Determination of Perchlorate in Drinking Water Using Reagent-Free™ Ion Chromatography

INTRODUCTION

Perchlorate (ClO_4^-) is an environmental contaminant and has been found in drinking, ground, and surface waters in several states in the U.S.[1] However, most contaminated sites appear to be geographically confined, particularly in the western U.S., and linked to identifiable sources, such as military installations and manufacturing sites.[2] Because perchlorate targets the thyroid gland at sufficiently high concentrations,[3] in 1998 the EPA's Office of Groundwater and Drinking Water placed this anion on its Contaminant Candidate List (CCL) for drinking water. Currently, the EPA has not established any enforceable health regulations for perchlorate in drinking water or related matrices, although some states have set individual action levels. For example, the California Department of Health Services (CDHS) has adopted an action level of 4 μg/L perchlorate.[4] If concentrations are detected above this level, then the CDHS recommends that utilities remove the drinking water source from service for proper treatment.

Dionex Application Note 134 describes the determination of perchlorate in environmental waters to 2 μg/L using a large-loop injection with an IonPac® AS16 column and suppressed conductivity detection with an ASRS® ULTRA operating in external water mode.[5] However, the U.S. EPA is currently investigating different analytical approaches that can improve the sensitivity and selectivity for perchlorate. The sensitivity for perchlorate can be improved by coupling a mass spectrometer (MS) to an ion chromatograph or by using a 2-mm IonPac AS16 column.[6,7]

Further improvements to the existing chromatographic conditions for determining perchlorate as outlined in U.S. EPA Method 314.0 can be accomplished by decreasing the baseline noise. In this application update, we describe a minor modification to EPA Method 314.0 that replaces the standard ASRS ULTRA with an improved ASRS ULTRA II suppressor. The ASRS ULTRA II can routinely produce peak-to-peak noise in the range of 1–2 nS when operated in external water mode, compared to 9–10 nS for the ASRS ULTRA. Nearly an order of magnitude decrease in baseline noise significantly enhances the detection of perchlorate to 1 μg/L or less. As an additional benefit, the ASRS ULTRA II does not require chemical regenerates, such as the sulfuric acid required for the AMMS III suppressor as described in AU 145.[7] This application update describes the determination of perchlorate using the procedure outlined in EPA Method 314.0.[8] This application used an integrated ion chromatography system with a 4-mm IonPac AS16 column, an EGC II KOH cartridge, a 1000-μL injection, and suppressed conductivity detection with an ASRS ULTRA II operated in external water mode. This application update evaluates and describes the linear range, initial demonstration of capability (EPA Method 314.0,[9] Section 9.2), matrix conductivity threshold (MCT) (EPA Method 314.0, Section 9.2.8), method detection limits (MDLs) (EPA Method 314.0 Section 9.2.6), and recovery of perchlorate in typical environmental matrices (EPA Method 314.0, Section 9.4).

EQUIPMENT

A Dionex ICS-2000 Reagent-Free Ion Chromatography (RFIC) System was used in this work. The ICS-2000 is an integrated ion chromatograph that includes:

Eluent Generator

Column Heater

Pump Degas

EluGen® EGC II KOH Cartridge (Dionex P/N 058900)

CR-ATC (Dionex P/N 060477)

AS50 Autosampler

Chromeleon® 6.5 Chromatography Workstation

Suppressor External Regen Installation Kit for External
    Water Mode (P/N 038018)

Conductivity Meter (Thermo Orion, Model 105)

This application update is also applicable to other
    RFIC systems.

REAGENTS AND STANDARDS

Deionized water, Type I reagent-grade, 18 MΩ-cm resistivity or better

Sodium Perchlorate ($NaClO_4$) (Aldrich 41,024-1)

Sodium Chloride (NaCl)
    (J. T. Baker; VWR P/N JT3625-1)

Sodium Sulfate (Na_2SO_4) (Aldrich 29,931-3)

Sodium Carbonate Monohydrate ($Na_2CO_3 • H_2O$)
    (Fisher S262-3)

CONDITIONS

Columns:	IonPac AS16 Analytical, 4 × 250 mm (P/N 055376)
	IonPac AG16 Guard, 4 × 50 mm (P/N 055377)
Eluent:	65 mM potassium hydroxide
Eluent Source:	ICS-2000 EG with CR-ATC
Flow Rate:	1.2 mL/min
Temperature:	30 °C
Injection:	1000 µL (with 10-µL cut volume from a 1100-µL sample loop)
Detection:	Suppressed conductivity, ASRS ULTRA II (4 mm), Autosuppression external water mode
	Power setting, 193 mA
System Backpressure:	~2500 psi
Background Conductance:	~1–2 µS
Noise:	~1–2 nS/min peak-to-peak
Run Time:	15 min

PREPARATION OF SOLUTIONS AND REAGENTS

Stock Perchlorate Standard Solution

Dissolve 0.1231 g of sodium perchlorate in 100 mL of deionized water for a 1000-mg/L standard solution. This stock standard is stable for at least one month when stored at 4 °C.

Working Standard Solutions

Prepare working standards at lower concentrations by diluting the appropriate volumes of the 1000-mg/L stock standard with deionized water. In this application, calibration standards were prepared at 1, 2, 10, 25, 50, and 100 µg/L perchlorate, with each standard injected in duplicate.

Mixed Common Anion Stock Solutions (EPA Method 314.0, Section 7.4.1)

Prepare 25 mg/mL (25,000 mg/L) each of chloride, sulfate, and carbonate. Dissolve 4.1213 g of sodium chloride in deionized water and dilute to 100 mL. Dissolve 3.6965 g of sodium sulfate in deionized water and dilute to 100 mL. Dissolve 5.1658 g of sodium carbonate monohydrate (4.416 g of sodium carbonate) in deionized water and dilute to 100 mL. These solutions were used to prepare 50, 100, 200, 400, 600, 800, and 1000 mg/L (ppm) of mixed anion (MA) standards of chloride, sulfate, and carbonate. These standards were used to determine the matrix conductivity threshold (MCT) (EPA Method 314.0, Section 9.2.8) and the MDLs (Section 9.2.6[8]).

Sample Preparation

Measure the conductance of the samples with a calibrated conductivity meter that has a minimum measuring range of 1–10,000 µS/cm. Verify the conductivity meter calibration by measuring the conductance of a commercially available reference solution or a prepared 745-mg/L KCl standard (EPA Method 314.0 Section 7.5) with a conductance of 1410 µS/cm at 25 °C. The conductivity meter must yield a value between 1380 and 1440 µS/cm to be considered calibrated. Compare the sample conductivity to the MCT

determined in your laboratory, as explained in EPA Method 314.0, Section 11. Filter all samples with a 0.2-μm syringe filter. Use a hydrophilic polypropylene or polyethersulfonate filter; do not use polyvinylidene fluoride (PVDF). Discard the first 300 μL of the filtrate and filter the remainder directly into a clean plastic autosampler vial. Qualify filters by analyzing a deionized water blank and a 10-μg/L perchlorate standard that has been passed through the filter. The blank should be free of peaks within the retention time window of perchlorate, and the recovery of the 10-μg/L standard should fall within 80–120%.

Samples that exceed the MCT can often be analyzed after an appropriate dilution followed by filtration with a 0.2-μm filter. EPA Method 314.0 Section 11.1.3 explains how to determine the sample's dilution factor based on the MCT. For diluted samples, the minimum reporting level (MRL) **must** be raised by a proportion equivalent to the dilution.

If sample dilution does not yield the desired results—or to avoid diluting samples—the concentration of the matrix ions can be reduced by treating the sample with Dionex OnGuard® cartridges. This procedure is explained in further detail in EPA Method 314.0, Section 11.1.4 and in Dionex Application Update 145.[7] In this application, no pretreatment or dilution was required for the samples analyzed.

SYSTEM PREPARATION AND SETUP

Install backpressure tubing in place of the column set to produce a total system pressure between 2000 and 2500 psi at a flow rate of 1 mL/min. Install an EGC II KOH cartridge (Dionex P/N 058900). Condition the cartridge as directed in the *EGC II Cartridge Quickstart Guide* (Document No. 031909) by setting the KOH concentration to 50 mM at 1 mL/min for 30 min. After completing the cartridge conditioning process, disconnect the backpressure tubing that was temporarily installed in place of the column set. Install a CR-ATC between the EGC II KOH cartridge and EGC degas. For more information on installing the CR-ATC, consult the *EGC II Cartridge Quickstart Guide.*

Install and configure the AS50 Autosampler. The precision and accuracy of the autosampler will vary depending on the injection mode. The most accurate and precise injections are made with a calibrated sample

loop, flushed with about five times the loop volume. The largest full-loop injection possible with the AS50 is 300 μL. To inject 1000 μL, use the partial-loop injection mode with an 1100 μL sample loop, and a programmed "Sample Loop Volume" of 1100 μL and a "Cut Volume" of 10 μL. This injection procedure should provide peak area precision of <1% RSD. Install a 1-mL sample syringe and set the syringe speed to 4 or 5 to make faster large-loop injections. Enter the correct "Sample Loop Size" and "Sample Syringe Volume" in the AS50 Plumbing Configuration Screen. Refer to the *Autoselect AS50 Autosampler Operator's Manual* (Document No. 31169) for details.

Install a 4 × 50 mm IonPac AG16 and a 4 × 250 mm IonPac AS16 column in the column oven. Make sure the system pressure is 2300 ± 200 psi when 65 mM KOH is delivered at 1.2 mL/min to allow the degas assembly to effectively remove electrolysis gases from the eluent. If necessary, install additional backpressure tubing between the degas assembly and the injection valve to adjust the system pressure to 2100–2500 psi. Do not allow the pressure to reach 3000 psi. Therefore, monitor the pressure periodically because pressure can gradually rise over time. To reduce pressure, trim the backpressure tubing.

Unlike the ASRS ULTRA suppressor, the ASRS ULTRA II does not require any *Quick Start* using acid regenerants, and the suppressor can be installed after hydration with deionized water. Configure the suppressor for external water mode according to the directions provided in the *ASRS ULTRA II Operator's Manual* (Document No. 031956).

The storage solution of the AS16 column is 35 mM NaOH; equilibrate the column with 65 mM KOH eluent at 1.2 mL/min for approximately 60 min, then analyze a system blank of deionized water. An equilibrated system has a background signal of less than 2 μS and peak-to-peak noise of less than 2 nS. No peaks should elute within the same retention time window as perchlorate. Inject a 25-μg/L perchlorate standard. The column is equilibrated when two consecutive injections of the standard produce the same retention time for perchlorate.

RESULTS AND DISCUSSION

U.S. EPA Method 314.0 specifies the use of an IonPac AS16 column with an eluent of 50 mM NaOH at a flow rate 1.5 mL/min, followed by suppressed conductivity detection with an ASRS ULTRA operated in the external water mode and a 1000-μL large-loop injection. However, Section 6.1.3 of the method states that "An equivalent suppressor device may be utilized provided that comparable conductivity detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background." Section 9.4.3 further states that, "In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns (which meet the criteria in Section 6.1.2.2), injection volumes, and/or eluents, to improve the separations or lower the cost of measurements." Therefore, a different eluent concentration, flow rate, and suppressor may be used for U.S. EPA Method 314.0, provided that the quality control parameters are met. We replaced the ASRS ULTRA specified in the method with an improved ASRS ULTRA II suppressor. The ASRS ULTRA II provides significantly lower noise of 2 nS/min or less, and therefore improves the detection limits for perchlorate. In addition, the KOH concentration was increased from 50 mM to 65 mM, and the flow rate was proportionally adjusted to 1.2 mL/min, as specified in Dionex Application Note 134.

Calibrate the system by injecting one blank and at least five standards to cover two orders of magnitude concentration range. Section 10.2.2 of the method states that the linear calibration range "should not extend over more than two orders of magnitude in concentration." Tabulate the peak area response against the perchlorate concentration injected using a linear regression fit. Table 1 summarizes the calibration data from duplicate injections of 1, 2, 10, 25, 50, and 100 μg/L perchlorate standards. The calibration curve is linear over two orders of magnitude with a correlation coefficient of 0.9998.

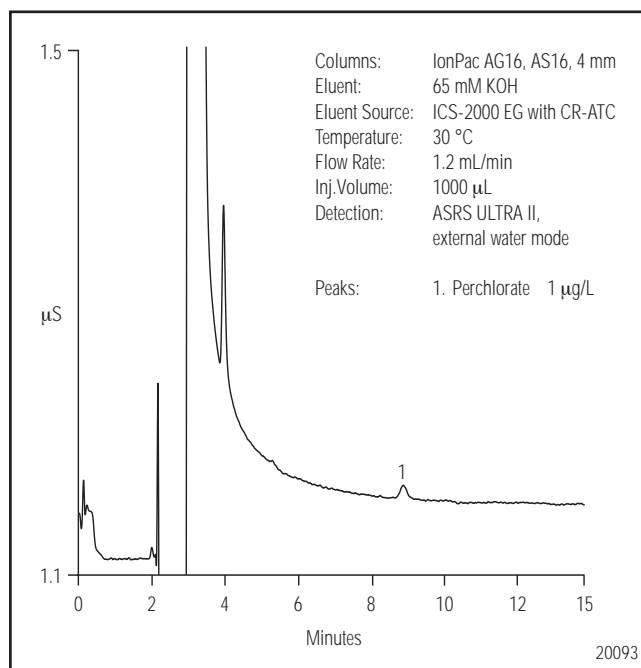|	
Columns:	IonPac AG16, AS16, 4 mm
Eluent:	65 mM KOH
Eluent Source:	ICS-2000 EG with CR-ATC
Temperature:	30 °C
Flow Rate:	1.2 mL/min
Inj. Volume:	1000 μL
Detection:	ASRS ULTRA II, external water mode
Peaks:	1. Perchlorate 1 μg/L

Figure 1. Determination of 1 μg/L perchlorate in deionized water.

Figure 1 shows a chromatogram of a 1-μg/L perchlorate standard using the conditions described in this application update. After establishing the calibration curve, a quality control standard (QCS) of 50 μg/L perchlorate was analyzed resulting in a recovery of 103.8%. This recovery meets the criteria outlined in Section 9.2.5 of the method that states the recovery of the QCS **must** be within ±10% of the stated value.

U.S. EPA Method 314.0 requires an initial demonstration of capability (IDC), as described in Section 9.2. The IDC is used to characterize the instrument and laboratory performance prior to performing any sample analyses by the method. This performance is determined by demonstrating an initial demonstration of accuracy (IDA) and an initial demonstration of precision (IDP) by performing seven replicate injections of a laboratory fortified blank (LFB) fortified with 25 μg/L perchlorate. To meet the requirements of the IDA and IDP, the recovery **must** be within ±10% and the percent RSD **must** be less than 10%, respectively. As shown in Table

Table 1: Calibration Data for Perchlorate		
Analyte	Range (μg/L)[a]	Linearity
Perchlorate	1–100	0.9998

[a] Calibration standards were 1, 2, 5, 10, 25, 50, and 100 μg/L, each injected in duplicate

Table 2: Initial Demonstration of Capability for Perchlorate					
	EPA Method 314.0 Performance Requirements			Experimental Values	
Requirement	Reference	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
IDA	9.2.3	90–110		103.1	
IDP	9.2.4		<10		0.5
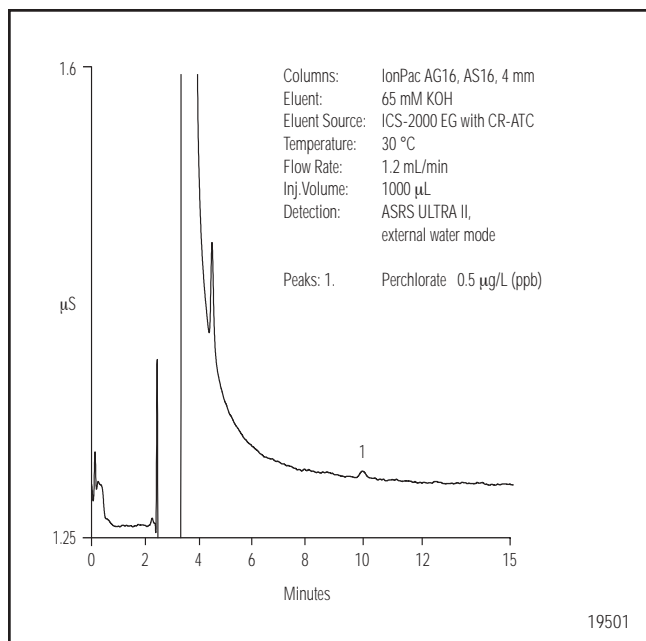
Figure 2. *Determination of 0.5 µg/L perchlorate in deionized water.*

Table 3: Perchlorate MDLs			
Matrix	MDL Standard (µg/L)	Retention Time RSD (%)	Calculated MDL[a] (µg/L)
Deionized Water	0.5	0.10	0.10
MA(50)[b]	0.5	0.20	0.10
MA(100)	0.5	0.05	0.13
MA(200)	1.0	0.27	0.24
MA(400)	2.0	0.07	0.18
MA(600)	5.0	0.07	0.24

[a]The MDLs were calculated as MDL = $(t) \times (SD)$ where t = Student's t value for a 99% confidence level and a standard deviation estimate with n – 1 degrees of freedom (t = 3.14 for seven replicate injections for the MDL standard) and SD = standard deviation of the replicate analyses.
[b]MA indicates a mixed common anion solution of chloride, sulfate, and carbonate included in the sample matrix at the parenthetical mg/L concentration for each anion.

2, our results for the IDA and IDP met the requirements described in Sections 9.2.3 and 9.2.4.

We determined the method detection limit (MDL), as described in Section 9.2.6, by performing seven replicate injections of deionized water fortified with perchlorate at a concentration of three to five times the estimated instrument detection limit. The concentration values determined from the calibration curve were used to calculate the MDL. Figure 2 shows a chromatogram of a 0.5 µg/L perchlorate MDL standard fortified in deionized water. This MDL value is only valid for perchlorate in a "clean" matrix, such as deionized water. The MDL is expected to change as the ionic strength of the sample increases. Therefore, in addition to deionized water, we determined the MDL in MA(50), MA(100), MA(200), MA(400), and MA(600), where MA indicates a mixed common anion solution of chloride, sulfate, and carbonate included in the sample matrix at the parenthetical mg/L concentration for each anion. Table 3 summarizes the results of this study. Because the MDL is based on precision and not accuracy, the determined MDL value for high-ionic-strength matrices, such as MA(400) and MA(600), do not meet the requirement of a fortified perchlorate concentration of three to five times the estimated instrument detection limits. Meeting this condition would typically require the analyst to repeat the MDL at a lower fortified perchlorate concentration. However, determining the MDL using lower perchlorate concentrations is not feasible because high concentrations of common anions interfere with the determination of perchlorate.

Section 9.2.8 describes the matrix conductivity threshold (MCT) as "an individual laboratory defined value" determined by preparing a series of sequentially increasing concentrations of chloride, sulfate, and carbonate fortified with a constant perchlorate concentration. Deionized water fortified with a recommended perchlorate concentration of 25 µg/L must be initially analyzed and followed by a series of increasing anionic solutions of chloride, sulfate, and carbonate, each containing 25 µg/L perchlorate. The recommended 25 µg/L perchlorate assumes that the MRL has been set between 3 µg/L and 5 µg/L. However, if an MRL of 1 µg/L is required, then the MCT should be determined at a perchlorate concentration of 5 µg/L. We determined the MCT using 5 µg/L and 25 µg/L perchlorate. To determine the MCT with 25 µg/L perchlorate, a standard was prepared in deionized water and injected in triplicate. Next, standards containing MA(50), MA(100), MA(200), MA(400), MA(600), MA(800), and MA(1000) were prepared by adding 0.2, 0.4, 0.8, 1.6, 2.4, 3.2, and 4 mL of each common anion from the stock solution (see the section "Preparation of Solutions and Reagents") to separate 120-mL polypropylene bottles. Then, 2.5 mL of perchlorate was added from a 1-mg/L secondary stock dilution standard to each MA solution and dilute each standard to a final volume of 100 mL. A calibrated conductivity meter measured and recorded the conductance for each of these prepared solutions. Section 9.2.8.5 states that the MA(400) solution "should display a conductance of between 3200 µS/cm and 3700 µS/cm."

Table 4 shows the results from this study. Based on multiple determinations, our laboratory determined the MCT with 25 µg/L perchlorate was a value varying from ~4500 µS/cm to 5330 µS/cm. However, individual results may vary within or between laboratories and analysts. The same procedure also determined the MCT using 5 µg/L perchlorate. In this study, the mixed anion solution did not exceed MA(600) because of a significant increase in the percent difference (PD) in the area to height (A/H) ratio. Table 5 shows the results for the MCT study using 5 µg/L perchlorate. Figure 3A and 3B show chromatograms of 5 µg/L and 25 µg/L perchlorate fortified in MA(200), respectively.

Table 4: MCT Study—Perchlorate Fortified at 25 µg/L							
Sample	Conductivty (µS/cm)	Measured ClO_4^- (µg/L)	Percent Recovery	Peak Area	Peak Height	A/H Ratio	PD(A/H) (%)
LFB	<1	25.84	102.6	0.0511	0.223	0.229	0.00
MA(50)	568	25.21	99.9	0.0493	0.211	0.234	2.01
MA(100)	1089	25.25	100.4	0.0494	0.207	0.239	4.25
MA(200)	1979	24.93	99.6	0.0487	0.196	0.249	8.71
MA(400)	3590	24.85	100.2	0.0486	0.182	0.268	16.8
MA(600)	4890	24.30	96.8	0.0475	0.170	0.279	22.0
MA(800)	6070	23.96	95.9	0.0456	0.158	0.288	25.7
MA(1000)	7380	22.76	91.5	0.0454	0.148	0.306	33.6

Table 5: MCT Study—Perchlorate Fortified at 5 µg/L							
Sample	Conductivty (µS/cm)	Measured ClO_4^- (µg/L)	Percent Recovery	Peak Area	Peak Height	A/H Ratio	PD(A/H) (%)
LFB	<1	5.09	101.3	0.0102	0.046	0.222	0.00
MA(50)	588	5.03	100.8	0.0101	0.045	0.224	1.22
MA(100)	1116	5.02	99.1	0.0101	0.045	0.226	1.90
MA(200)	1977	4.76	96.1	0.0094	0.040	0.233	4.93
MA(400)	3660	4.57	90.8	0.0090	0.035	0.259	16.6
MA(600)	4900	3.97	78.1	0.0075	0.026	0.284	28.1

Columns: IonPac AG16, AS16, 4 mm
Eluent: 65 mM KOH
Eluent Source: ICS-2000 EG with CR-ATC
Temperature: 30 °C
Flow Rate: 1.2 mL/min
Inj.Volume: 1000 µL
Detection: ASRS ULTRA II, external water mode

Peaks: 1. Perchlorate 5 µg/L

20094
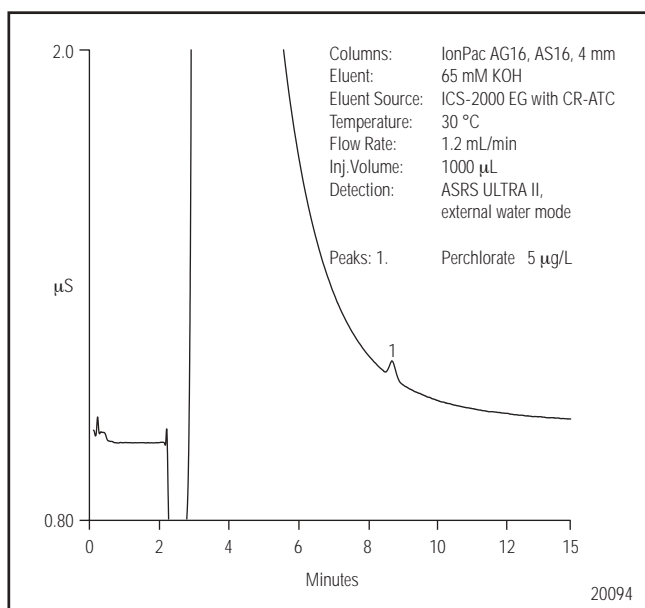
Figure 3A. *Determination of 5 µg/L perchlorate in 200 mg/L each of chloride, sulfate, and carbonate.*

Columns: IonPac AG16, AS16, 4 mm
Eluent: 65 mM KOH
Eluent Source: ICS-2000 EG with CR-ATC
Temperature: 30 °C
Flow Rate: 1.2 mL/min
Inj.Volume: 1000 µL
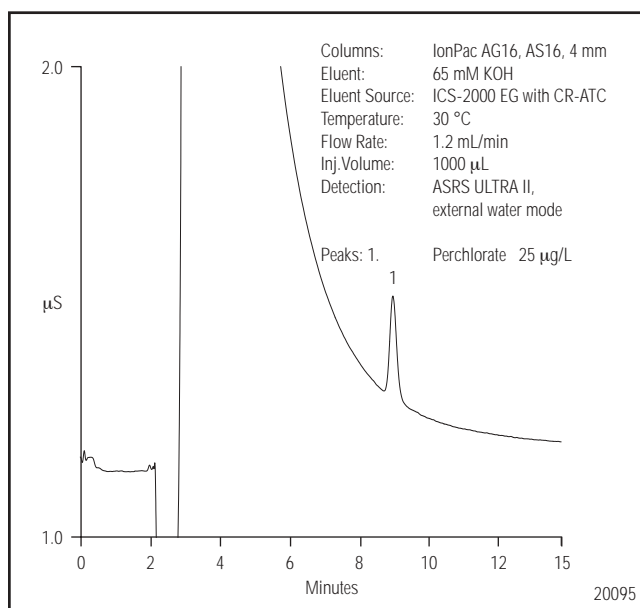Detection: ASRS ULTRA II, external water mode

Peaks: 1. Perchlorate 25 µg/L

20095

Figure 3B. *Determination of 25 µg/L perchlorate in 200 mg/L each of chloride, sulfate, and carbonate.*

Chromatographic performance of perchlorate can deteriorate at high ionic concentrations, primarily due to the presence of high concentrations of chloride, sulfate, and carbonate. Before samples are analyzed, the conductance **must** be determined. If the conductance is greater than the determined MCT, the samples should either be appropriately diluted or pretreated to reduce the common anion concentrations. One way to assess matrix effects is to prepare a laboratory fortified matrix (LFM). An LFM is accomplished by spiking the sample with a known amount of analyte and then determining the percent recovery from the amount added. This application analyzed four matrices: deionized water, drinking water, raw (untreated) drinking water, and surface water. Each matrix was spiked with 1 or 2 μg/L perchlorate and the recoveries were calculated with the equation provided in Method 314.0, Section 9.4.1.3. Table 6 shows the results of this study. The calculated perchlorate recoveries were ~97–108%, which was well within the 80–120% (Section 9.4.1.4) range specified by the method. Figure 4 shows a chromatogram of surface water spiked with 1 μg/L perchlorate.

Table 6: Perchlorate Recoveries from Laboratory Fortified Matrices (LFM)					
Matrix	Conductivty (μS/cm)	Amount Added (μg/L)	Number of Replicates	Precision (% RSD)	Recovery (%)
Deionized water	<1	1.0	7	2.76	99.3
Drinking water	130	1.0	8	10.0	106.8
Raw (untreated) Drinking water	467	2.0	8	5.26	97.6
Surface water	670	1.0	8	12.6	108.2

CONCLUSION

This application update demonstrates an approved approach compared to Dionex Application Note 134 for the determination of perchlorate in environmental samples using U.S. EPA Method 314.0. The lower baseline noise from an ASRS ULTRA II compared to the ULTRA I suppressor improved the limit of detection and quantification of perchlorate resulting in a calculated MDL of 0.1 μg/L in deionized water. The MDLs in high-ionic-strength matrices containing up to 600 ppm each of chloride, sulfate, and carbonate ranged from 0.1 μg/L to 0.24 μg/L perchlorate. Calibration is linear over the range of 1–100 μg/L in deionized water, and acceptable recoveries were obtained for perchlorate spiked at 1–2 μg/L in typical environmental samples. The MCT determined in our lab using 5 μg/L perchlorate was ~3900 μS/cm and the MCT ranged from ~4500 to 5300 μS/cm using 25 μg/L perchlorate. However, results from individual laboratories or analysts may vary. The results presented in this application update meet or exceed the performance requirements specified in U.S. EPA Method 314.0.
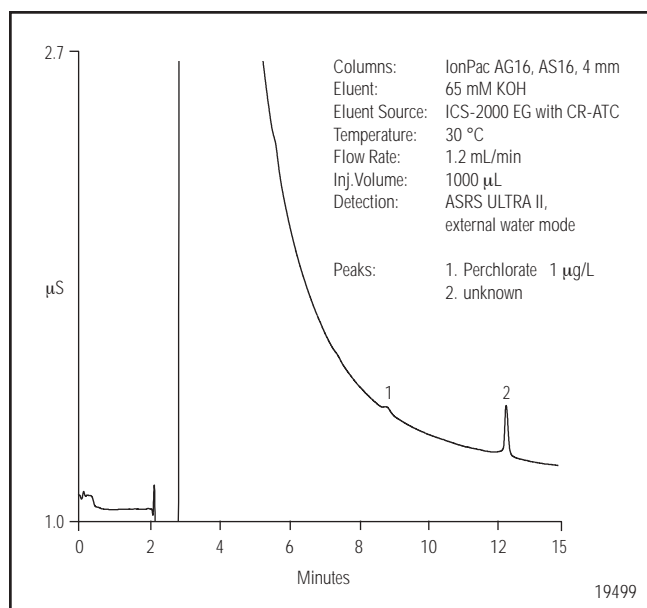

Columns: IonPac AG16, AS16, 4 mm
Eluent: 65 mM KOH
Eluent Source: ICS-2000 EG with CR-ATC
Temperature: 30 °C
Flow Rate: 1.2 mL/min
Inj.Volume: 1000 μL
Detection: ASRS ULTRA II, external water mode

Peaks: 1. Perchlorate 1 μg/L
2. unknown

19499

Figure 4. Trace-level perchlorate spiked into surface water.

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2. Urbansky, E. T.; Collette, T. W. *J. Environ. Monit.* **2001**, 3, 454.

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7. Dionex Corporation. *Determination of Perchlorate in Drinking Water by Ion Chromatography*. Application Update 145; Sunnyvale, CA.

8. *Determination of Perchlorate in Drinking Water Using Ion Chromatography*. Method 314.0; U.S. Environmental Protection Agency; Cincinnati, Ohio, 1999.