of 0.003 µg/L enables a minimum quantitation limit (LOQ) of 0.009 µg/L for hexavalent chromium, which is adequate for routine analysis at the California PHG of 0.02 µg/L and current proposed detection limit for purposes of reporting of 0.1 µg/L<sup>2)</sup>.

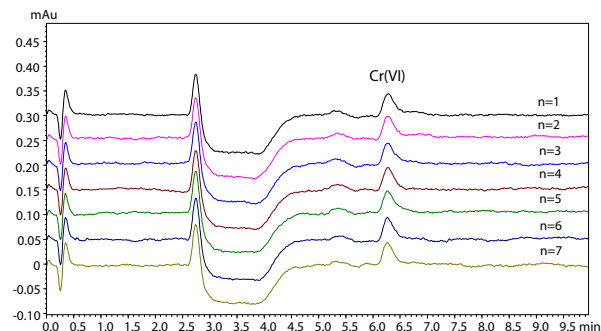


Fig. 4 Chromatograms of seven replicated analyses of 0.02 µg/L hexavalent chromium standard

Table 3 MDL calculation

**MDL : 0.003 µg/L**

$$MDL = (t) \times (s)$$

t = Student's t value for n-1 degrees of freedom at the 99% confidence level;

t = 3.143 for six degrees of freedom

s = standard deviation of the replicate analyses

## ■ Analysis Example of Mineral Water and Tap Water

Following the procedure described in EPA Method 218.7, three kinds of commercially available mineral water and tap water were analyzed seven replicates. For the fortified sample matrix test, low and high concentrations of hexavalent chromium standard were added to samples, and seven replicated analyses were measured.

Table 4 shows the analysis results, and Table 5 shows the results of the fortified sample matrix test. Fig. 5 shows example chromatograms of the analysis results. The method for calculating the recovery is described in EPA Method 218.7. All the recovery results passed the requirements.

Table 4 Analytical results of samples

Sample	Mineral water A	Mineral water C	Mineral water S	Tap water
Mean measured value (µg/L)	<MDL	0.80	0.0095	0.021
Relative standard deviation (%RSD)		0.37	3.74	6.14

Table 5 Analytical results of fortified sample matrix test

Sample	Fortification (µg/L)	Mean measured value (µg/L)	Mean recovery *1 (%)	Relative standard deviation (%RSD)
Mineral water A	0.05	0.051	101.9	4.79
	0.2	0.20	101.5	0.95
Mineral water C	0.2	0.99	97.2	0.46
Mineral water S	0.05	0.058	97.5	3.89
	0.2	0.21	97.9	4.68
Tap water	0.05	0.071	99.4	3.55
	0.2	0.22	101.7	0.85

EPA 218.7 requirement: Percent relative standard deviation must be  $\leq 15\%$ , mean recovery within  $\pm 15\%$  of the true value

\*1 Mean recovery (%) = (mean measured value of fortified sample - mean measured value of sample) / fortification  $\times 100$

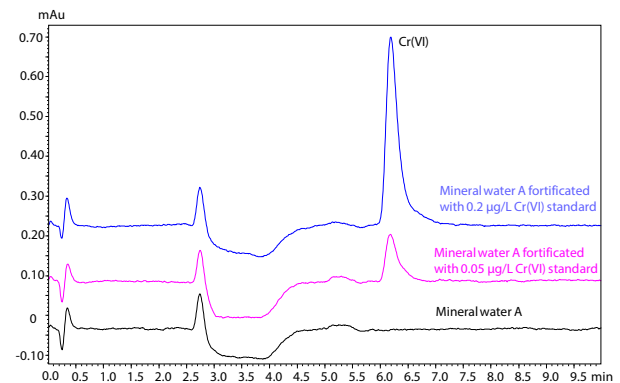


Fig. 5 Chromatograms of mineral water A and mineral water A fortified with 0.05 µg/L and 0.2 µg/L hexavalent chromium standard

## ■ Conclusion

The analysis example of hexavalent chromium in drinking water according to EPA Method 218.7 using Shimadzu Nexera lite inert system is introduced in this application. Low concentrations of hexavalent chromium in water can be chromatographically separated from potential interferences and selectively detected using post-column derivatization. The LOQ of 0.009 µg/L for hexavalent chromium, which is adequate for routine analysis at the California PHG and current proposed detection limit for purposes of reporting of 0.1 µg/L.

### <References>

- 1) EPA Method 218.7: Determination of Hexavalent Chromium in Drinking Water by Ion Chromatography with Post-Column Derivatization and UV-Visible Spectroscopic Detection, Version 1.0
- 2) Hexavalent Chromium  
[https://www.waterboards.ca.gov/drinking\\_water/certlic/drinkingwater/Chromium6.html](https://www.waterboards.ca.gov/drinking_water/certlic/drinkingwater/Chromium6.html) (Accessed in Feb, 2024)

### <Related Applications>

1. Analysis Method of Dissolved Hexavalent Chromium According to EPA 218.6, [Application News 01-00380A-EN](#)

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