METHOD STATUS: APPROVED FOR ROUTINE USE **METHOD REVISION NO. : 0**

Public Health Chemist Environmental Biochemist

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DETERMINATION OF PERCHLORATE BY ION CHROMATOGRAPHY

1. Scope and Application

1.1 This method covers the determination of the perchlorate anion by ion chromatography.

1.2 The applicable matrices are shown below.

1.1.1 Drinking water, groundwater and reagent waters: This method has been found to perform adequately on water samples with conductivities up to 1000 μmhos/cm. Water samples with conductivities >1000 μmhos/cm have not been tested.

1.2.2 Although not specifically tested, this method is potentially applicable to surface water, mixed domestic water, and industrial wastewaters. See also Sect. 1.7.

1.3 The Method Detection Limit (MDL defined in Sect. 3.10) and Reporting Limit (RL defined in Sect. 3.12) for perchlorate in reagent water is 0.7 μg/L (pooled data) and 4.0 μg/L, respectively. See Table 1. The MDL and RL for a specific matrix may differ from that listed, depending upon the nature of the sample.

1.4 The linear calibration range for perchlorate is approximately 2.5 to 500 μg/L. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to a concentration within the calibration range and reanalyzed.

1.5 Figure 1 shows the chromatograms for 4 μg/L of perchlorate added to reagent water and a groundwater sample.

1.6 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

1.7 When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of a fortified sample matrix. The fortification procedure is described in Sect. 11.6.

1.8 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results with this method, using the procedures described in Sect. 9.

1.9 **DISCLAIMER** Mention of trade names or commercial products does not constitute endorsement or recommendation for use. Equivalent product substitutions may be made by laboratories using this method as a reference.

2. Summary of Method

2.1 A fixed volume of sample is injected into an ion chromatographic system, where the perchlorate anion is separated from other interfering anions and quantified.

1.2 To detect perchlorate in the low ppb (µg/L) range without sample preconcentration, a high volume sample loop is used.

2.3 To minimize hydrophobic interaction of the perchlorate anion with the anion exchange support resin, p-cyanophenol is added to the eluent to deactivate the active sites on the resin. Without column deactivation, the perchlorate peak elutes with a longer retention time, is broader (isocratic elution), and tails severely, thus resulting in poor peak detection as the perchlorate sample concentration decreases.

3. Definitions

3.1 CALIBRATION BLANK (CB) -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

3.2 CALIBRATION STANDARD (CAL) -- A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.3 FIELD DUPLICATES (FD) -- Two separate samples collected at the same time and location under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.4 INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.5 LABORATORY FORTIFIED BLANK (LFB) -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the method is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.6 LABORATORY FORTIFIED SAMPLE MATRIX (LFM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.7 LABORATORY REAGENT BLANK (LRB) -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.8 LINEAR CALIBRATION RANGE (LCR) -- The concentration range over which the instrument response is linear.

3.9 MATERIAL SAFETY DATA SHEET (MSDS) -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.10 METHOD DETECTION LIMIT (MDL) -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. (See Ref. 16.3.)

3.11 QUALITY CONTROL SAMPLE (QCS) -- A solution of method analyte(s) of known concentration(s) that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.12 REPORTING LIMIT (RL) -- The reporting limit used in this method is defined as the minimum quantifiable concentration level at which a sample concentration result may be reported and is equal to five times the MDL.

4. Interferences

4.1 Interferences can be caused by substances with retention times that are similar to and overlap the anion of interest. High concentrations of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2 The large water dip or negative peak is due to the large aliquot of sample injected onto the column. However, the perchlorate anion is retained for a sufficient length of time in the column and elutes free of interference from the water dip.

4.3 Due to the strength of the eluent, the majority of the anions in a water sample will elute soon after the water dip. Because of the large sample volume injected, the detector response from these anions may be very high, depending on the amount of dissolved solids present in the sample. With the longer retention time, the perchlorate anion elutes on the tail end of these early eluting anions and therefore, the detection and quantification of perchlorate is largely unaffected. See Figure 1.

4.4 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.5 Samples that contain particles larger than 0.45 microns and reagents solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

5. Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

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5.3 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.

5.3.1 Sodium hydroxide (Sect. 7.2).

5.3.2 Sulfuric acid (Sect. 7.3).

5.3.3 Potassium perchlorate (Sect. 7.4).

6. Equipment and Supplies

1.1 Balance -- Analytical, capable of accurately weighing to the nearest 0.1 mg.

6.2 Ion chromatograph -- Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed gasses and detectors.

6.3 Sample loop: approximately 740 µL (12' x 0.02" I.D. tubing).

6.4 Anion guard column: Dionex IonPac AG5 (P/N 35396), or equivalent.

6.5 Anion separator column: Dionex IonPac AS5 (P/N 35395), or equivalent. This column produces the separation shown in Figure 1.

6.6 Anion suppressor device: Dionex AMMS-II (P/N 43074) suppressor system, or equivalent.

6.7 Conductivity detector -- Dionex CDM-II, or equivalent.

6.8 Chromatography data system: The data presented in this method were generated using the Dionex ACI-I computer interface and the Dionex AI-450 Data Chromatography Software. An equivalent data collection and chromatography processing system may also be used.

6.9 Sample bottles: polyethylene, 125 mL, or larger.

7. Reagents and Standards

7.1 Reagent water: Distilled or deionized water, free of the anion of interest. The reagent water should contain particles no larger than 0.20 µm.

7.2 Eluent solution: 50% (w/w) Sodium hydroxide (CASRN 1310-73-2) 120 mM, p-cyanophenol (CASRN 767-00-0) 2.0 mM. Dissolve 19.20 g of 50% (w/w) sodium hydroxide (NaOH) and 0.4765 g of p-cyanophenol (NCC_6H_4OH, 95%, Aldrich P/N C9,400-9, or equivalent) in degassed reagent water and

dilute to 2 L. The 50% (w/w) NaOH should be fresh with minimal contamination from dissolved CO_2 (carbonate formation).

7.3 Regenerant solution (micro-membrane suppressor): Sulfuric acid (CASRN 7664-93-9) 0.035N. Dilute 3.9 mL reagent grade conc. sulfuric acid (H_2SO_4) to 4 L with reagent water.

7.4 Stock standard perchlorate solutions, 1000 mg/L (1 mg/mL): The stock standard solution is prepared from ACS reagent grade material. Dissolve 1.3931 g potassium perchlorate ($KClO_4$, CASRN 7778-74-7) in reagent water and dilute to 1 L.

7.4.1 Prepare a 1000 mg/L perchlorate ($KClO_4$) stock solution for use in preparing the instrument calibration solutions and IPC solutions.

7.4.2 Prepare a 1000 mg/L perchlorate stock solution using a material source different from that of the calibration stock for use in preparing the QCS, LFB and LFM. The QCS is used to verify the accuracy of the instrument calibration.

7.4.3 The analyst should be aware of the purity of the potassium perchlorate used to prepare the stock standard. A weight correction must be made when the solid material is less than 99% pure.

7.5 Intermediate stock standard perchlorate solutions. Prepare 10, 1.0 and 0.10 mg/L standard solutions from the stock standard solutions.

NOTE: Stability of standards: The stock standard is stable for at least one month when stored at 4°C. The intermediate stock and dilute working standards should be prepared weekly.

8. Sample Collection, Preservation and Storage

1.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.2 Sample preservation and holding time for perchlorate determined by this method are as follows:

Analyte	Preservation	Holding Time
Perchlorate	Store at 4°C	28 days *

* Note: Based on the stability of chlorate. Under the same conditions, the stability of perchlorate is expected to be equivalent to, or more stable than, chlorate.

9. Quality Control

1.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

9.2 Initial Demonstration of Performance

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDL) prior to performing analyses by this method.

9.2.2 Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.2.3 Quality Control Sample (QCS) -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ± 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

9.2.4 Method Detection Limit (MDL) -- The MDL must be established for the analyte, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit (Ref. 16.3). To determine the MDL value, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report

the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

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].

S = standard deviation of the replicate analyses.

MDLs should be determined every year, when a new operator begins work or whenever there is a significant change in the background or instrument response.

9.3 Assessing Laboratory Performance

9.3.1 Laboratory Reagent Blank (LRB) -- The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate that laboratory or reagent contamination should be suspected and corrective action must be taken before continuing the analysis.

9.3.2 Laboratory Fortified Blank (LFB) -- The laboratory must analyze at least one LFB with each batch of samples. Calculate accuracy as percent recovery (Sect. 9.4.1.2). If the recovery of perchlorate falls outside the required control limits of 90-110%, perchlorate is judged out of control, and the source of the problem should be identified and resolved before continuing with the analysis.

9.3.2.1 The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = x + 3S$$
$$\text{LOWER CONTROL LIMIT} = x - 3S$$

9.3.2.2 The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to ten new recovery measurements, new control limits can be

calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations included in the LFB. These data must be kept on file and be available for review.

9.3.2.3 Replicates of LFBs should be analyzed quarterly, or sooner, to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality.

9.3.3 Instrument Performance Check Solution (IPC) -- For all determinations the laboratory must analyze the IPC (a midrange check standard) and a calibration blank immediately following daily calibration, after every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within ± 10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ± 10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

9.4 Assessing Analyte Recovery and Data Quality

9.4.1 Laboratory Fortified Sample Matrix (LFM) -- The laboratory must perform a matrix spike on a minimum of 10% of the routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The spiked perchlorate concentration must be high enough to be detected above the original sample concentration and should not be less than five times the MDL. The added perchlorate concentration should be the same as that used in the LFB.

9.4.1.1 In a blind matrix spike, if the concentration of fortification is less than 25% of the background concentration of the matrix the matrix recovery should not be calculated.

9.4.1.2 Calculate the percent recovery for perchlorate, corrected for the concentration measured in the unfortified sample, and compare the value to the initial LFM recovery range of 75-

125%. Percent recovery may be calculated using the following equation:

$$R = \frac{C_A - C}{A} \times 100\%$$

where,
R = percent recovery.
C_A = fortified sample concentration.
C = sample background concentration.
A = concentration equivalent of analyte added to sample.

9.4.1.3 When sufficient internal performance data becomes available (a minimum of 20 analyses) develop control limits from percent mean recovery (X) and the standard deviation (S) of the mean recovery, as in Sect. 9.3.2.1.

9.4.1.4 If the recovery of the analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Sect. 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.

9.4.2 Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

9.4.3 In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of an anion concentrator column, different columns and/or eluents, to improve the separation, quantification, or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in Sect. 9.

9.4.4 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak in the chromatogram, confirmatory techniques such as sample dilution and fortification, must be used. Whenever possible, the laboratory should perform analysis of quality control check samples and participate in relevant performance evaluation sample studies.

10. Calibration and Standardization

10.1 Establish ion chromatographic operating parameters equivalent to those indicated in Table 1.

10.2 Prepare calibration standards at a minimum of five concentration levels and a blank by adding accurately measured volumes of one or more intermediate stock standards (Sect. 7.5) to a volumetric flask and diluting to volume with reagent water. Perform a full instrument calibration on a monthly basis, or whenever a significant change in instrument response is observed or expected.

10.2.1 During this procedure, the perchlorate retention time must be recorded.

10.2.2 To confirm the linearity of the calibration curve, the predicted concentration for each calibration standard should be calculated by using the established linear regression curve and response from each standard concentration. If the predicted response for any standard varies from the expected response by more than ±10%, perform corrective action.

10.3 The calibration curve must be verified by analyzing the IPC solutions on each working day, or whenever the anion eluent is changed, and after every 20 samples. If the response or retention time for perchlorate varies from the expected values by more than ± 10%, the test must be repeated, using fresh IPC solutions. If the results are still more than ± 10%, a new calibration curve must be prepared.

10.4 Nonlinear response can result when the analytical column capacity is exceeded (overloading). The response of the detector to the sample when diluted 1:1, and when not diluted, should be compared. If the calculated responses are the same, the sample need not be diluted.

11. Procedure

11.1 Table 1 summarizes the recommended operating conditions for the ion chromatograph. Included in the table is the estimated retention time that can be achieved by this method.

11.2 Check the system calibration daily and, if required, recalibrate as described in Sect. 10.

11.3 Analyze the IPCs, QCS, LRB, samples, LFB, LFMs, and blanks.

11.4 The width of the retention time window used to make the perchlorate identification should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.5 If a sample concentration exceeds the calibration range, the sample must be diluted with reagent water to fall within the working range.

11.6 If the resulting chromatogram fails to produce adequate resolution, or if identification of the specific anion is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

NOTE: Retention time is inversely proportional to concentration. In some cases this peak migration may produce poor resolution or identification.

12. Data Analysis and Calculations

12.1 Peak integration may be performed using either the peak height or the peak area method. However, the method of peak height is frequently preferable to the method of peak area, as the peak height determination is generally less affected by baseline placement as compared to the peak area determination.

12.2 Examine the chromatograms for perchlorate baselines set by the parameters used in the chromatography method. Correct any baseline improperly set by the method by modifying the integration parameters in the method. Save the corrected baseline to the raw data file.

12.3 As a check on system performance, the response for a low concentration standard (e.g. 4 μg/L perchlorate standard solution) should be monitored and recorded. If the daily detector response is more than three standard deviations lower than the recorded mean response, perform corrective action.

12.4 Prepare the calibration curve by plotting the instrument response against the standard concentration. Compute the sample concentration (corrected for any sample dilution) by comparing the sample response with the standard curve.

12.5 Report only those values that fall between the RL (Table 1) and the highest calibration standard. Samples exceeding the highest standard should be diluted and reanalyzed.

12.6 Report the perchlorate results in μg/L.

13. Method Performance

13.1 Table 1 gives the single laboratory MDL under the conditions listed.

13.2 Tables 2 and 3 give the single laboratory accuracy and precision for perchlorate in reagent water and in groundwater for the listed conditions.

1.3 Table 4 gives the single laboratory precision for replicate analyses of perchlorate in groundwater samples.

14. Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15. Waste Management

15.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Sect. 14.3.

16. References

16.1 Record 269, Dionex Chromatography Database 4.2.0, Dionex Corp., Sunnyvale, CA, 94086.

16.2 Method 300.0, Revision 2.1, “Determination of Inorganic Anions by Ion Chromatography,” August 1993, Environmental Monitoring Systems Laboratory, Office of Research and Development, USEPA, Cincinnati, OH, 45268.

16.3 Code of Federal Regulations 40, Ch. 1, Part 136, Appendix B.

17. Acknowledgement

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FIGURE 1. CHROMATOGRAMS OF 4 µg/L PERCHLORATE ADDED TO REAGENT WATER AND TO A GROUNDWATER SAMPLE

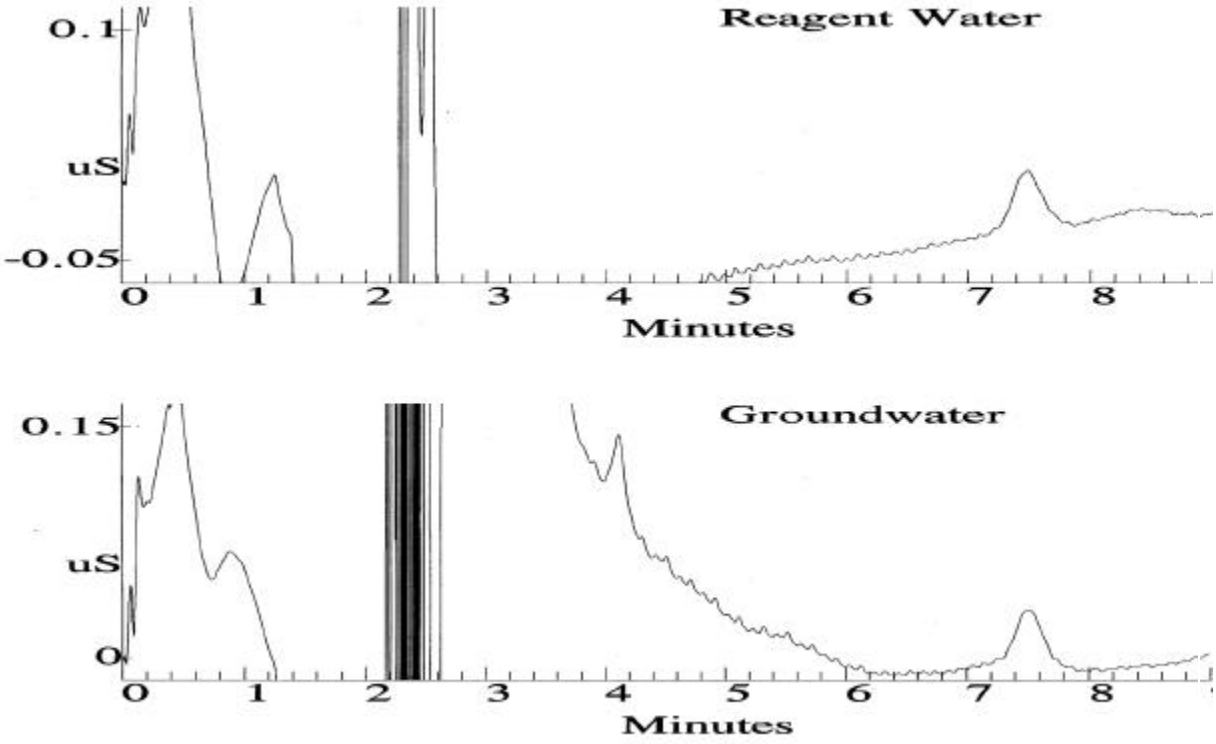
Reagent Water
0.1
uS
-0.05
0
1
2
3
4
5
6
7
8
Minutes
Groundwater
0.15
uS
0
0
1
2
3
4
5
6
7
8
Minutes

TABLE 1. CHROMATOGRAPHIC CONDITIONS AND DETECTION LIMIT IN REAGENT WATER

Perchlorate Spike Conc. (µg/L)	No. of Spiked Replicates	Mean Perchlorate Recovery (µg/L)	Standard Deviation (µg/L)	Calculated MDL (µg/L)
1.0	14	0.87	0.24	0.6
2.5	16	2.3	0.32	0.8
4.0	16	3.9	0.26	0.7

Pooled MDL (*df* = 43)	**0.7 µg/L**
RL (5 x MDL)	**4 µg/L**
Retention Time	**~ 7.4 min.**

Perchlorate peak height response for 4.0 µg/L ≈ 0.04 µS

Equipment and Standard Conditions Used to Produce Data in this Method:

Dionex 4500 Ion Chromatograph with Autosampler

Detector: Dionex CDM-2

Ion Suppressor: Dionex AMMS-II

Columns: Dionex IonPac AG5 Guard column (P/N 35396)

Dionex IonPac AS5 Analytical (P/N 35395)

Column Temperature: Ambient

Injector Loop: 740 µL (approximate volume)

Eluent: 120 mM NaOH + 2.0 mM p-Cyanophenol

Eluent Flow rate: 1.0 mL/min.

Regenerant: 35 mN H_2SO_4

Regenerant Flow rate: 10 mL/min.

Conductivity Detector Background Reading: <12 µS

TABLE 2. SINGLE-OPERATOR ACCURACY AND PRECISION FOR PERCHLORATE STANDARD SOLUTIONS

Sample Type	Sample Matrix	Known Conc. (µg/L)	Number of Replicates	Mean Recovery		SD (µg/L)	RSD (%)
				(µg/L)	(%)		
IPC Standard	RW	5.0	48	4.9	98	0.35	7.1
		100	47	100	100	4.2	4.2
QCS	RW	4.0	16	4.0	100	0.31	7.8
		100	4	100	100	2.8	2.8
LFB	RW	4.0	22	3.9	98	0.33	8.5

RW = reagent water

TABLE 3. SINGLE-OPERATOR ACCURACY AND PRECISION FOR PERCHLORATE MATRIX SPIKES

Sample Type	Sample Matrix	Spike Conc. (µg/L)	Number of Spiked Pairs	Duplicate Spike Mean Recovery		Mean RPD (%)	SD of Mean RPD (%)
				(µg/L)	(%)		
Matrix Spike/ Matrix Spike Duplicate	GW	4.0	20	3.8	95	2.1	0.02

GW = groundwater

TABLE 4. SINGLE-OPERATOR PRECISION FOR PERCHLORATE SAMPLE REPLICATES

Sample Type	Sample Matrix	Number of Replicate Pairs**	Mean RPD (%)	SD of Mean RPD (%)
Sample/Sample Duplicate	GW	14	1.4	0.02

GW = groundwater

**Note: Samples with perchlorate concentration ≥ 4.0 µg/L.