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Improved method for the determination of trace perchlorate in ground and drinking waters by ion chromatography

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Abstract

Ammonium perchlorate, a key ingredient in solid rocket propellants, has been found in ground and surface waters in a number of U.S. states, and perchlorate contamination of public drinking water wells is now a serious problem in California. Perchlorate poses a health risk and preliminary data from the U.S. EPA reports that exposure to less than 4–18 $\mu\text{g}/\text{l}$ provides adequate human health protection. An improved ion chromatographic method was developed for the determination of low $\mu\text{g}/\text{l}$ levels of perchlorate in ground and drinking waters based on a Dionex IonPac AS16 column, an hydroxide eluent generated using an EG40 automated eluent generator, large loop (1000 μl) injection, and suppressed conductivity detection. The method is free of interferences from common inorganic anions, linear over the range of 2–100 $\mu\text{g}/\text{l}$ perchlorate, and quantitative recoveries are obtained for low $\mu\text{g}/\text{l}$ levels of perchlorate in spiked ground and drinking water samples. The MDL of 150 ng/l permits quantification of perchlorate below the levels that ensure adequate health protection. © 2000 Elsevier Science B.V. All rights reserved.

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1. Introduction

Ammonium perchlorate, a key ingredient in solid rocket propellants, has been found in ground and surface waters in a number of states in the U.S., including California, Nevada, Utah, Texas, New York, Maryland, Arkansas, and West Virginia [1,2]. Perchlorate poses a human health concern as it interferes with ability of the thyroid gland to utilize iodine to produce thyroid hormones. Current data from the U.S. Environmental Protection Agency (EPA) indicates that exposure to less than 4–18 $\mu\text{g}/\text{l}$

(ppb) perchlorate provides adequate health protection [3].

Perchlorate contamination of public drinking water wells is now a serious problem in California. The California Department of Health Services (CDHS) has adopted an action level for perchlorate of 18 $\mu\text{g}/\text{l}$ and recommends that utilities remove from service drinking water sources that contain concentrations of perchlorate above the action level. To date, perchlorate has been detected in over 100 public drinking water wells in California, with more than 20 wells now closed due to contamination [3]. In addition, perchlorate has recently been found in sodium nitrate fertilizer derived from Chilean caliche [4].

Perchlorate is listed on the U.S. EPA Contaminant

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Candidate List (CCL) as a research priority under the categories of health, treatment, analytical methods and occurrence priorities [5]. In addition, the EPA has recently revised the Unregulated Contaminant Monitoring Rule (UCMR) and added perchlorate to List 1 for assessment monitoring [6]. Monitoring of List 1 contaminants will occur at 2774 large (serving >10 000 customers) Public Water Systems (PWS) and a representative sample (800 out of 65 600) of small PWS, beginning in January 1, 2001 [7]. The results from this monitoring will be used to estimate the national occurrence of the compounds on List 1 and to evaluate and prioritize contaminants on the CCL. These efforts to establish the extent of perchlorate contamination across the U.S. and possibly set regulatory limits has prompted considerable interest in the development of improved methods for the analysis of low level perchlorate.

Ion chromatography (IC) is now well established as a regulatory tool for the analysis of inorganic anions in drinking water [8]. This relatively straightforward analytical technique is emerging as perhaps the most viable means for the routine quantification of trace level perchlorate, although trace level perchlorate has been determined by electrospray ionization mass spectrometry after ion-pair extraction [9]. The IC analysis of so called 'polarizable' anions, such as perchlorate, is complicated as they are strongly retained on conventional anion-exchange resins and typically display poor peak shape [10]. Consequently, an organic modifier, such as methanol or *p*-cyanophenol, is often added to the mobile phase to minimize adsorption and improve peak shape [11,12]. Based upon this approach, the CDHS developed an IC method in 1997 for the determination of trace perchlorate that utilized a large loop injection with an IonPac AS5 column and a hydroxide eluent containing *p*-cyanophenol [11].

An updated IC method employing an IonPac AS11 column, hydroxide eluent, and suppressed conductivity detection with an ASRS suppressor, was developed in 1998 [13]. The primary advantage of using the more hydrophilic IonPac AS11 column was that *p*-cyanophenol was not required in the eluent. This approach then enabled the use of an electrolytic self regenerating suppressor, which adds considerable convenience to the operation of the ion chromatograph. A recent collaborative study involv-

ing 19 laboratories demonstrated that IC methods based upon the IonPac AS5 and AS11 columns gave similar performance, in terms of linearity, MDLs, freedom from interferences, and spiked recoveries [14]. The study samples consisted of well water at three total dissolved solids levels of 72, 144, and 288 mg/l, which were spiked with perchlorate at concentrations of 6, 18 ppb and 36 µg/l. Both the AS5 and AS11 columns were found to be satisfactory for perchlorate analysis in typical ground and surface waters.

In a previous paper, we discussed the suitability of the AS11 column for the determination of trace perchlorate by IC [15]. In this paper, we will report on the development and application of a new anion-exchange column for the determination of trace level perchlorate using ion chromatography. The IonPac AS16 column is more hydrophilic and has a significantly higher ion-exchange capacity than either the AS5 or AS11 columns. This column allows the injection of higher ionic strength samples and can be used in conjunction with an automated KOH eluent generator, which eliminates the need to manually prepare eluents, increasing the level of automation and ease-of-use of the IC system. The method detection limit, linear concentration range, and potential method interferences will also be discussed.

2. Experimental

2.1. Instrumentation

The ion chromatograph used for this work was a Dionex Corporation (Sunnyvale, CA, USA) DX-500 IC System consisting of a GP50 Gradient Pump, EG40 Eluent Generator, AS40 Automated Sampler, LC30 Chromatography Compartment, and CD20 Conductivity Detector. Separations were performed using Dionex IonPac AS11 and AS16 (250×4.0 mm) analytical columns and IonPac AG11 and AG16 (50×4.0 mm) guard columns. Anions were detected by suppressed conductivity detection; suppression was achieved using a Dionex ASRS[®]-ULTRA, operated at 300 mA in the external water mode.

2.2. Reagents and procedures

All solutions were prepared from ACS reagent-grade chemicals (where possible) in 18 M Ω water, obtained from a Water Pro PS purification system (Labconco, Kansas City, MO). Sodium hydroxide, 50% w/w aqueous solution, was obtained from Fisher Scientific (Pittsburgh, PA). Sodium perchlorate standard, 99% ACS reagent-grade, was obtained from Aldrich (Milwaukee, WI). ACS reagent grade sodium salts, obtained from Fisher Scientific, Aldrich, Sigma (St. Louis, MO), EM Science (Gibbstown, NJ) and Fluka Chemicals (Ronkonkoma, NY), were used for the preparation of solutions for the interference study; with the exception of technical grade humic acid from Aldrich and ACS reagent-grade potassium cyanide from Sigma.

The manually prepared 35 sodium hydroxide eluent was prepared by weighing 997.2 g of deionized water into a 1.0 l polypropylene eluent bottle. The water was then degassed for 5 min, after which 2.80 g of 50% sodium hydroxide was carefully added directly to the eluent bottle. The solution was mixed and the bottle quickly transferred to the instrument and pressurized with helium (8 p.s.i.). Such precautions are necessary when manually preparing the hydroxide eluents in order to avoid carbonate contamination [16,17].

3. Results and discussions

3.1. Column characteristics

The IonPac AS16 analytical and AG16 guard column set used for this work were developed for the routine determination of polarizable anions by IC. These 'hydrophobic' anions, which include solutes such as thiosulfate, iodide, thiocyanate, and perchlorate, have an easily deformed electron cloud, low hydration energy, and usually a small hydrated radius [10]. They are generally difficult to analyze as they are strongly retained, hence difficult to elute from conventional anion-exchange resins with a reasonable peak shape. The addition of an organic modifier, such as methanol or *p*-cyanophenol, to the mobile phase can minimize adsorption and improve peak shape, although this increases eluent and dis-

posal costs and also reduces the sensitivity of suppressed conductivity detection for this application [15].

The packing material for the AS16 column was optimized in terms of capacity, hydrophobicity, and selectivity to provide improved performance for the analysis of polarizable anions, and perchlorate, in particular. In order to achieve sufficient sensitivity for the analysis of low $\mu\text{g/l}$ levels of perchlorate, a large loop injection ($\sim 1000 \mu\text{l}$) is necessary. This, in turn, requires the use of a modest to high capacity column to avoid overloading (hence peak distortion) when injecting drinking or ground water samples. The stationary phases typically used in suppressed IC are low capacity, pellicular resins. A monolayer of fully functionalized latex particles are electrostatically attached to a surface functionalized, non-porous core particle [10]. The ion-exchange capacity of the resin can be increased by using a larger diameter latex, although this approach ultimately results in band broadening and decreased efficiency when producing relatively high capacity columns [18].

To overcome this problem, the AS16 column uses a macroporous (2000 Å pore size) resin consisting of ethylvinylbenzene crosslinked with 55% divinylbenzene. This core particle allows the latex layer to be thinly coated on both the exterior and interior surfaces of the resin and provides a simple way to increase the capacity of the resin while using a standard diameter (80 nm) latex. The anion-exchange latex layer used for the AS16 packing material was functionalized with very hydrophilic (1% cross-linking) alkanol quaternary ammonium groups. The latex layer has a controlled thickness, resulting in excellent mass transfer characteristics and consequently high chromatographic efficiency [19]. The net result of this development is a column that (a) has relatively high capacity, (b) is compatible with conventional HPLC solvents, (c) is very hydrophilic with a high selectivity toward hydroxide eluents, and (d) provides high chromatographic efficiency and excellent peak shape for polarizable anions, and other well retained solutes, such as polyphosphates and polythionates. Fig. 1 shows a comparison of the IonPac AS11 column, which was previously recommended for the analysis of polarizable anions [16], and the recently developed AS16 column for the separation of thiosulfate, iodide, thiocyanate, and perchlorate, in

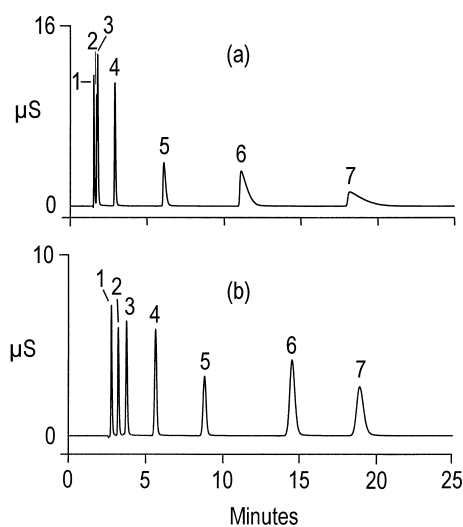


Fig. 1. Separation of common inorganic anions and polarizable anions obtained using AS11 and AS16 columns. Conditions: (a) guard column, IonPac AG11; analytical column, IonPac AS11; (b) guard column, IonPac AG16; analytical column, IonPac AS16; eluent, 35 mM sodium hydroxide; flow-rate, 1.0 ml/min; detection, suppressed conductivity with ASRS-ULTRA operated at 300 mA in recycle mode; injection volume, 10 μ l; solutes, 1=fluoride (2 mg/l), 2=chloride (3 mg/l), 3=sulfate (5 mg/l), 4=thiosulfate (10 mg/l), 5=iodide (20 mg/l), 6=thiocyanate (20 mg/l), 7=perchlorate (30 mg/l).

addition to common inorganic anions, fluoride, chloride, and sulfate.

The AS16 has a significantly higher capacity than the AS11 column (170 vs. 45 μ equiv./column), hence common inorganic anions, such as chloride and sulfate, show more retention on the higher capacity column, as would be expected. The polarizable anions exhibit similar retention times on both columns as a result of the fact that the very hydrophilic AS16 resin minimizes any adsorption interactions with the stationary phase. Consequently, the AS16 column (b) exhibits superior peak shape for polarizable anions, e.g. peaks 5, 6, and 7, compared to the AS11 column (a), which is a relatively hydrophilic column itself. In addition, the average number of hydroxyl groups incorporated into the alkanol functionality is higher for the AS16 column, which produces a stationary phase with a greater selectivity toward hydroxide eluents. These two effects produce a column that is ideal for the analysis of low level perchlorate by IC, in that good peak

shape can be achieved within a reasonable timeframe using a modest ionic strength hydroxide eluent.

3.2. Automated eluent generation

The fact that perchlorate can be eluted using hydroxide eluents in the 30–70 mM concentration range on the AS16 column makes this application suitable for use with the EG40 Eluent Generator, a device that automates the production of high purity hydroxide eluents. Great care must be taken when manually preparing hydroxide eluents to minimize contamination by atmospheric carbon dioxide, which results in elevated levels of carbonate in the eluent. The contamination problem is most evident when performing gradient elution with hydroxide eluents, as the presence of carbonate causes a significant baseline shift during the gradient [20]. The presence of carbonate contamination can also cause variation in analyte retention times, particularly for weakly retained anions, and increased (suppressed) background conductance, even when using hydroxide eluents with isocratic elution. Consequently, hydroxide eluents are best prepared from fresh 50% (w/w) sodium hydroxide aqueous solution, rather than pellets. The hydroxide should be weighed and quickly transferred into an eluent bottle containing an appropriate volume of degassed water, which should then be pressurized with helium [16,17].

These problems can be minimized by using an electrolytic eluent generation device which automates the production of high purity hydroxide eluents [20]. An eluent of potassium hydroxide is produced in the EG40 Eluent Generator by pumping deionized water through an electrolysis chamber and applying a DC current between the cathode (in the chamber) and anode (in an electrolyte reservoir cartridge). Under the applied field, electrolysis of water occurs and hydronium ions generated at the anode displace electrolyte potassium ions across a cation-exchange membrane. The potassium ions combine with hydroxide ions generated at the cathode to produce a KOH solution, with the concentration being directly proportional to the applied current. The typical cartridge lifetime would be around 2000 h when using eluent conditions similar to those described in Fig. 1 [21]. For this application, where a relatively high ionic strength eluent is used

with isocratic elution, the EG40 offers equivalent performance to a carefully prepared, manually produced hydroxide eluent. However, the use of the EG40 simplifies the method by eliminating the need to manually prepare eluents, hence increasing the level of automation of the IC system.

3.3. Method performance

Fig. 2 shows a typical chromatogram of a 20 $\mu\text{g/l}$ perchlorate standard obtained using the AS16 column with a 1000 μl injection loop, a 65 mM hydroxide eluent operated at a flow-rate of 1.2 ml/min, and suppressed conductivity detection. In order to maximize sensitivity for this application, it was necessary to use both a large injection loop and also operate the anion self-regenerating suppressor (ASRS-ULTRA) at 300 mA in the external water mode. Under these conditions, perchlorate elutes within 10 min while the common inorganic anions all elute at (or near) the column void volume. An eluent of 50 mM hydroxide operated at a flow-rate of 1.5 ml/min, as recommended for use with the AS16 column in U.S. EPA Method 314.0, results in an almost identical retention time for perchlorate [17]. However, the eluent conditions which used the lower flow-rate gave slightly greater peak area response for perchlorate, resulting in lower method detection limits (MDLs). The method linearity was determined to ensure accurate quantification of perchlorate in the

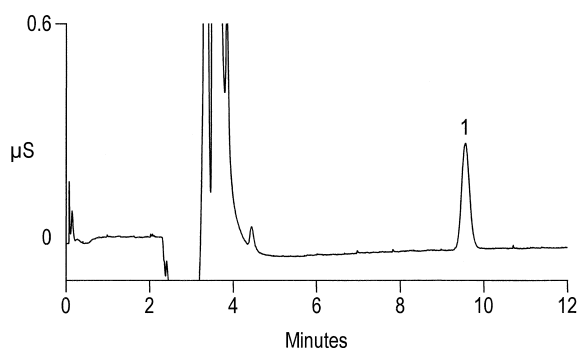


Fig. 2. Perchlorate standard at 20 $\mu\text{g/l}$. Conditions: guard column, Dionex IonPac AG16; analytical column, Dionex IonPac AS16; eluent, 65 mM potassium hydroxide; eluent source, EG40; flow-rate, 1.2 ml/min; detection, suppressed conductivity with ASRS-ULTRA operated at 300 mA in external water mode; injection volume, 1000 μl ; peaks, 1=perchlorate (20 $\mu\text{g/l}$).

low $\mu\text{g/l}$ range. A correlation coefficient of $r^2=0.9999$ was obtained for a plot of peak area vs. concentration over the range of 2 to 100 $\mu\text{g/l}$. The MDL was determined using the procedure outlined in U.S. EPA Method 300.0 [8]. Seven replicates of a 2.0 $\mu\text{g/l}$ perchlorate standard were injected and the MDL was calculated to be 150 ng/l.

Despite the use of a large loop injection, no appreciable evidence of column overloading, in terms of reduced retention times or distorted peak shapes, was observed when analyzing typical ground and drinking water samples on the AS16 column. Fig. 3 shows a chromatogram of Sunnyvale, CA tap water spiked with 4.0 $\mu\text{g/l}$ perchlorate. The method was tested for interferences by injecting 100 $\mu\text{g/l}$ solutions of the 22 solutes listed in Table 1. These data show that only iodide and thiocyanate exhibit any significant retention under these elution conditions. Perchlorate is resolved by approximately 2 min from the nearest eluting anion, thiocyanate, which would not be typically found at high levels in ground or drinking waters.

Ground water samples may contain high concentrations of common anions, particularly carbonate, chloride, and sulfate. The effect of elevated levels of these common anions on perchlorate recovery when using the AS16 column was investigated by injecting solutions of 20 $\mu\text{g/l}$ perchlorate in the presence of 50, 200, 600, and 1000 mg/l of carbonate, chloride, or sulfate. Quantitative recoveries (80–120%) were obtained for perchlorate in all cases, as shown in Table 2. Fig. 4 shows an overlay of the chromatograms of 20 $\mu\text{g/l}$ perchlorate

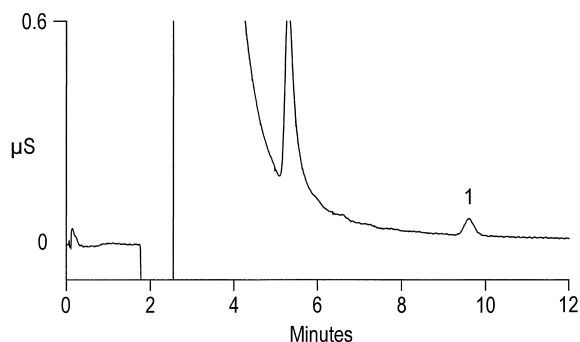


Fig. 3. Sunnyvale tap water spiked with 4.0 $\mu\text{g/l}$ perchlorate. Conditions: as for Fig. 2 except; peaks, 1=perchlorate (3.7 $\mu\text{g/l}$).

Table 1
Comparison of the retention times of 22 anions and perchlorate on the IonPac AS16 column^a

Anion	Anion retention time (min)	Perchlorate retention time (min)
Arsenate	<4	9.78
Arsenite	<4	9.75
Bromate	<4	9.72
Bromide	<4	9.73
Carbonate	<4	9.72
Chlorate	<4	9.72
Chloride	<4	9.68
Chromate	<4	9.68
Cyanide	<4	9.65
Humic acid	<4	9.67
Iodate	<4	9.65
Iodide	5.28	9.65
Molybdate	<4	9.63
Nitrate	<4	9.65
Nitrite	<4	9.63
Phosphate	<4	9.63
Phthalate	<4	9.62
Selenate	<4	9.60
Sulfate	<4	9.60
Sulfite	<4	9.60
Thiocyanate	7.72	9.60
Thiosulfate	<4	9.58

^a An eluent of 50 mM hydroxide at 1.5 ml/min was used for this study.

Table 2
Effect of ppm levels of common anions on perchlorate recovery (20 µg/l) on the IonPac AS16 column^a

Anion	Anion concentration (mg/l)	Perchlorate recovery
Carbonate	50	97.6%
Carbonate	200	94.4%
Carbonate	600	95.4%
Carbonate	1000	93.5%
Chloride	50	96.1%
Chloride	200	96.7%
Chloride	600	109.6%
Chloride	1000	97.4%
Sulfate	50	94.4%
Sulfate	200	96.3%
Sulfate	600	94.7%
Sulfate	1000	95.5%

^a An eluent of 50 mM hydroxide at 1.5 ml/min was used for this study.

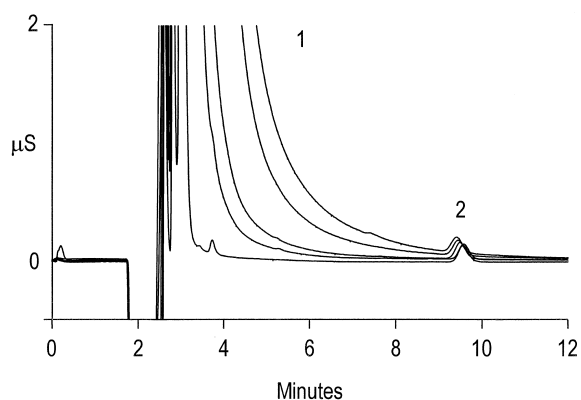


Fig. 4. Perchlorate in the presence of 0, 50, 200, 600 and 1000 mg/l of sulfate. Conditions: as for Fig. 2 except; eluent, 50 mM potassium hydroxide; flow-rate, 1.5 ml/min; peaks, 1=sulfate at 0, 50, 200, 600 and 1000 mg/l, 2=perchlorate (20 µg/l).

in the presence of 0–1000 mg/l sulfate. The standard perchlorate retention time decreased slightly (from 9.55 min to 9.40 min) in the presence of 1000 mg/l sulfate, peak efficiency also decreased slightly (from 6997 theoretical plates (USP) to 6310 theoretical plates) in the presence of 1000 mg/l sulfate, while the peak gaussian factor was unchanged in the presence of 1000 mg/l sulfate. These modest changes in retention time and peak efficiency in the presence of up to 1000 mg/l sulfate did not affect the identification or integration of the perchlorate peak. Similar plots were obtained for perchlorate in the presence of 50–1000 mg/l carbonate or chloride.

In addition to ground waters, the analysis of perchlorate in other high ionic strength matrices is also important, e.g. fertilizers, fermentation media from bioreactors, and brines (produced from the regeneration of ion-exchange cartridges used in water treatment). The utility of the AS16 column for such samples was demonstrated by analyzing a brine with a chloride concentration of 10 000 mg/l that had been spiked with perchlorate at a concentration of 100 µg/l. One commonly used approach for determining anionic solutes in extreme chloride matrices is to pretreat the sample with an SPE cartridge packed with cation-exchange resin in the Ag⁺ form, which removes chloride from the sample by precipitating it as silver chloride [22]. Fig. 5(b) shows the analysis of the spiked brine sample after treatment with an Ag⁺ form, cation-exchange car-

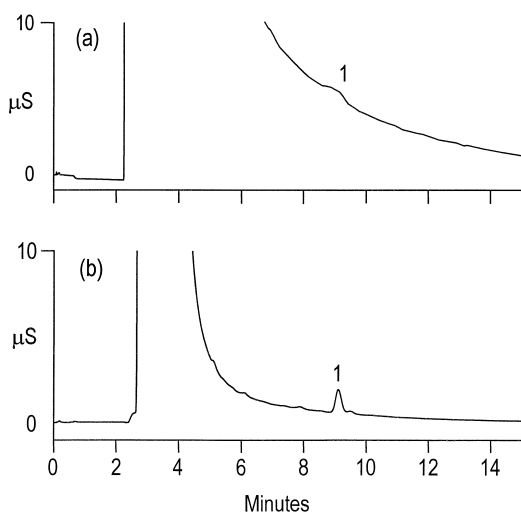


Fig. 5. Effect of silver form SPE cartridge on perchlorate recovery from spiked brine. Conditions: as for Fig. 2 except; (a) sample, synthetic brine containing 10 000 mg/l chloride spiked with 100 $\mu\text{g/l}$ perchlorate; (b) sample, synthetic brine after pretreatment with OnGuardAg SPE cartridge; peaks, 1= perchlorate (92.6 $\mu\text{g/l}$).

tridge. This approach removes approximately 90% of the sample chloride while maintaining good recovery (93%) for the perchlorate anion. A chromatogram of the untreated brine is shown in Fig. 5(a).

A more convenient approach for determining perchlorate in such a sample is to dilute the sample and/or reduce the eluent concentration. The same brine containing chloride at 10 000 mg/l and perchlorate at 100 $\mu\text{g/l}$ was diluted 5-fold and the diluted sample was then analyzed using two different eluent strengths, 35 mM and 65 mM potassium hydroxide. Fig. 6(a) shows that with the higher concentration eluent, perchlorate is somewhat difficult to quantify because it elutes on the trailing edge of the large (now 2000 mg/l) chloride peak. The use of a weaker eluent improves the resolution of perchlorate from the large chloride peak, as shown in Fig. 6(b). This allows more accurate integration (hence quantitation) of the target anion, resulting in a recovery of 88%. Using these conditions, perchlorate at concentrations as low as 20 $\mu\text{g/l}$ could be analyzed in a sample containing up to 30 000 mg/l of chloride, after a 10-fold dilution. Because the AS16 column can tolerate high ionic strength matrices, the U.S. EPA has now specified this column

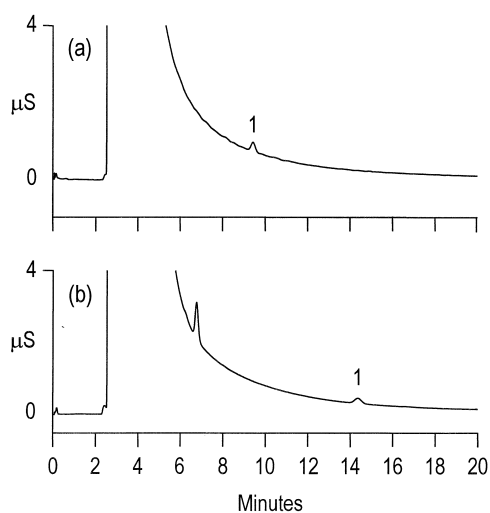


Fig. 6. Effect of sample and eluent dilution on perchlorate recovery from spiked brine. Conditions: as for Fig. 2 except; (a) sample, synthetic brine containing 10 000 mg/l chloride spiked with 100 $\mu\text{g/l}$ perchlorate after 5 \times dilution; peaks, 1 - perchlorate (17.7 $\mu\text{g/l}$); (b) eluent, 35 mM potassium hydroxide; sample, synthetic brine after 5 \times dilution; peaks, 1=perchlorate (17.6 $\mu\text{g/l}$).

for perchlorate analysis in surface, ground, and finished drinking waters, according to EPA Method 314.0 [17]. This method has been specified for the assessment monitoring of perchlorate in drinking water at large Public Water Systems, as required by the recent changes to the Unregulated Contaminant Monitoring Rule [7].

4. Conclusions

The use of ion chromatography with a hydrophilic IonPac AS16 column, EG40 automated potassium hydroxide eluent generator, large loop injection and suppressed conductivity detection provides a simple, interference-free method for the determination of trace level perchlorate in ground and drinking waters. The method is linear over the range of 2–100 $\mu\text{g/l}$ perchlorate and quantitative recoveries are obtained for low $\mu\text{g/l}$ levels of perchlorate in spiked drinking and ground water samples. The MDL of 150 ng/l permits quantification of perchlorate below the levels that ensure adequate health protection, as recommended by the U.S. EPA. The AS16 column

provides improved performance for the analysis of high ionic strength waters compared to columns previously recommended for the determination of trace level perchlorate, such as the IonPac AS5 or AS11 columns. The AS16 is the column specified for perchlorate analysis by ion chromatography in surface, ground and finished drinking waters in U.S. EPA Method 314.0, which is the analytical method designated for monitoring perchlorate at large Public Water Systems, as required by recent revisions to the Unregulated Contaminant Monitoring Rule.

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