Shortening run times for perchlorate determination in drinking water using refinements in ion chromatography

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Abstract

Purpose: To demonstrate a method to determine perchlorate in drinking water samples using a Thermo Scientific™ Dionex™ IonPac™ AS16-4µm column with electrolytically generated eluent and a high-pressure ion chromatography (HPIC™) system.

Method: Anion exchange chromatography with electrolytic suppression and Reagent Free IC (RFIC) was used.

Results: The method shown allows the determination of perchlorate in under 12 minutes. In four drinking water samples, perchlorate was determined to be either <1 ug/L or below detection limits.

Introduction

Perchlorate (ClO_4^-), an environmental contaminant, is found in drinking, ground, and surface waters due to improper use and release of perchlorate salt-containing materials. Perchlorate is mainly released from perchlorate salts like ammonium perchlorate widely used in propellant for rocket, fireworks, and other aerospace materials. Perchlorate contamination in drinking water can cause developmental issues in children and metabolic problems in adults by affecting hormone production and inhibiting thyroid function. United States Environmental Protection Agency (EPA) Method 314.0 uses high performance anion exchange chromatography with suppressed conductivity detection to determine perchlorate in drinking water.[1] Thermo Scientific Application Update 148[2] and Application Note 73267[3] describe methods to determine perchlorate in drinking water according to EPA Method 314.0 and ISO 19340[4]. Application Note 73267 and the data shown here describes the use of the Thermo Scientific™ Dionex™ IonPac AS16-4µm (2 × 250 mm) anion-exchange column set, which uses smaller, 4 µm particles to significantly improve on the performance of the conventional Dionex IonPac AS16 column, increasing sample throughput by 20% and saving three minutes per injection. This is achieved using eluent generation and an upgraded suppressor, an ideal combination for determining perchlorate in drinking water.

Materials and methods

Figure 1 shows the primary components of an IC system with replacement of manual eluent preparation by Reagent-free IC (RFIC), which removes user to user variability and adds the capability of using gradients for method optimization

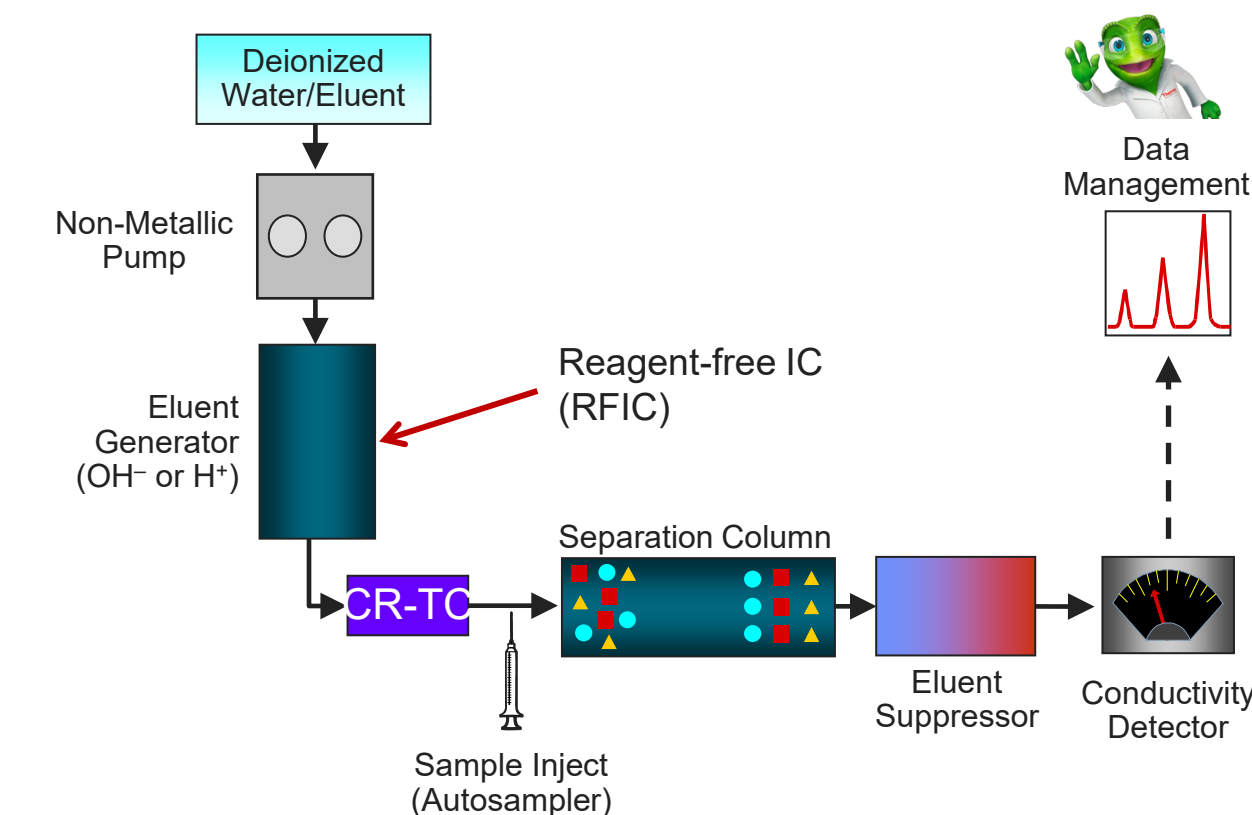**Figure 1. Schematic of a typical ion chromatography system using eluent generation.**
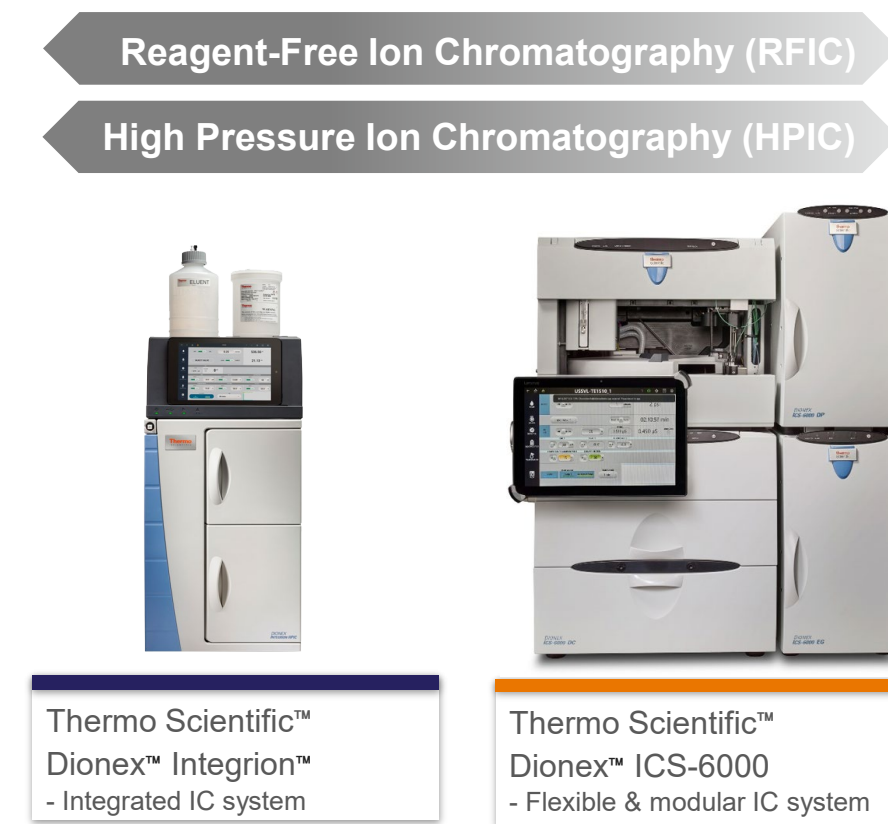

Results

Drinking water samples from different municipalities were analyzed for perchlorate. To quantify the samples, the response of perchlorate to concentration was determined using triplicate injections of 1, 2, 5, 10, 25, and 50 µg/L perchlorate. The response was shown to be linear without forcing through zero, with a coefficient of determination, r2 = 0.99965.

Figure 3 shows a zoomed in view of the chromatograms of 25 µg/L perchlorate in DI water, in 200 mg/L each chloride, sulfate, and carbonate (MA 200), and 1,000 mg/L each chloride, sulfate and carbonate (MA 1,000).

Figure 3. Determination of 25 µg/L perchlorate in (A) DI water, (B) MA 200, and (C) MA 1,000.

Four drinking water samples were tested for perchlorate and the results are shown in Table 2. Perchlorate was not detected in two samples and less than 1 µg/L perchlorate was detected in the other two samples.

	DW1	DW2	DW3	DW4
Perchlorate (µg/L)	0.94	n.a	0.83	n.a.

Table 2. Determination of perchlorate in four drinking water samples

To evaluate method accuracy, 4 µg/L perchlorate was added to each drinking water sample and recovery was calculated (Figure 4). The range of recovery for perchlorate spiked in drinking water was 86–100%.

Figure 2. Dionex IC systems with RFIC

Reagent-Free Ion Chromatography (RFIC)

High Pressure Ion Chromatography (HPIC)

Thermo Scientific™ Dionex™ Integrion™
- Integrated IC system

Thermo Scientific™ Dionex™ ICS-6000
- Flexible & modular IC system

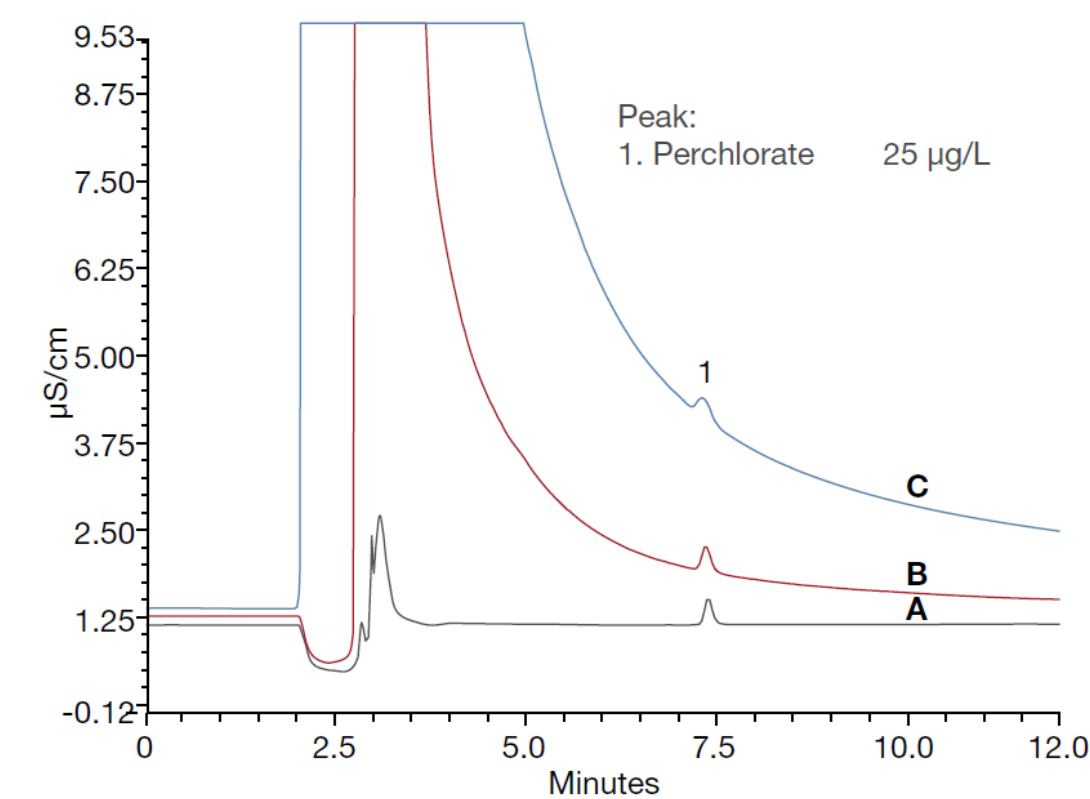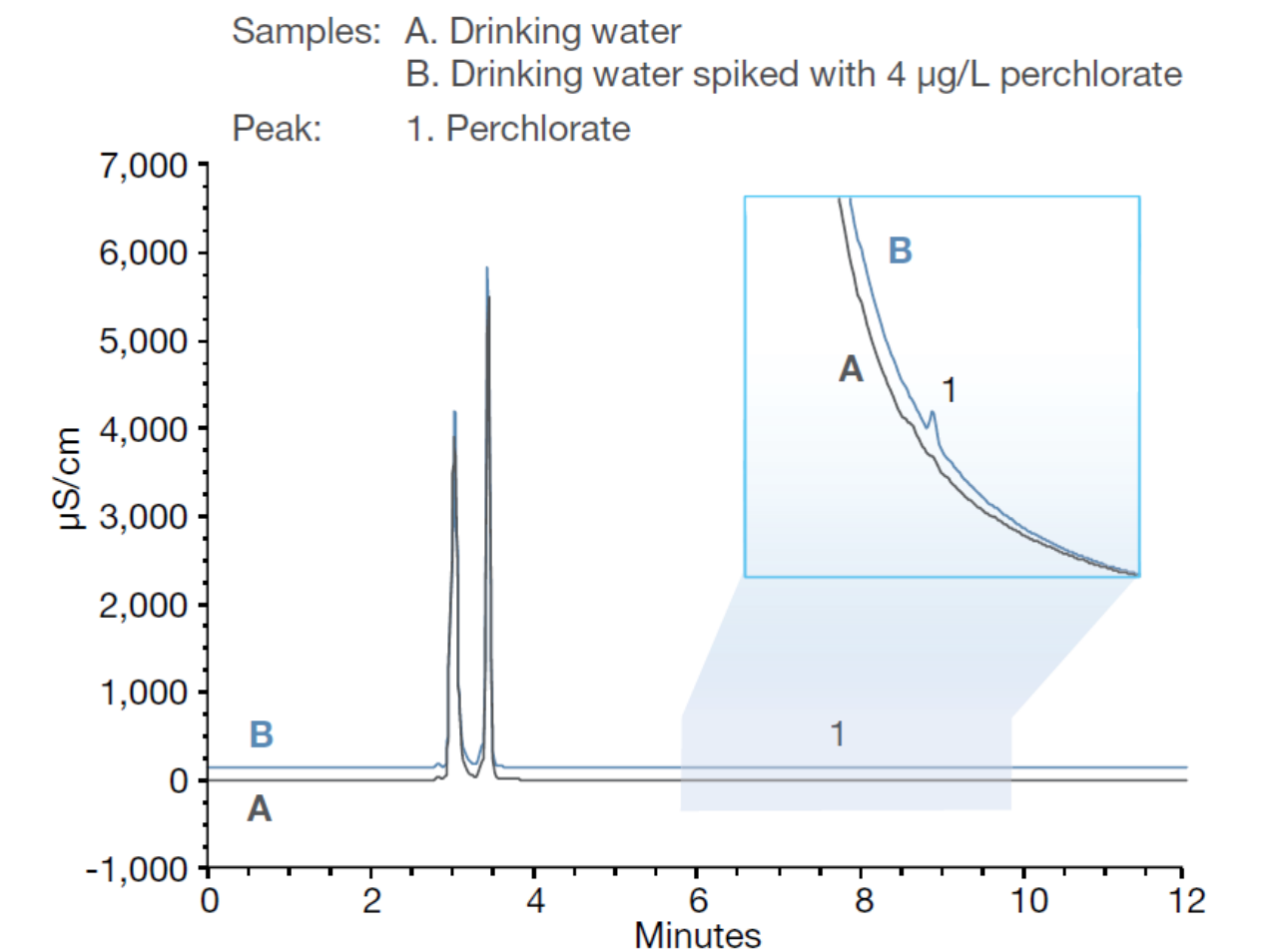**Table 1. Instrument method parameters.**

Parameter	Value
Instrument	Dionex IC system with column heater, pump degas module, and eluent generation
Autosampler	Thermo Scientific™ Dionex™ AS-DV autosampler
Columns	Dionex IonPac AS16-4µm, 2 mm column set
Eluent	65 mM KOH via RFIC eluent generation
Flow rate	0.38 mL/min
Column temp.	30 ˚C
Injection volume	250 µL
Detection	Suppressed conductivity, Thermo Scientific™ Dionex™ ADRS 600 (4 mm) suppressor, 62 mA , constant current, recycle mode
System backpressure	~3,800 psi (100 psi = 689.5 kPa)
Background conductance	<1.4 µS/cm
Noise	<1.1 nS/cm
Run time	12 min
Software	Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software version 7.3.2

Figure 4. Determination of perchlorate in drinking water

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Samples: A. Drinking water
B. Drinking water spiked with 4 µg/L perchlorate

Peak: 1. Perchlorate

Conclusions

- The method showed that a 4 µm anion exchange column can be used to determine perchlorate in drinking water in under 12 minutes
- The response data showed excellent correlation with concentration range of 1 – 50 µg/L
- Spike recovery studies demonstrated that the method is accurate.

References

1. U.S. EPA Method 314.0; U.S. Environmental Protection Agency; Cincinnati, OH, 1997.
2. Thermo Fisher Scientific Application Update 148: Determination of perchlorate in drinking water using a reagent-free ion chromatography system.
3. Thermo Fisher Scientific Application Note 73267: Determination of perchlorate in drinking water using ion chromatography.
4. International Organization for Standardization. (2017). Water quality – Determination of dissolved perchlorate - Method using ion chromatography (IC) (ISO Standard No. 19340:2017).

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