

Determination of Trace Perchlorate in High-Salinity Water Samples by Ion Chromatography with On-Line Preconcentration and Preelution

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A simple, automated system for the determination of trace perchlorate by ion chromatography (IC) with an online preconcentration technique is reported. The sample is preconcentrated, and less strongly held ions preeluted before the analyte is transferred to the principal separation system. This approach provides low limits of detection (LOD) and is particularly robust toward the effect of high concentrations of common anions, such as those present in groundwater samples. It compares favorably with currently promulgated EPA method 314.0. The LOD (S/N = 3) is 0.77 $\mu\text{g/L}$ for a 2-mL reagent water sample and decreases more-or-less proportionately with increasing sample volume, at least up to 20 mL. Even with a sample of conductivity 14.7 mS/cm (approximately that of 0.1 M Na_2SO_4), the recovery of added perchlorate at the 25.0 $\mu\text{g/L}$ level was still 92%. The concentration of added perchlorate in the range of 1–400 $\mu\text{g/L}$ was linearly correlated to the peak area, with an r^2 value of 0.9997. The recovery of perchlorate from artificial samples with different conductivity by the present method compares favorably with those from the currently recommended EPA Method. The ability of this approach to remove matrix interferences suggests that it would be also promising for perchlorate analysis in other challenging samples.

Trace levels of perchlorate in drinking water and groundwater are currently of great concern. Even at very low levels, perchlorate exposures can affect a developing nervous system and lead to other human health problems. Perchlorate interferes with iodide uptake by the thyroid gland; the consequent dysfunction in producing thyroid hormones can cause cancer. There have been confirmed perchlorate releases in at least 20 states throughout the United States. Based on a preliminary toxicity assessment, the draft estimate of the U.S. Environmental Protection Agency (EPA) for a future regulatory limit of perchlorate in drinking water is 1 $\mu\text{g/L}$. With the exception of bromate, no other ions in water have ever been regulated at such a low level. The California Department of Health Services has already adopted an action level

for perchlorate in drinking water of 4 $\mu\text{g/L}$.¹ Extensive information is available on the topic from the EPA^{2–5} and specifically in papers by Urbansky.^{6–10} Urbansky has also critically reviewed analytical methods available for perchlorate. At the levels of interest, any direct method involving photometry or electrochemical measurement is generally inadequate with respect to attainable limits of detection (LODs); there are also possible issues with specificity. Successful separation from other constituents by capillary electrophoresis (CE)^{11–14} has been reported, but achieving necessary LODs without preconcentration would not generally be possible, and electrophoretic preconcentration is complicated by the fact that it is sample-ionic-strength-dependent.^{15,16} Only suppressed conductometric CE^{17,18} would have had the potential to be sufficiently sensitive without preconcentration, but the necessary instrumentation is not commercially available. We thus agree wholeheartedly with Urbansky's final assessment regarding practical perchlorate measurement that "... in the near future, we can expect ion chromatography to dominate environmental analytical chemistry both because of the limit of detection and the availability of the instrumentation ...".¹⁰

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In some cases, especially when legal issues are involved, mass spectrometry must be used after IC separation to provide unambiguous identification.^{19,20} Several direct mass spectrometric approaches without prior separation have also been advanced.^{8,9,21,22} It should be noted, however, that the use of MS approaches without prior IC separation does not generally provide better LODs than that attainable by suppressed conductometric IC, especially for high-salt groundwater samples. For routine monitoring, IC is presently the only practical choice. Further, the very high retention time (t_R) of ClO_4^- relative to other common ions removes a lot of uncertainties commonly associated with retention-time-based identification. The ionic charge of the peak suspected to be perchlorate can also be confirmed by a change in the eluent concentration (C) from the known dependence of $\log t_R - \log C$ relationship on the analyte charge.^{23,24}

Although LODs as low as 0.1 $\mu\text{g/L}$ have been reported for drinking water analysis after chloride removal with an Ag^+ -form resin and 20-fold evaporative preconcentration of the sample,²⁵ this benefit would be generally applicable to any analysis method; moreover, this is not particularly suitable for routine automated analysis. To summarize other IC approaches for measuring perchlorate appearing in the last 5 years, Nair et al.²⁶ described a new methacrylate-based stationary phase and Maurino and Minero²⁷ described cyanuric acid as a novel eluent. In both cases, chromatograms were obtained at many milligram per liter levels; no LOD was specified, and t_R values were long. Okamoto et al.²⁸ described a practical determination procedure with a phenoxide eluent, and an LOD of 0.7 $\mu\text{g/L}$ was reported for a 740 μL injection with a t_R of ~ 8 min; the presence of 600–1000 mg/L sulfate (specific conductance 1500–2600 $\mu\text{S/cm}$) reduced perchlorate recovery to 91–93%. However, *p*-cyanophenol is a toxic and harmful substance in the TSCA inventory of EPA, and oxidation products of the eluent slowly poison column and suppressor components, reducing their useful lifetime. Better approaches appeared almost concurrently: with the use of 100 mM NaOH eluent and an AS11 column, a 1000- μL injection produced an LOD of 0.3 $\mu\text{g/L}$ in reagent water, and the concurrent presence of 50–1000 mg/L sulfate reduced the recovery of 20 $\mu\text{g/L}$ ClO_4^- to vary from 93 to 100%.²⁹ An improved method relying on an AS16 stationary phase (developed exclusively for perchlorate determination) appeared subsequently, with a reported LOD of 0.15 $\mu\text{g/L}$ for 1000- μL injection of a reagent water matrix and a t_R of ~ 10 min; 50–1000 mg/L sulfate caused recovery of 20 $\mu\text{g/L}$ ClO_4^- to

vary from 94 to 97%.³⁰ This is also the basis of the currently promulgated EPA method.³¹ Recently, DeBorba et al. described the use of poly(vinyl alcohol)-based gel resin columns for the determination of perchlorate^{32–34} and provided validation studies.

Over large areas of the western and southwestern United States, gypsum layers in the bedrock are common, and groundwater can contain sulfate at very high levels. This is particularly important because many military bases are in this same geographic region where groundwater needs to be examined for contamination. In many cases, there is also a need for routine monitoring as remediation efforts are carried out. Chromatograms in the literature^{29,30} indicate that it would be impossible to determine low or submicrogram per liter levels of perchlorate by direct adaptation of the above approach, as we found with relevant groundwater samples from the Texas panhandle. The EPA recommendations³¹ involve sequential pretreatment of such samples through Ba^{2+} , Ag^+ , and H^+ -form resins. This is cumbersome and difficult to automate; moreover, we were actually unable to attain acceptable recoveries in most of our groundwater samples. We provide here a simple, automated analytical procedure that is applicable not only to drinking water samples but also to these much more difficult high-sulfate groundwater samples. A short hydrophilic column is used to preconcentrate perchlorate. A dilute NaOH solution is used to prewash the sample loaded in the preconcentrator column to remove the less strongly held anions prior to switching the preconcentration column to the main separation column. In this paper, we discuss the operational conditions, linear dynamic range and LOD, applicable sample volume, and effects of sample conductivity. The performance of the system is compared with EPA method 314.0.

EXPERIMENTAL SECTION

Reagents. All solutions were prepared in 18.2 $\text{M}\Omega\cdot\text{cm}$ Milli-Q water with a 0.45- μm outlet filter. Sodium hydroxide, 50% (w/w) aqueous solution, NaCl, Na_2SO_4 , Na_2CO_3 , and KCl were analytical reagent grade (Fisher). Sodium perchlorate standard solution was obtained as an IC custom standard from AccuStandard, Inc. (New Haven, CT). The two highly saline groundwater samples, known not to contain perchlorate and used as the blank matrix in this work, were obtained from (1) Pantex, a facility operated by the U.S. Department of Energy (www.Pantex.com) and (2) field wells of west Texas, provided by the Texas Commission on Environmental Quality (TCEQ).

The eluent (100 mM NaOH) was prepared by dissolving 8.0 g of 50% NaOH in reagent water to a final volume of 1.0 L and used under a 5 psi helium blanket. Artificial samples containing various common anions of measured conductivity, the perchlorate standard solutions, perchlorate spiked samples, and the conductivity calibration standards were prepared as prescribed.³¹

Instrumentation. A Dionex IC25 chromatograph equipped with a LC25 oven maintained at 35 °C and an AS40 autosampler

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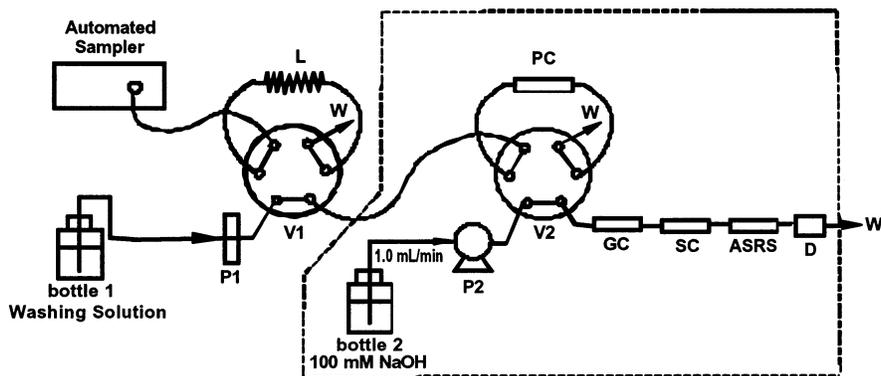


Figure 1. Schematic diagram for proposed system. P1, peristaltic pump; L, sample loop; PC, pre-concentration column; GC, guard column; SC, separation column; ASRS, electrodialytic suppressor; D, conductivity detector; V1, low pressure 6-port valve; V2, 6-port chromatographic injector; P2, chromatographic pump; W, waste.

was used. An AG11 (4×50 mm) guard column was used in conjunction with an AS16 (4×250 mm) analytical column for separation. The 100 mM NaOH eluent was used isocratically at a flow rate of 1.0 mL/min. An ASRS-Ultra suppressor operated at 300 mA in the external water mode was also used. An Accumet AB30 conductivity meter (Fisher Scientific) was used to measure the conductivity of samples.

Figure 1 shows the present system schematic diagram. The dashed enclosure shows the normally used^{30,31} configuration, except that the sample loop is replaced by a pre-concentration column, PC (TAC-LP1 4×35 mm, Dionex). A low-pressure 6-port loop injector (shown with a 1.0-mL loop, although up to 18-mL loops have been used, VICI, with an electric actuator) is situated between the autosampler and the load port of the high-pressure injector. The actuation of the low-pressure valve is controlled by software command using one of the relay outputs of the IC. A peristaltic pump (MasterFlex C/L, model 77120-70, Cole-Parmer Instrument Co., provided with 0.020×0.092 in. i.d. \times o.d. "solvent-flexible" Tygon pump tubing) provides the capability of washing/preeluting the PC using a dilute NaOH solution; a flow rate of 0.6–0.8 mL/min is used. All tubing used for connections are PEEK (0.32-mm i.d.). System control is carried out with Dionex PeakNet 6.0 software.

Procedure. (1) The autosampler fills the sample loop L in V1. (2) V1 then switches to the inject position after 4.5 min (varied depending on loop volume) so that the loop contents are transferred to the PC by the wash solution pumped by P1. (3) The sample transferred to the PC is washed for a period (hereinafter referred to as prewash time). (4) V2 is switched to the inject position for 1.2 min so that the remaining sample is injected into the separation system proper. (5) After perchlorate is separated from the other anions in the analytical column, the corresponding suppressed signal is detected by the conductometer D. P1 can be kept running during the entire period or shut off through another relay output from the IC to conserve the wash solution during the time V2 is in the inject mode.

RESULTS AND DISCUSSION

Wash Solution Concentration and Prewash Volume/Period. The prewashing strategy takes advantage of the fact that the matrix ions are generally less strongly retained than perchlorate and can thus be preferentially eluted before the precon-

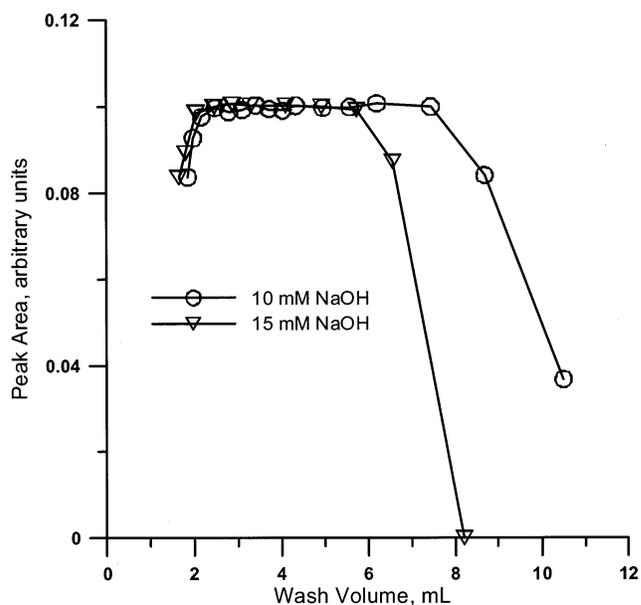


Figure 2. Peak area as a function of preeluent volume. 10 and 15 mM NaOH were used at flow rates of 0.62 and 0.82 mL/min, respectively.

trated sample is injected. However, an excessively high concentration of the wash solution can interfere with the uptake of the analyte, and if premature elution of perchlorate itself is to be avoided, the total optimal prewash volume may be so short for a high-concentration wash that accurate control will be difficult. On the other hand, a minimum prewash volume is necessary to transfer the loop contents to the PC. Additionally, too dilute a wash solution will result in inefficient washing of the column (permitting a greater than desirable amount of the matrix to be injected to the separation system) or too great a wash time will be needed. For a maximum sample-throughput rate, the sum of the sample load and the prewash period should not be greater than the chromatographic analysis time. For the sample matrix range of interest, 10 or 15 mM NaOH was found to be adequate as the preeluent. Figure 2 shows the experimental results for 8.0 ng of perchlorate ($8.0 \mu\text{g/L}$, 1.0 mL) loaded on the column. For 10 mM NaOH as the preeluent, a 2.2–7.4-mL wash volume is permissible; for 15 mM NaOH, the corresponding volume is 2.0–5.8 mL. At very low wash volumes, loop contents are not completely trans-

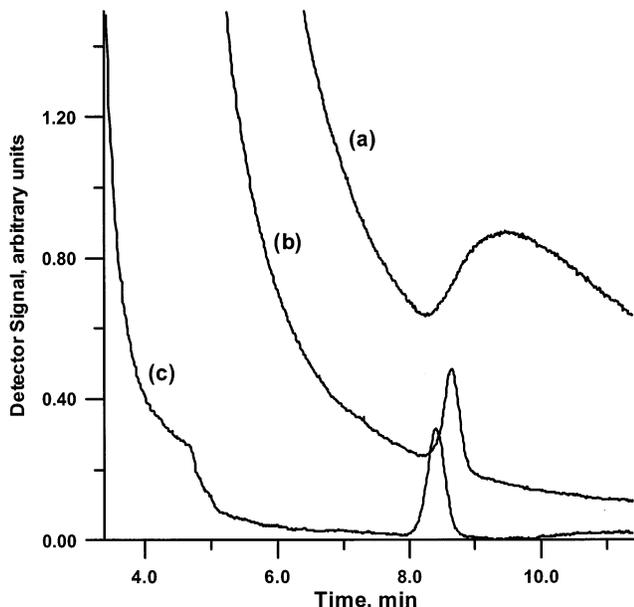


Figure 3. Chromatograms of 2.0 mL Pantex groundwater sample ($\kappa = 855 \mu\text{S}/\text{cm}$) fortified with 20- $\mu\text{g}/\text{L}$ perchlorate: (a) direct loop injection, (b) present system used with 2.0 mL of 15 mM NaOH wash, and (c) same as b, except a wash volume of 4.1 mL.

ferred to the PC, and at high wash volumes, it will be lost out of the PC. However, the maximum permissible wash volume will decrease as the salinity of the sample and loaded amount increases.

Within the permissible wash volume range, using more wash solution will lead to a lower background but will increase the analysis time for each sample. Figure 3 shows the chromatogram of a sample consisting of 20 $\mu\text{g}/\text{L}$ perchlorate spiked into the Pantex groundwater matrix (specific conductance $\kappa = 885 \mu\text{S}/\text{cm}$) sample using (a) standard chromatographic conditions with large loop injection akin to EPA method 314.0, (b) the present method with 2.0 mL of 15 mM NaOH preeluent, and (c) the

present method with 4.1 mL of 15 mM NaOH preeluent. Note that the large peak centered at ~ 10 min in chromatogram a is *not* perchlorate; in fact, the perchlorate peak is not even discernible in this chromatogram. Although both chromatograms b and c will produce acceptable quantitation of the perchlorate peak eluting at 8.5–8.7 min, clearly c is better. For a given sample matrix, the wash volume may thus need to be optimized.

Injection Duration. The sample injection valve V2 must remain in the inject position long enough to inject the perchlorate on the PC fully to the separation system; beyond that, time is wasted and in certain samples may result in components more strongly retained than perchlorate being on the column. In this context, it is very important to note that, although sample preconcentration columns are often used in a mode in which the sample is backflushed into an analytical system, in the present case, the sample loading and elution flow direction upon injection must be the same, as indicated in Figure 1. Figure 4a shows that for a relatively high level of perchlorate loaded on the PC, a minimum injection period of ~ 60 s is needed to fully transfer the analyte to the separation system. Figure 4b demonstrates that essentially the same response is observed for a much lower perchlorate concentration for injection periods of 65–180 s.

Limit of Detection. For low salinity samples, such as many drinking water samples, the use of the preconcentration column allows one to lower the LOD by increasing the volume of the sample injected. For an 8.0 $\mu\text{g}/\text{L}$ perchlorate sample in reagent water injected, the response, peak area (A), was linear from 0.5 to 20 mL of injected sample volume (V , in mL).

$$A = (0.0280 \pm 0.0003) V - (0.0125 \pm 0.0025), \quad r^2 = 0.9985 \quad (1)$$

This indicates that at least 20 mL of standard solution can be concentrated. The attainable LOD at different injection volumes was studied using the following five injection volumes: 2.0, 3.0, 4.0, 8.0, and 18.0 mL. The injected concentration was 1.0 $\mu\text{g}/\text{L}$

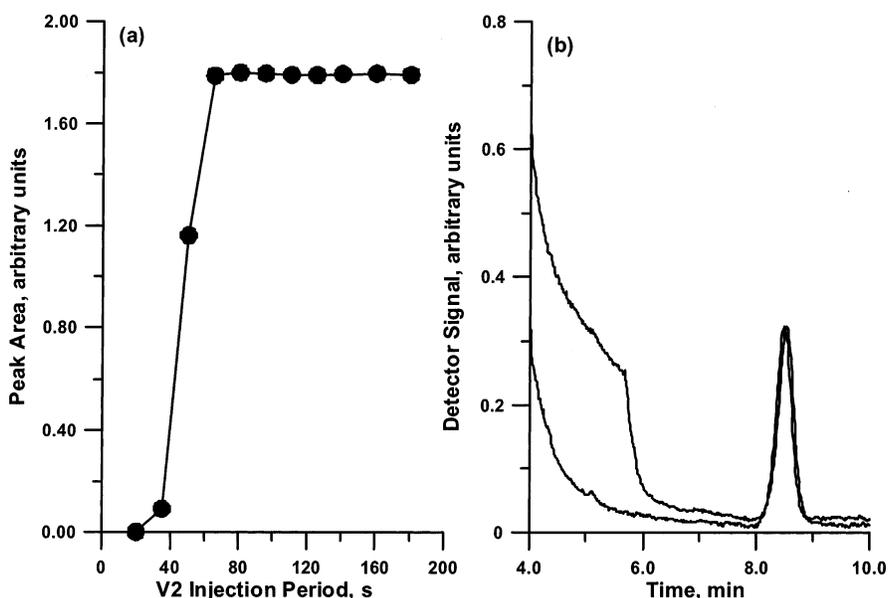


Figure 4. (a) Peak area as a function of the V2 injection period, 2.0 mL of 300 $\mu\text{g}/\text{L}$ perchlorate in Pantex groundwater blank; (b) ion chromatogram of the same sample matrix, except fortified with 20 $\mu\text{g}/\text{L}$ perchlorate using injection periods of 65 and 180 s. A 4.6 mL aliquot of 15 mM NaOH was used as the preeluent in both cases.

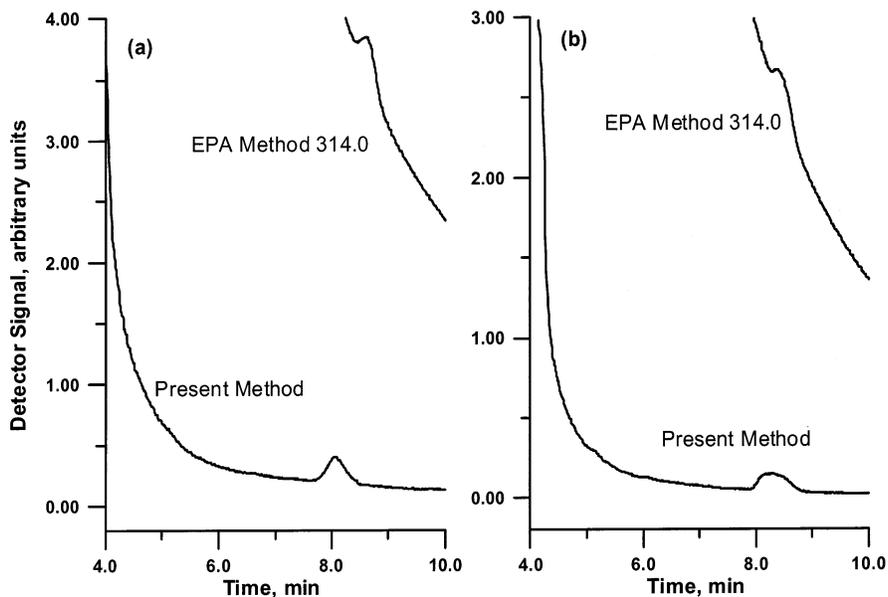


Figure 5. Chromatograms obtained by present and EPA method 314.0 1.0 mL samples: (a) 25 µg/L perchlorate spiked into TCEQ groundwater sample ($\kappa = 4.7$ mS/cm), present method, prewash 2.3 mL of 10 mM NaOH; and (b) 25 µg/L perchlorate spiked into matrix containing 2000 mg/L each of Cl^- , SO_4^{2-} , and CO_3^{2-} ($\kappa = 14.7$ mS/cm), present method, prewash 2.7 mL of 10 mM NaOH.

Table 1. Data for Experiments on Accuracy and Precision Study

amt ClO_4^- added $\mu\text{g/L}$	amt ClO_4^- detected $\mu\text{g/L}$	recovery (%) \pm SD
4.00	3.92 ± 0.147	98.1 ± 3.68
50.0	51.3 ± 0.418	103 ± 0.917
100	104.4 ± 0.112	104 ± 1.12

for the 2.0 mL injection and 0.5 µg/L in all of the other cases, with a minimum of three injections performed. On the basis of a S/N ratio of 3, the computed LODs for the above injection volumes were 0.77, 0.41, 0.32, 0.13, and 0.052 µg/L, respectively. Although performance with a reagent water sample matrix as above cannot establish what can be attained with highly saline matrixes, this gives at least a basis for comparing performance with other published reports, all of which specify LODs based on injection of standards in reagent water.

Linearity of Response. For 2.0-mL injections of 1.0, 4.0, 8.0, 25, 50, 100, 150, 200, 250, 300, 350, and 400 µg/L perchlorate in reagent water (V2 injection duration 90 s, 4.6 mL of 15 mM NaOH preluent), the response could be described by the equation

$$A = (0.00583 \pm 0.00003) [\text{ClO}_4^-, \mu\text{g/L}] - (0.0172 \pm 0.0067),$$

$$r^2 = 0.9997 \quad (2)$$

Precision and Recovery from a Spiked Groundwater Sample. The Pantex groundwater sample was spiked with 4.0, 50, and 100 µg/L perchlorate and quantitated by triplicate injection (2.0 mL volume) on the basis of a calibration curve made from standards in reagent water. The data in Table 1 show recoveries in the range of 98–104% and a precision of 0.9–3.7%.

Performance in High-Salinity Samples. Two high-salinity samples, each containing 25 µg/L perchlorate, were prepared in

Table 2. Recoveries of Present Method vs Method 314.0

sample matrix specific conductivity $\mu\text{S/cm}$	% recovery		% recovery after 4 month column use	
	present method	EPA 314.0	present method	EPA 314.0
<1	100 ^a	100 ^a	100 ^a	100 ^a
1767	98.8	99.4		
3425	99.6	97.3	97.5	93.7
4850	99.0	96.5		
8063	98.8	94.0	95.3	83.5
9451	99.7	93.7		
10850	99.4	92.0	89.0	77.9
12650	96.4	88.9	83.9	73.4
14680	92.0	84.2	77.0	66.5

^a Reference basis.

(a) TCEQ supplied groundwater matrix ($\kappa = 4.7$ mS/cm) and (b) an EPA method 314.0³¹ test matrix containing 2000 mg/L each of SO_4^{2-} , Cl^- , and CO_3^{2-} ($\kappa = 14.7$ mS/cm). Figure 5 shows the experimental results and clearly demonstrates the effectiveness of the new method.

Recovery for High-Salinity Matrixes. A series of solutions containing different levels of SO_4^{2-} , Cl^- , and CO_3^{2-} were made ranging in conductivity from <1 µS/cm to 14.7 mS/cm (see paragraph above) using the process described in EPA method 314.0.³¹ Each solution contained 25 µg/L perchlorate. The samples were analyzed using both the currently prescribed method and EPA method 314.0. The recoveries are listed in Table 2. It is worthwhile to note that 92% recovery was possible, even with the highest conductivity matrix. The relative standard deviations ranged from 0.2 to 1.1%, generally worsening with increasing matrix salinity for both methods. The longevity of the column systems was of interest. Four months later, with the system being routinely used for this type of analysis, the recoveries were measured again. Although the recoveries did decrease under the

same analytical protocol, the recovery was still acceptable (95%) for the presently proposed method with an 8 mS/cm matrix.

In summary, we have developed a simple automated procedure for the determination of low levels of perchlorate in high salinity samples; the method is also equally applicable to drinking water samples.

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