thermo scientific

Determination of perchlorate in drinking water using ion chromatography

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INTRODUCTION

Perchlorate is widely used as the oxidizing component in solid propellants for rockets, munitions, and fireworks.[1] However, perchlorate can contaminate soil, groundwater, and drinking water, leading to adverse health effects. Perchlorate in high doses can disrupt thyroid function and hormone production, and therefore negatively affect the growth and development of fetuses and children and healthy metabolism in adults.

The U.S. Environmental Protection Agency (EPA) placed perchlorate on the first Contaminant Candidate List (CCL1) in 1998 and determined that perchlorate meets the Safe Drinking Water Act's criteria for regulation as a contaminant in February 2011. However, perchlorate is still not federally regulated.

U.S. EPA Method 314.0 uses ion chromatography with suppressed conductivity detection.[2] The EPA method specifies the use of a Dionex IonPac AS16 4 mm column with an eluent of 50 mM NaOH at a flow rate 1.5 mL/min. This poster updates the approach with a high-resolution Dionex IonPac AS16-4µm 2 mm column. In comparison to the conventional Dionex IonPac AS16 column, the Dionex IonPac AS16-4µm column exhibits higher peak efficiency while maintaining chromatographic selectivity. Using the Dionex IonPac AS16-4µm column increases the sample throughput by 20% and saves three minutes per injection, thereby improving productivity for perchlorate determinations in drinking water.

MATERIALS AND METHODS

Equipment

Thermo Scientific™ Dionex™ ICS-5000+ HPIC™*:
Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2.9
*This method can be run on any Thermo Scientific Dionex HPIC instrument

Chromatographic conditions

Columns:	Dionex IonPac AG16-4µm Guard Column, 2 × 50 mm (P/N 302756) Dionex IonPac AS16-4µm Analytical Column, 2 × 250 mm (P/N 302755)
Eluent:	65 mM KOH
Eluent source:	Dionex EGC 500 KOH cartridge with Dionex CR-ATC 500
Flow rate:	0.38 mL/min
Injection volume:	250 µL
Column temperature:	30 °C
Detection:	Suppressed conductivity
Suppressor:	Dionex ADRS 600 (2 mm) Suppressor, external water mode (flow rate = 0.38 mL/min), 62 mA current
Detection/suppressor compartment:	20 °C
Cell temperature:	35 °C
Background conductance:	<0.5 µS/cm
System backpressure:	~4100 psi (28.3 MPa)
Noise:	<0.5 nS/cm
Run time:	12 min

Samples:

Three residential tap waters were collected from three cities in the San Francisco Bay Area in California. (DW #1-3)

Standards:

Calibration standards were prepared at 1, 2, 5, 10, 25, and 50 µg/L.

Table 2. Standard mixture of 13 anions

Analyte	13-anion stock standard mixture concentration (mg/L)	1000 ppm stock volume (µL)	13-anion standard mixture concentration (µg/L)
Fluoride	2	200	20
Chloride	3	300	30
Sulfate	5	500	50
Nitrite	5	500	50
Carbonate	5	500	50
Nitrate	5	500	50
Bromide	5	500	50
Phosphate	5	500	50
Thiosulfate	10	1000	100
Iodide	20	2000	200
Thiocyanate	2	200	20
Perchlorate	5	500	50
4-chlorobenzene sulfonate	5	500	50

Column:	Dionex IonPac AG16/AS16-4µm column set, 2 mm
Eluent:	65 mM KOH
Eluent source:	Dionex EGC 500 KOH cartridge with Dionex CR-ATC 500
Inj. volume:	250 µL
Column temperature:	30 °C
Flow rate:	0.38 mL/min
Detection:	Dionex ADRS 600 (2 mm) Suppressor, external water mode

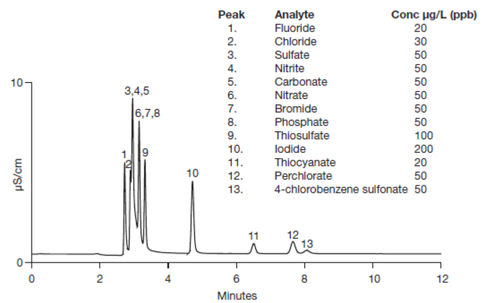

Figure 1: Separation of 13 anions showing resolution between Perchlorate and 4-Chlorobenzene sulfonate

Figure 1 shows a separation of perchlorate and common anions within 12 min using a Dionex IonPac AS16-4µm column. As this figure shows, perchlorate is well resolved from common inorganic anions such as chloride, sulfate, and carbonate. 4-chlorobenzene sulfonate can be found in leachates from some hazardous waste sites.

RESULTS

As Figure 1 shows, 4-chlorobenzene is resolved from perchlorate. Compared to the Dionex IonPac AS16 column, the Dionex IonPac AS16-4µm column demonstrates better resolution between perchlorate and the inorganic anion peaks under the same conditions. In addition, 4-chlorobenzene is not resolved from the perchlorate peak on the Dionex IonPac AS16 column. Thus, a shorter method (12 min), which still resulted in excellent separation of perchlorate was used in this study compared to the 15 min method. In this shorter method, the eluent was 65 mM at a flow rate 0.38 mL/min using a 2 mm diameter Dionex IonPac AS16-4µm column.

Chromatographic performance of perchlorate can deteriorate at high anionic concentrations, primarily due to the presence of high concentrations of chloride, sulfate, and carbonate. Figure 2 shows chromatograms of 25 µg/L perchlorate in DI water, MA 200, and MA 1000. "MA" indicates mixed common anion solution with each anion (chloride, sulfate and carbonate)included in the sample matrix at the parenthetical mg/L concentration for each anion.[2]

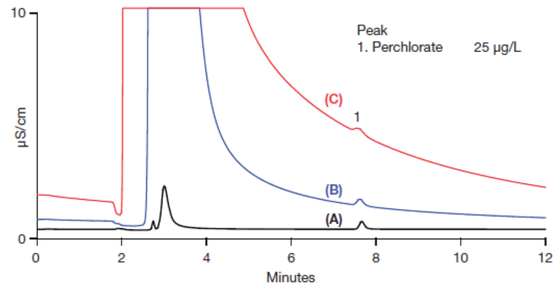

Figure 2. Determination of 25 µg/L perchlorate in (A) DI water, (B), MA 200, and (C) MA 1000

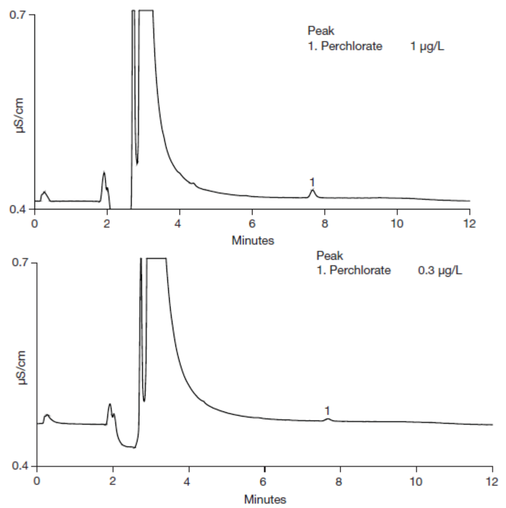

Figure 3. Determination of Perchlorate at 1 and 0.3 µg/L perchlorate in DI water

Sample analysis

Three residential tap waters were collected from three cities in the San Francisco Bay Area, California. Perchlorate is detected in drinking water #1 at 0.84 µg/L. Perchlorate is not detected in drinking waters #2 and #3.

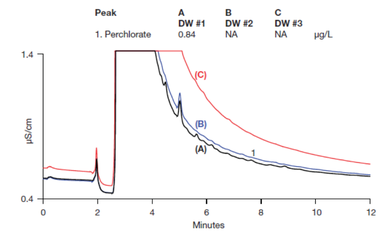

Figure 4. Determination of perchlorate in three drinking water samples

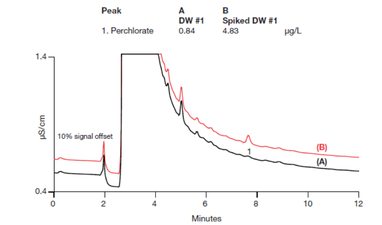

Figure 5. Overlay of chromatograms of (A) DW #1 and (B) spiked DW #1

CONCLUSIONS

This study demonstrates that perchlorate can be determined sensitively and accurately in municipal drinking water using a Dionex IonPac AS16-4µm column. The excellent resolution of the 4-µm-column allows for faster analysis (12 min) without compromising data quality. The Reagent-Free ion chromatography system provides excellent reproducibility, thereby yielding greater quantification accuracy and consistently reliable results.

REFERENCES

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4. Thermo Scientific Dionex IonPac AS16-4µm column manual. https://assets.thermofisher.com/TFS-Assets/CMD/manuals/Man-065723-IonPac-AS16-4um-Man065723-EN.pdf

TRADEMARKS/LICENSING

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