

# Determination of Perchlorate in Drinking Water by Ion Chromatography

Dave Thomas and Jeff Rohrer  
Thermo Fisher Scientific, Sunnyvale, CA, USA

## Introduction

Perchlorate anion ( $\text{ClO}_4^-$ ) is an environmental contaminant whose occurrence is most clearly linked with the use of its salts as an oxidizer in rocket propellants, fireworks, matches, and highway safety flares.<sup>1</sup> Through accidental releases and improper disposal of materials containing perchlorate salts, perchlorate has entered the soil, surface water, and groundwater. Its solubility, mobility, and persistence have allowed it to contaminate drinking water in several states throughout the western United States.

Perchlorate presents an environmental health-based risk to humans, particularly women of childbearing age and children. Exposure to perchlorate may cause hypothyroidism by disrupting uptake of iodide by the thyroid gland.

The prescribed analytical method for the determination of perchlorate in drinking and groundwaters is U.S. Environmental Protection Agency (EPA) Method 314.0.<sup>2</sup> Dionex (now part of Thermo Scientific) Application Note 134 (AN134) describes perchlorate determination in water at the 2–4  $\mu\text{g/L}$  level.<sup>3</sup> Dionex (now part of Thermo Scientific) Application Note 151 (AN151) describes perchlorate determination in water at the sub- $\mu\text{g/L}$  level by IC-MS.<sup>4</sup>

This application update describes two modifications to Application Note 134 that can significantly improve routine monitoring of perchlorate in environmental waters. The first modification is to substitute a 2 mm Thermo Scientific™ Dionex™ IonPac™ AS16 column set in place of the 4 mm AS16 column set recommended in Application Note 134. The injection volume is kept at 1000  $\mu\text{L}$ , leading to a potential four-fold increase in sensitivity for perchlorate. The second modification is to substitute a 2 mm Thermo Scientific Dionex AMMS™ III Anion MicroMembrane™ Suppressor operated in the chemical suppression mode in place of the recommended 4 mm Thermo Scientific Dionex ASRS™ ULTRA suppressor. The AMMS III suppressor can provide typical baseline noise of approximately 1–3 nS/minute, which is generally lower than the typical noise of the Dionex ASRS ULTRA suppressor operated as in AN134. For many sample matrices, lower baseline noise will increase the sensitivity of this method for perchlorate.

For water samples that contain appreciable levels of common inorganic anions, matrix elimination can improve detection of perchlorate in the presence of the large matrix ion peak. This application update provides detailed instructions for using Thermo Scientific Dionex OnGuard™ cartridge pretreatment to reduce the concentration of chloride, sulfate, and carbonate in the sample matrix.

In this updated method, the perchlorate anion is determined in 15 min by using a 2 mm Dionex IonPac AS16 column, EG50-generated hydroxide eluent, a 1000  $\mu\text{L}$  injection, and suppressed conductivity detector. The resulting single-operator method detection limit (MDL) for perchlorate in a simulated high-ionic-strength water sample was 0.10  $\mu\text{g/L}$ .

## Equipment

- Thermo Scientific Dionex ICS-2500 system:
  - GP50 Gradient Pump with vacuum degas option
  - EG50 Eluent Generator module
  - Thermo Scientific Dionex EluGen™ Hydroxide Cartridge (EGC II KOH) (P/N 058900)
  - EG50 Degas conversion kit (P/N 055431)
  - Thermo Scientific Dionex AS50 Automated Sampler with thermal compartment
  - 1.0 mL sample syringe for AS50 (P/N 055066)
  - CD25A Conductivity Detector with conductivity cell for AS50 TC (P/N 055400)
  - Thermo Scientific Dionex Chromeleon™ Chromatography Workstation
- Syringe filters (Gelman IC Acrodisc® 0.2  $\mu\text{m}$ , P/N 4483)
- Suppressor External Regen Kit (P/N 038018)

| <b>Conditions</b>         |   |
|---------------------------|---|
| Columns:                  | Dionex IonPac AS16 2-mm Analytical, 2 × 250 mm (P/N 055378)<br>Dionex IonPac AG16 2-mm Guard, 2 × 50 mm (P/N 055379)<br>Dionex IonPac ATC-HC (P/N 059604) |
| Eluent:                   | 50 mM potassium hydroxide (KOH)   |
| Eluent Source:            | EG50  |
| Flow Rate:                | 0.4 mL/min  |
| Temperature:              | 30 °C   |
| Injection:                | 1000 µL (with a 10 µL cut volume from an 1100 µL sample loop)   |
| Detection:                | Suppressed conductivity, Dionex AMMS III, 2 mm<br>Chemical regeneration mode with 50 mN sulfuric acid   |
| Temperature Compensation: | 1.7%/ °C  |
| Background conductance:   | 3–4 µS  |
| Noise:                    | 1–3 nS/min peak-to-peak   |
| Backpressure:             | Adjusted to ~2500 psi   |
| Run Time:                 | 15 min  |

## Preparation of Solutions and Standards

### Reagents and Standards

Prepare all solutions from analytical reagent-grade chemicals.

- Ammonium perchlorate ( $\text{NH}_4\text{ClO}_4$ ) (Alpha Aesar 11658, A.C.S. grade)
- Perchlorate standard, 1000 mg/L (SPEX CertiPrep AS-CIO49-2Y)
- Sodium hydroxide (NaOH) 50% w/w (Fisher Scientific SS254-500)
- Sulfuric acid, 98% (J.T. Baker®, Baker Instra-Analyzed® 9673-33)
- Tribromoacetate ( $\text{CBr}_3\text{COOH}$ ), 99%, (Aldrich, T4, 820-8)

### Deionized Water

Type I reagent-grade distilled or deionized water with a specific resistance of 18.0 MΩ-cm or greater, filtered through a 0.2 µm filter immediately before use.

### Eluent Solution

Generate the 50 mM KOH eluent on-line by using the EG50 module with an EGC II KOH cartridge. Fill the plastic eluent reservoir with deionized water and maintain an inert helium atmosphere of 3–5 psi in the eluent reservoir.

Alternatively, prepare 50 mM NaOH by pipetting 4.0 g of 50% (w/w) aqueous NaOH from the middle portion of the reagent bottle into a 1.00 L volumetric flask containing about 900 mL of degassed deionized water. Do not shake the 50% (w/w) NaOH bottle or pipette from the top of the solution where sodium carbonate may have formed. Dispense the aliquot of NaOH below the surface of the liquid to avoid introducing carbon dioxide from the air into the eluent. Bring to volume with degassed deionized water. Mix and degas by sparging with helium or sonicating under vacuum for 10 min. Store the eluent in a plastic eluent reservoir under an inert helium atmosphere of 3–5 psi to minimize carbonate contamination.

Note that atmospheric carbon dioxide readily dissolves in dilute basic solutions to form carbonate. Carbonate contamination of eluents can cause higher variance in retention time and lower sensitivity. Use of the EG50 module eliminates carbonate contamination of hydroxide eluents and improves retention time stability and sensitivity.

### Regenerant Solution

Prepare 50 mN  $\text{H}_2\text{SO}_4$  by adding 5.7 mL (10.2 g) of concentrated sulfuric acid (18 M) to 4 L of deionized water. Transfer cautiously to the 4 L pressurizable plastic bottle included in the suppressor external regeneration kit.

### Stock Standard Solutions

Purchase a certified perchlorate standard or prepare a 1000 mg/L stock standard solution of perchlorate anion by dissolving 0.1181 g of ammonium perchlorate (0.1231 g of sodium perchlorate) in deionized water and diluting to 100 mL. Store in glass, high-density polyethylene or polypropylene bottles at 4 °C. This stock standard is stable for at least one month.

### Working Standard Solutions

Prepare working standards at lower concentrations by diluting appropriate volumes of the 1000 mg/L stock standard with deionized water. Prepare working standards daily if they contain less than 100 mg/L of perchlorate.

### Surrogate Solution

Prepare a 500 mg/L stock solution by dissolving 0.0500 g of tribromoacetate in deionized water and diluting to 100 mL. Prepare a 50 mg/L spiking solution by diluting 10 mL of the stock solution to 100 mL with deionized water. Store in the refrigerator and bring to room temperature before use. The stability of this solution has not been determined.

## Sample Preparation

Measure the conductivity of samples with a calibrated conductivity meter. Compare the sample's conductivity to the matrix conductivity threshold (MCT) determined in your laboratory, as explained in U.S. EPA Method 314.0, Section 11. Filter low-ionic-strength samples through a 0.2 µm IC syringe filter. Use a hydrophilic polypropylene or polyethersulfone filter; do not use polyvinylidene fluoride (PVDF). Discard the first 300 µL of 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 between 80–120%.

Samples that exceed the MCT can often be analyzed after simply diluting and filtering through a 0.2 µm filter. U.S. EPA Method 314.0 provides guidelines for determining the MCT and the appropriate dilution factor. For diluted samples, the MDL **must** be raised in proportion to the dilution factor.

To analyze high-ionic-strength samples without dilution, reduce the concentration of matrix ions by treating the sample with Dionex OnGuard cartridges. Sulfate can be removed by using the Dionex OnGuard II Ba cartridge, chloride can be removed by treating with the OnGuard II Ag cartridge, and carbonate can be reduced by treating with the OnGuard II H cartridge, followed by sparging with helium gas. These cartridges can be used separately or in combination. Follow the instructions for using all three Dionex OnGuard II cartridges in the combination given in the Installation and Troubleshooting Guide for the OnGuard II Cartridges (Document No. 031688). Refer to Section 2.9.1 and Figure 4 in Section 2.9.4 of that guide.

Prepare the Dionex OnGuard cartridges by rinsing each cartridge separately with 15 mL of deionized water. Arrange in series an OnGuard II Ba 2.5 cc (P/N 057094) followed by an OnGuard II Ag 2.5 cc (PN 057090).

**Important:** You must modify the procedure depicted in Figure 4 of Section 2.9.4 of the Dionex OnGuard installation and troubleshooting guide. Insert a Gelman IC Acrodisc 0.2 µm filter between the Dionex OnGuard II Ag cartridge and the final OnGuard II H 2.5 cc (P/N 057086). This filter will trap colloidal silver particles that otherwise might foul the Dionex IonPac AG16 guard column, AS16 analytical column, or suppressor.

The Dionex OnGuard cartridge installation and troubleshooting guide recommends spiking the sample with a divalent displacing agent ( $Mg^{+2}$  or  $Ca^{+2}$ ) before pretreatment to improve sulfate elimination. Omit this step unless you observe significant sulfate breakthrough with a particular matrix. Many high-ionic-strength samples contain enough sodium or other cations to ensure good results without this added step. If necessary, follow the recommended procedure to see if sulfate elimination improves.

After preparing the cartridges, assemble in series as instructed above. Pass 10 mL of the undiluted sample through the cartridge. Discard the first 6 mL and collect the remainder for dilution or direct injection. With each batch of Dionex OnGuard pretreated samples, include at least one laboratory reagent blank (LRB) using deionized water through each step of the procedure. The blank should be free of peaks within the retention time window of perchlorate. If not, increase the deionized water rinse volume used to prepare each Dionex OnGuard cartridge to 45 mL and analyze a new LRB.

Samples pretreated before analysis should be used to prepare a laboratory fortified matrix (LFM) as described in U.S. EPA Method 314.0. Fortify an aliquot of the sample with perchlorate at 50–100% of the level determined in the native sample. To reduce the concentration of common anions, pretreat the fortified aliquot and then analyze. The recovery of perchlorate should be 80–120% to rule out matrix effects and confirm that the laboratory performed the Dionex OnGuard pretreatment correctly.

We also recommend adding a surrogate to the sample before pretreatment. A surrogate is a chemical species that has chemical and physical properties similar to the analyte, is stable and nonreactive, and can be added in a known and easily measured quantity. Add 100 µL of the 50 mg/L tribromoacetate\* spiking solution to 50 mL of deionized water. This produces a level of 100 µg/L. Determine the peak area for tribromoacetate by directly injecting onto the ion chromatograph. Add 100 µL of the 50 mg/L tribromoacetate spiking solution to 50 mL of the sample before pretreatment. Pretreat with Dionex OnGuard cartridges and analyze. The peak area of tribromoacetate in the treated sample should lie between 80–120% of that in the spiked water sample.

\* Tribromoacetate was used as a surrogate for this work. Other surrogates may be more appropriate.

## System Preparation and Setup

Verify that the pump flow rate is within specifications and recalibrate if necessary. Verify that the conductivity cell constant is within specifications and recalibrate if necessary. Consult the pump or detector manuals for procedural details.

Install the EG50 module, and then install the EG50 degas conversion kit (P/N 055431) to purge the electrolysis gas from the freshly generated eluent. Install a Dionex IonPac ATC-HC column between the pump outlet and the inlet of the Dionex EGC II KOH EluGen Cartridge. The new Dionex ATC-HC column has a higher capacity (4.2 meq/column) and provides an overall cleaner product than previous anion trap columns. Regenerate it as needed by using a trap column/suppressor cleanup kit (P/N 059659) according to the Installation and Instruction Manual (Document No. 031835)

Condition the Dionex EluGen II KOH cartridge as directed in the EG50 module manual by running a gradient from 1 to 60 mN KOH in 20 min, then 60 mN for 40 min at 1 mL/min. For instructions on EG50 module installation and use, see the operator's manual for the EG50 Eluent Generator System (Document No. 031908).

Install and configure the autosampler and thermal compartment. The precision and accuracy of the autosampler will vary depending on the mode of injection. The most accurate and precise injections are made with a calibrated sample loop, flushed with about five loop volumes of sample. The largest full-loop injection possible with the Dionex AS50 autosampler is 300  $\mu\text{L}$ . To inject 1000  $\mu\text{L}$ , use the partial-loop injection mode with a 1100  $\mu\text{L}$  sample loop, and a programmed “Sample Loop Volume” of 1100  $\mu\text{L}$  and “Cut Volume” of 10  $\mu\text{L}$ . 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 Dionex AS50 Plumbing Configuration screen. Refer to the Dionex AutoSelect AS50 Autosampler Operator’s Manual (Document No. 31169) for details.

Install a 2  $\times$  50 mm Dionex IonPac AG16 and a 2  $\times$  250 mm IonPac AS16 column. Make sure the system pressure displayed by the pump is at least 2300 psi when 50 mM KOH is delivered at 0.4 mL/min, so the degas assembly can effectively remove electrolysis gas from the eluent. If necessary, install backpressure coils supplied with the EG50 module ship kit to adjust the system pressure to 2300–2800 psi. Because the system pressure can rise over time, trim the backpressure coil as necessary to maintain system pressure under 3000 psi. Do not exceed 3000 psi or the degas assembly tubing may rupture.

Prepare the Dionex AMMS III suppressor for use by hydrating the eluent chamber. Pump approximately 5 mL of regenerant (50 mN  $\text{H}_2\text{SO}_4$ ) through the Regen In port. Pump approximately 5 mL of eluent through the Eluent In port. Allow the suppressor to sit for approximately 20 min to fully hydrate the suppressor screens and membranes. Install the Dionex AMMS III suppressor in the external pressurized bottle mode by following the Installation and Troubleshooting Instructions for the AMMS III (Document No. 031727). Adjust the head pressure on the external regenerant reservoir to deliver a flow rate of 2–3 mL/min. (We coupled a 3 in. piece of 0.01 in. i.d. PEEK™ tubing to the very end of the suppressor waste line, then trimmed it until a pressure of 10–15 psi in the regenerant reservoir provided a flow of 2–3 mL/min.)

The storage solution that ships with the Dionex IonPac AS16 is 35 mM NaOH. Equilibrate the column with 50 mM KOH eluent for 60 min, then analyze a system blank of deionized water. An equilibrated system has a background signal of less than 4  $\mu\text{S}$  and peak-to-peak noise of less than 3 nS. There should be no peaks eluting within the retention time window of the perchlorate anion.

Inject a 10  $\mu\text{g/L}$  perchlorate standard. The column is equilibrated when two consecutive injections of the standard produce the same retention time for perchlorate. Confirm that the resulting chromatogram resembles the chromatogram of the 2  $\mu\text{g/L}$  standard shown in Figure 1A.

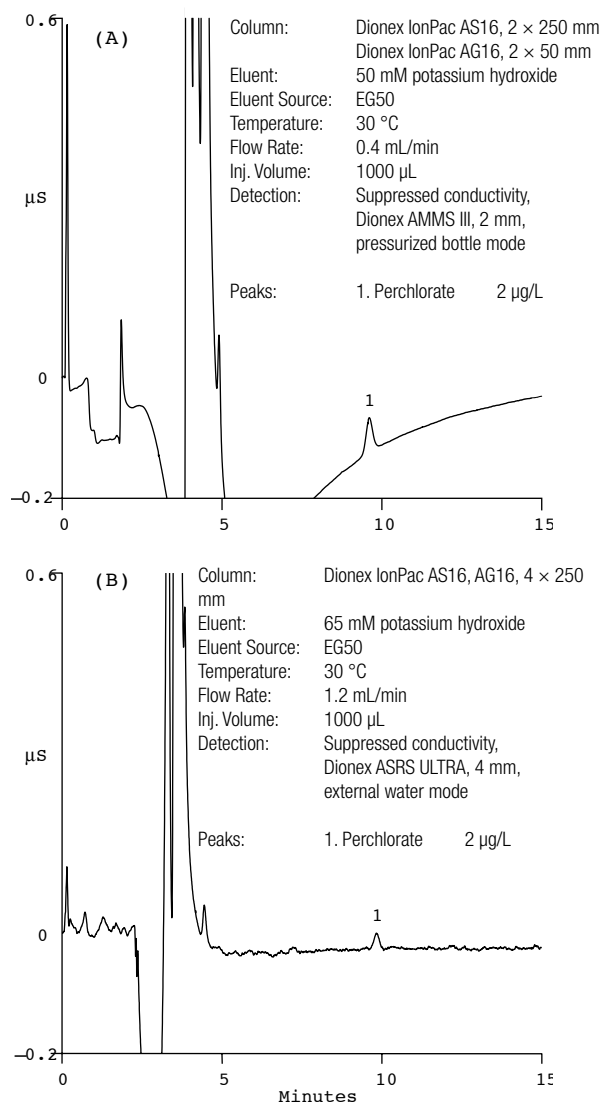


Table 1. Comparison of 2 ppb perchlorate by AU 145 (A) and AN 134 (B).

## Results and Discussion

Calibrate the system by injecting one blank and at least five standards for every two decades of the calibration range. Plot the peak area for perchlorate versus the concentration injected and use a linear regression to fit the data. Table 1 summarizes the calibration data for a typical calibration curve obtained by injecting calibration standards at 0.25, 0.5, 1, 2, 4, 10, 50, and 100 µg/L. The calibration curve is linear over two orders of magnitude with a correlation coefficient of 1.0000. Figure 1A shows a chromatogram of a 2 µg/L perchlorate standard obtained by using the optimized conditions described above, compared to the chromatogram in Figure 1B of a 2 µg/L perchlorate standard obtained by using the conditions described in AN134.

Table 1. Linear range and MDLs for perchlorate.

| Matrix          | Range (µg/L) | R <sup>2</sup> | MDL Standard (µg/L) | SD (µg/L) | *Calculated MDL (µg/L) |
|-----------------|--------------|----------------|---------------------|-----------|------------------------|
| Deionized Water | 0.25-100     | 1.000          | 0.5                 | 0.019     | 0.1                    |
| **HIW           |              |                | 0.5                 | 0.032     | 0.1                    |

\* 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 replicates of the MDL standard) and  $SD$  = standard deviation of the replicate analysis.<sup>5</sup>

\*\* High-ionic-strength water (HIW) contained 600 mg/L each of chloride, sulfate, and carbonate.

Determine the MDL for perchlorate by making seven injections of a low-level solution fortified with perchlorate at a level yielding a signal-to-noise of about 3–5. Then use the concentration values determined from the calibration curve to calculate the MDL.

We determined the MDL for perchlorate in deionized water by making seven replicate injections of deionized water fortified with perchlorate at 0.5 µg/L. Figure 2 shows the chromatogram of one MDL standard and Table 1 shows the calculated MDL. The MDL is defined as the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.<sup>5</sup> It is basically a measure of the precision of preparing and analyzing low-level samples according to the method. The MDL for perchlorate in deionized water obtained by this updated method is approximately three times lower than that demonstrated in AN134. The improvement is the result of both the 2 mm column format and the lower noise provided by the Dionex AMMS III suppressor operating in chemical regeneration mode.

Column: Dionex IonPac AS16, 2 × 250 mm  
 Dionex IonPac AG16, 2 × 50 mm  
 Eluent: 50 mM potassium hydroxide  
 Eluent Source: EG50  
 Temperature: 30 °C  
 Flow Rate: 0.4 mL/min  
 Inj. Volume: 1000 µL  
 Detection: Suppressed conductivity, Dionex AMMS III, 2 mm, pressurized bottle mode

Peaks: 1. Perchlorate 0.5 µg/L (ppb)  
 2. Tribromoacetate 100

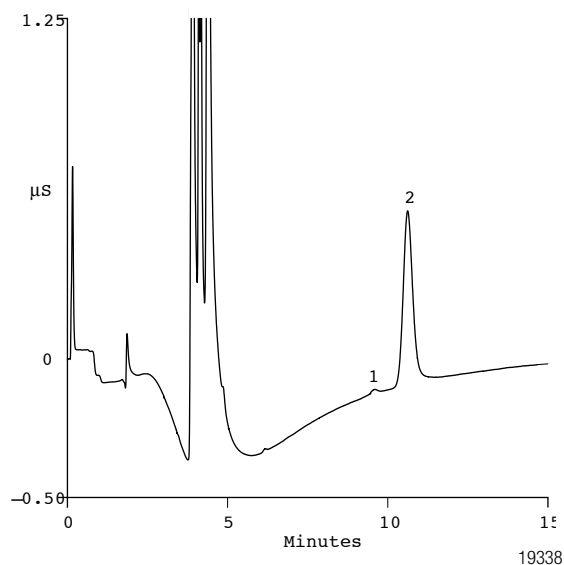


Figure 2. Analysis of a 0.5 µg/L perchlorate standard and tribromoacetate surrogate.

Chromatographic performance of the perchlorate method can deteriorate as the ionic strength of the sample matrix increases due to increased concentration of sulfate, chloride, and carbonate. HIW samples must be diluted or pretreated to restore optimal chromatographic performance. One approach is to pretreat HIW matrix samples by using Dionex OnGuard cartridges to remove common matrix ions. This pretreatment prevents overloading of the Dionex IonPac AS16 column by the matrix ions and improves the perchlorate peak shape (that is, taller and narrower). Also, if these steps are not taken, the perchlorate peak can elute on the tailing edge of the matrix peak, making it more difficult to reliably integrate and quantify at low ppb levels. U.S. EPA Method 314.0, Section 11, gives rules to help decide which samples should be pretreated. Each laboratory should follow the rules in Section 9.28 to determine the MCT for each matrix. Measure each sample's conductivity before analysis and decide whether the sample can be directly injected, diluted and injected, or pretreated with Dionex OnGuard cartridges prior to injection.

We used the Dionex OnGuard pretreatment procedure to determine the MDL for perchlorate in a HIW matrix containing chloride, sulfate, and carbonate at 600 mg/L each. Seven injections were made of HIW samples fortified with perchlorate at a concentration of 0.5 µg/L. The fortified HIW samples were treated with the Dionex OnGuard Ba, OnGuard Ag, and OnGuard H cartridges as described in the "Sample Treatment" section. Figure 3 compares the chromatogram of the HIW MDL sample before treatment (3A) with the chromatogram after treatment with Dionex OnGuard cartridges (3B). Table 1 presents the MDL calculated for the HIW. The favorable comparison between the MDL in deionized water and the MDL in HIW treated with Dionex OnGuard II cartridges demonstrates that pretreatment can effectively achieve high sensitivity, even in challenging samples. The MDL of 0.10 µg/L permits a minimum limit (ML) for quantitation of 0.3–0.5 µg/L for perchlorate, which is adequate for routine analysis at the level of 1.0 µg/L.

Although the Dionex IonPac AS16 column specified in U.S. EPA Method 314 has a relatively high capacity of 170 µeq/column, the large injection volume used in this application update increases the possibility of interference from other anions in the sample. Some drinking and ground waters can contain elevated levels of chloride, sulfate, and carbonate, as well as other common anions.

Column: Dionex IonPac AS16, 2 × 250 mm  
Dionex IonPac AG16, 2 × 50 mm  
Eluent: 50 mM potassium hydroxide  
Eluent Source: EG50  
Temperature: 30 °C  
Flow Rate: 0.4 mL/min  
Inj. Volume: 1000 µL  
Detection: Suppressed conductivity,  
Dionex AMMS III, 2 mm,  
pressurized bottle mode

Peaks: 1. Perchlorate 0.5 µg/L (ppb)  
2. Tribromoacetate 100  
Matrix: Chloride 600 mg/L  
Carbonate 600  
Sulfate 600

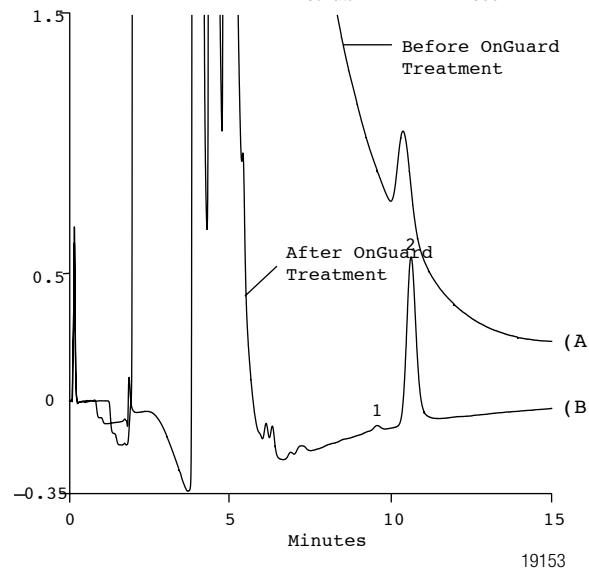


Figure 3. Determination of perchlorate at 0.5 µg/L before (A) and after (B) using Dionex OnGuard sample pretreatment.



One way to assess matrix effects is to prepare laboratory fortified matrix (LFM) samples. This assessment is done by spiking suspect matrices with a known amount of analyte and measuring the percent recovery of the amount added. Some EPA methods require that this be done in duplicate, because the precision of the LFM analysis provides additional information on matrix effects. We assessed recovery of perchlorate spiked into four matrices: deionized water, drinking water, groundwater, and a laboratory-prepared HIW matrix—all before and after pretreatment with Dionex OnGuard cartridges. Table 2 shows the results of this study.

This method provides acceptable performance (that is, greater than 80% recovery) in the presence of up to 600 mg/L chloride, 600 mg/L sulfate, and 600 mg/L carbonate. Typical ground and drinking waters are not expected to contain more than 200 mg/L chloride or 500 mg/L sulfate.

### Interferences

Most common anions elute well before perchlorate under these conditions, as shown in Application Note 134. High levels of chloride, sulfate, and carbonate can produce a tailing matrix ion peak that makes integration of the perchlorate peak more difficult. High levels of these ions can also distort the peak shape of perchlorate enough to make integration difficult. As in U.S. EPA Method 314.0, an MCT should be established for the method by each laboratory, and samples exceeding the MCT should be diluted or treated with Dionex OnGuard cartridges. Broad

tailing peaks are occasionally observed in some drinking water matrices. These peaks may be pyrophosphate or other higher-order phosphates.

### Precautions

Peak area precision and accuracy depend on autosampler performance. Replace the water in the flush reservoir daily with freshly filtered and degassed water. Inspect the Dionex AS50 autosampler daily for bubbles in the sample syringe or its tubing. Purge to remove any bubbles by following the instructions in the Dionex AS50 autosampler manual.

Strongly retained compounds from injected samples can accumulate on the column and degrade its performance. Signs of a fouled column include loss of capacity, loss of resolution, shortened retention times, higher noise and background, spurious peaks, and peak tailing. The Dionex IonPac AS16 column can be flushed with up to 100% acetonitrile to help remove contaminants from the column. For more information on column troubleshooting and cleanup, see the Installation Instructions and Troubleshooting Guide for the IonPac AS16 Analytical Column (Document No. 031475).

Some groundwater samples contain particulates that will plug the column and increase the backpressure. Use a guard column to protect the analytical column; change the guard column if a sample causes a sudden increase in total backpressure to greater than 3000 psi.

Table 2. Perchlorate recovery from laboratory fortified matrices (LFMs).

| Matrix          | No Treatment        |              |                   | With Dionex OnGuard Treatment |              |                   |
|-----------------|---------------------|--------------|-------------------|-------------------------------|--------------|-------------------|
|                 | Amount Added (µg/L) | Recovery (%) | Precision (% RSD) | Amount Added (µg/L)           | Recovery (%) | Precision (% RSD) |
| Deionized Water | 0.5                 | 100          | 3.9               | 0.5                           | 97           | 1.5               |
| Deionized Water | 0.5                 | 78           | ND                | 0.5                           | 114          | ND                |
| Groundwater     | 1.0                 | 93           | ND                | 1.0                           | 84           | 9.4               |
| *HIW            | 0.5                 | ND           | ND                | 0.5                           | 98           | 9.4               |

\* HIW contained 600 mg/L each of chloride, sulfate, and carbonate.  
ND = not determined

## Summary

Modifications to AN134— including the use of a 2 mm Dionex IonPac AS16 column set, 2 mm Dionex AMMS III suppressor operating in chemical suppression mode, and Dionex OnGuard pretreatment to reduce interference from common matrix ions—significantly increase the sensitivity for perchlorate. The resulting MDL for perchlorate is 0.1 µg/L in a high-ionic-strength water matrix containing 600 mg/L each of chloride, sulfate, and carbonate. Calibration is linear over the range 0.25–100 µg/L in deionized water and quantitative recoveries were obtained for perchlorate spiked at 0.5 µg/L in drinking water. The modified method provides acceptable performance—in terms of peak shape and recovery—in the presence of up to 600 mg/L chloride, 600 mg/L sulfate, and 600 mg/L carbonate.

These results were obtained using a 2 mm Dionex IonPac AG16/AS16 column set with a 1000 µL injection of sample. To achieve comparable results with a 4 mm column set would require injecting 4000 µL of sample.

## References

1. Jackson, P. E.; Gokhale, S.; Streib, T.; Rohrer, J. S.; Pohl, C. A. *J. Chromatogr., A* **2000**, *888*, 151.
2. *Determination of Perchlorate in Drinking Water Using Ion Chromatography*. Method 314.0; U.S. Environmental Protection Agency; Cincinnati, Ohio, 1999.

3. Dionex Corporation (now part of Thermo Scientific). *Determination of Low Concentrations of Perchlorate in Drinking and Ground Waters Using Ion Chromatography*. Application Note 134; Sunnyvale, CA.
4. Dionex Corporation (now part of Thermo Scientific). *Determination of Perchlorate in Environmental Waters by Ion Chromatography Coupled with Electrospray Mass Spectrometry (IC-MS)*. Application Note 151; Sunnyvale, CA.
5. *The Determination of Inorganic Anions in Water by Ion Chromatography*. Method 300.0; U.S. Environmental Protection Agency; Cincinnati, Ohio, 1993.

## Suppliers

Aldrich Chemical Company, Inc., 1001 West Saint Paul Avenue, P.O. Box 355, Milwaukee, WI 53233 USA, Tel.: 800-558-9160, [www.aldrich.sial.com](http://www.aldrich.sial.com).

Alfa Aesar, 30 Bond St., Ward Hill, MA 01835 USA, Tel.: 800-343-0660, [www.alfa.com](http://www.alfa.com).

Fisher Scientific, 2000 Park Lane, Pittsburgh, PA 15275-1126 USA, Tel.: 800-766-7000, [www.fishersci.com](http://www.fishersci.com).

VWR Scientific Products, 1310 Goshen Parkway, West Chester, PA 19380 USA, Tel.: 800-932-5000, [www.vwr.com](http://www.vwr.com).

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**Japan** +81 6 6885 1213  
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**Netherlands** +31 76 579 55 55  
**Singapore** +65 6289 1190  
**Sweden** +46 8 473 3380

**Switzerland** +41 62 205 9966  
**Taiwan** +886 2 8751 6655  
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