

**DEL MAR ANALYTICAL  
STANDARD OPERATING PROCEDURE  
DETERMINATION OF PERCHLORATE BY ION CHROMATOGRAPHY  
EPA 314.0**

SOP Number: 314\_0.SOP  
Revision: 0

Effective Date: 05/03/02  
Supersedes: None

**1. SCOPE AND APPLICATION/SUMMARY**

- 1.1. This method is applicable to drinking and surface waters, groundwater, and reagent waters. A fixed volume of sample is introduced into an ion chromatographic system. A large loop injection (1000  $\mu$ L) is used with a Dionex IonPac AG16 column and suppressed conductivity detection with an external water mode to quantify perchlorate at low levels.
- 1.2. The standard reporting limit for perchlorate is 4.0  $\mu$ g/L.

**2. APPARATUS AND MATERIALS**

- 2.1. Dionex DX-500 Ion Chromatograph or equivalent
- 2.2. Analytical balance
- 2.3. Pipettes and disposable pipette tips
- 2.4. 3 ml syringes
- 2.5. 10 ml syringe
- 2.6. 50 and 100 ml volumetric flasks
- 2.7. 15 ml disposable test tubes
- 2.8. 0.45 $\mu$ m Acrodisc filters
- 2.9. 0.22 $\mu$ m filters
- 2.10. Conductivity meter
- 2.11. Matrix pretreatment cartridges—barium form (Dionex OnGuard-Ba or equiv.)
- 2.12. Matrix pretreatment cartridges—silver form (Dionex OnGuard-Ag or equiv.)
- 2.13. Matrix pretreatment cartridges—hydrogen form (Dionex OnGuard-H or equiv.)

**3. STANDARDS AND REAGENTS**

- 3.1. De-ionized water, Type I reagent grade, with a resistance of 18 M $\Omega$ -cm or better
- 3.2. NaOH 50% w/w
- 3.3. Sodium Perchlorate 99% ACS reagent grade or better ( NaClO<sub>4</sub>, CASRN 7601-89-0)
- 3.4. Mixed common anion stock solution (NaCl, Na<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>CO<sub>3</sub>)
- 3.5. Conductivity meter calibration solution (Potassium chloride)
- 3.6. Perchlorate standard 1000ppm from Spex or equivalent source.

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**4. SAFETY**

- 4.1. Since all of the hazards of samples and chemicals used in this procedure are not entirely known, strict adherence to safety rules and use of prescribed personal protection equipment is mandatory. The health hazards of the standards, reagents and samples are not entirely known so caution must be exercised in all cases.
- 4.2. Employees performing this procedure must be familiar with the Chemical Hygiene Plan (CHP), and the precautions stated on the appropriate Material Safety Data Sheets (MSDS).
- 4.3. Personal Protective Equipment Required: Safety Glasses and Gloves

**5. DEFINITIONS**

- 5.1. A/H—Peak Area to Height Ratio. The ratio of the peak area divided by the peak height which is used to monitor analytical performance
- 5.2. ICCS—Initial Calibration Check Standard. A calibration standard solution at the MRL concentration and used to verify a previously established calibration.
- 5.3. IPC—Instrument Performance Check Solution. A solution containing a specific concentration of perchlorate (from the same source as the calibration) and used to evaluate instrument performance.
- 5.4. IPC-MA—Instrument Performance Check Solution in Mixed Anion Matrix. A solution containing a specific concentration of perchlorate in a matrix of other test substances (namely chloride, sulfate, carbonate) and used to evaluate instrument performance with respect to a defined set of criteria
- 5.5. MCT—Matrix Conductivity Threshold. The highest permitted conductance of a sample which is used to determine when matrix dilution or pretreatment is required.
- 5.6. See the laboratory QAPM and CADHS method and EPA 314.0 for additional definitions.

**6. INTERFERENCES**

- 6.1. Contaminants in the reagent water, reagents, glassware, or other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms, may cause method interferences.
- 6.2. Samples that contain particles larger than 0.45 microns require filtration to prevent damage to instrument columns and flow systems.
- 6.3. Pretreatment cartridges are effective in removing certain matrix interference. An additional pretreated Matrix spike/matrix spike duplicate and Method blanks are analyzed when samples need pretreatment.
- 6.4. High concentrations of anions interfere with the baseline of perchlorate. All samples must be monitored for conductivity prior to analysis. If the Matrix Conductivity threshold is exceeded, sample dilution and/or pretreatment are necessary.

**7. SAMPLE HANDLING AND PRESERVATION**

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- 7.1. The holding time for all samples is twenty eight (28) days.
- 7.2. Water samples: The samples may be stored at 4°C ± 2°C prior to analysis but room temperature storage is all that is required.
- 7.3. Soil samples: the samples must be stored at 4°C ± 2°C prior to analysis.
- 7.4. Notify the project manager immediately if the method hold time has been exceeded.

**8. PROCEDURE**

8.1. Standard Preparation

8.1.1. Calibration Standards

8.1.1.1. Prepare a 10 mg/L intermediate calibration standard by pipetting 500µL of commercially prepared 1000 mg/L calibration standard stock solution into a 50mL volumetric flask and bringing up to volume with Ultra pure water. The intermediate standard is prepared *once a month*.

8.1.1.2. Calibration standards are made by aliquoting the following amounts of the 10ppm intermediate standard into 100 ml volumetric flasks and bringing up to 100ml with ultrapure water. The diluted working standards should be *prepared daily*. The final concentrations are as follows:

Standard	Concentration	Amount Added
Blank	0 ppb	0 µL/100 mL
Std 1	2.0 ppb	20 µL/100 mL
Std 2	4.0 ppb	40 µL/100 mL
Std 3	10 ppb	100 µL/100 mL
Std 4	25 ppb	250 µL/100 mL
Std 5	50 ppb	500 µL/100 mL
Std 6	100 ppb	1000 µL/100 mL

8.1.2. Second Source Calibration Check Standard

8.1.2.1. The second source standard checks solution made from ACS grade Sodium perchlorate dried at 105°C for 30 minutes. Prepare the 1000ppm standard check solutions by adding 0.3078gram of Sodium Perchlorate (anhydrous) into 250mL of Ultra pure water. The standard is stable for one year when stored at 4° C.

8.1.2.2. Prepare a 10 mg/L intermediate second source stand check solution by pipetting 500uL of 1000ppm standard check into 50mL volumetric flask and bringing up with Ultra pure water.

8.1.3. Mixed Common Anion Solution

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- 8.1.3.1. Prepare the Mixed common anions solution by dissolving the following salts in reagent water to the final volume of 25mL: 1.0 grams sodium chloride, 0.93 grams sodium sulfate, and 1.1 grams sodium carbonate.
- 8.1.4. 50mM NaOH Eluent
- 8.1.4.1. Weigh out 1992 grams out degassed Ultra pure water and adding 8 grams of NaOH 50% w/w. Invert six times to mix. Do not shake vigorously. This will produce a concentration of 50mM NaOH. **Prepared daily.**
- 8.1.4.2. Notice: When measuring NaOH, take the aliquot from the middle of the bottle carefully. Do not disturb the NaOH, since Carbonate contamination can easily be produced in the NaOH from the outside air.
- 8.1.5. Prepare a 25 ppb Instrument Performance Check (IPC) calibration standard by adding 250  $\mu$ L of the 10 mg/L intermediate solution to a 100 ml volumetric flask and bringing up to volume with ultrapure water. **Prepared daily.**
- 8.1.6. Prepare a 25 ppb Instrument Performance Check in Mixed Anion Matrix (IPC-MA) calibration standard by adding 250  $\mu$ L of the 10 mg/L intermediate solution to a 100 ml volumetric flask and bringing up to volume with a mixed common anions solution that reflects a conductance near  $\pm 10\%$  the MCT (see section 8.2). **Prepared daily.**
- 8.1.7. Prepare a 50 ppb LCS/ICV/CCV by adding 500  $\mu$ L of the 10 ppm intermediate second solution to a 100ml volumetric flask and bringing up to volume with ultrapure water. **Prepared daily.**
- 8.1.8. Prepare the 4 ppb ICCS by adding 40uL of the 10ppm intermediate standard into a 100mL volumetric flask. **Prepared daily.**
- 8.1.9. Enter the standards information into ELMNT. Print out a copy of the standard and reagent information, have it reviewed and place in the standards binder. Store the solution in the refrigerator.
- 8.1.10. Enter the reagent information into the reagent logbook. Assign a unique identification code to all reagents used.
- 8.2. Determination of Matrix Conductivity Threshold (MCT)
- 8.2.1. Add 0.2mL, 0.3mL, 0.4mL,0.5mL,0.6mL,0.8mL,and 1.0mL of the mixed common anion into separate 25mL volumetric flasks. Add 0.0625mL of the perchlorate secondary stock solution standard (10ppm) to each flask. There should be eight volumetric flasks each contain 25 $\mu$ g/L perchlorate and ascending levels of the mixed common anion stock solution.
- 8.2.2. Measure and record the conductivity of each solution.
- 8.2.3. Analyze each solution and record the peak area to height ratio(A/H) and concentration of perchlorate.

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- 8.2.4. Calculate the A/H ratio percent difference (PD) between the LFB and each of the eight solutions as follows:

$$PD_{A/H} = \frac{|A/H_{IPC} - A/H_{MA}|}{A/H_{IPC}} \times 100$$

Where  $A/H_{LFB}$  = Area/height ratio for the IPC (no anion matrix)

$A/H_{MA}$  = Area/height ratio for each Mixed Anion Solution

- 8.2.5. The MCT is the conductance level of the highest mixed anion solution which yields a PD (A/H) value less than 20%.
- 8.2.6. After establishing the MCT, confirm the perchlorate MRL in a mixed common anion solution which reflects a conductance within  $\pm 10\%$  of the MCT. The recovered perchlorate concentration must be within 70-130% of the MRL. If the MRL recovery fails, the MCT should be lowered 10% and the verification repeated.
- 8.3. Calibration and Standardization
- 8.3.1. Perform a calibration initially and as needed after that (usually 4-6 weeks). Re-calibrate more frequently if the midpoint is  $>20\%$ , or whenever needed.
- 8.3.2. Prepare the calibration standards as described in 8.1.1. Standard S1 is at a concentration at or below the reporting limit. The other concentrations will define the working range of the detector. An acceptable calibration curve has a correlation coefficient of  $\geq 0.995$ .
- 8.3.2.1. The S1 standard at 2.0 ppb is analyzed with every calibration. If the correlation coefficient is not acceptable due to the 2.0 ppb standard, then the 2.0 ppb standard may be excluded from the calibration, as long as the 4.0 ppb standard is included in the curve. Note: If a client specifically requests a reporting limit of 2.0 ppb, then the 2.0 ppb standard must remain in the calibration curve.
- 8.3.3. Analyze the 4ppb ICCS. The perchlorate recovery must be 75-125% of the true value.
- 8.3.4. Analyze the IPC. The perchlorate recovery must be within 10% of the true value and the retention time must be within 5% of the retention time of the previous analytical batch.

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- 8.3.5. Analyze the IPC-MA. Calculate the PD<sub>(H/A)</sub> (Section 8.2.4), by comparing the peak area to height ratio to the value for the IPC and IPC-MA. The value for PD<sub>(A/H)</sub> must be less than 25%. The perchlorate recovery must be within 20% of the true value. The perchlorate retention time must be within 5% of the retention time of the previous analytical batch.
- 8.3.6. Analyze the ICV and ICB. The ICV is from a source separate from that used to make the calibration standards. The ICV must read 85-115% of the true value.
- 8.4. Sample Screening and Pre-Treatment
  - 8.4.1. The conductivity must be checked prior to analysis for all samples since the sample loop is 1mL and a high result may damage or overload the column. Peak saturation can occur when the analytical column capacity is exceeded. The response of 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
  - 8.4.2. If the samples have conductance greater than MCT level, then the sample can be diluted if it is detected. If the sample is not detected then pretreatment is required.
  - 8.4.3. Pretreat the samples and QC by stacking the cartridges from top to bottom (Ba/Ag/H). As a rinse, filter 3mL through the cartridges and discard. Filter sample through the stacked cartridges at a rate of 1mL/minute and collect 5-10mL.
  - 8.4.4. After pretreatment the samples are processed as follows:
    - 8.4.4.1. If the initial conductivity of the sample was less than twice the MCT, then the pretreated sample can be analyzed.
    - 8.4.4.2. If the initial conductivity of the sample was equal to or greater than twice the MCT, then the conductivity of the pretreated sample must be measured and recorded.
      - 8.4.4.2.1. If it is below the MCT, analyze the pretreated sample.
      - 8.4.4.2.2. If the conductivity is NOT below the MCT, check that the flow rate was not too fast and repeat the pretreatment. In some cases, double pretreatment cartridges may need to be applied.
- 8.5. Analysis
  - 8.5.1. Before starting the analysis, make enough fresh eluent to start and run the instrument for the day.
  - 8.5.2. Place eluent on top of the instrument and screw the cap on.
  - 8.5.3. Turn on the helium gas then turn knob #1 to approximately 15 psi and knob #2 to approximately 50 Kpa.

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- 8.5.4. Open the appropriate method file in the peaknet software. The instrument will turn on and start running. The pressure will rise up to 2000 to 2300 psi based on the column used.
- 8.5.5. To prime the system:
- 8.5.5.1. Change the IP20 isocratic pump from <Remote> to <Local>. Press enter.
- 8.5.5.2. Arrow to the right and change from Method to Direct Control. Press enter.
- 8.5.5.3. Arrow up and change 50 psi to 0 psi. Press enter. Now the pressure limits will read 0 - 2000 psi.
- 8.5.5.4. Open knob, then press Prime. Turn the lever to the left and inject 10 ml of isopropyl alcohol with a 10 ml syringe. Be careful when injecting isopropyl alcohol as to not inject any air bubbles into the system. While still pushing on the syringe handle, turn the lever back to the middle position.
- 8.5.5.5. Allow eluent to rinse through the line for approximately 60 seconds. Then turn off Prime and close knob.
- 8.5.5.6. Change back to 50 psi, Method, and Remote.
- 8.5.6. Allow the instrument to stabilize for approximately 1 hour.
- 8.5.7. Analyze samples after the instrument stabilizes. Following is an example of a sequence:

(For samples with conductances **less than** the MCT)

1. Blank
2. ICCS 4 ppb (25% recovery)
3. IPC 25ppb (10% recovery)
4. ICV/LCS (second source) 50ppb (15% recovery)[BS1]
5. ICB [MB1]
6. Sample Runs 1-10; including MS1/MSD1 (20% recovery)
7. CCV(second source) 25ppb (15% recovery)
8. CCB
9. Sample Runs 11-20
10. CCV (second source) 75ppb (LCS2)
11. CCB (MB2)

(For samples with conductances **greater than** the MCT)

1. Blank
2. ICCS 4 ppb (25% recovery)
3. IPC 25ppb (10% recovery)
4. IPC-MA 25ppb (20% recovery)
5. ICV(second source) 50ppb (15% recovery)
6. ICB
7. BS1 50ppb (20% recovery) **pretreated**
8. MB1 **pretreated**
9. Sample Runs 1-10; including MS1/MSD1 (20% recovery) **pretreated**
10. CCV(second source) 25ppb (15% recovery)

PD (A/H) between IPC and IPC-MA <25%
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11. CCB
  12. Sample Runs 11-20 **pretreated**
  13. CCV (second source) 75ppb (*15% recovery*)
  14. CCB
- 8.6. Instrument Conditions
- 8.6.1. Columns: IonPac AS16 Analytical; 4x250 mm (P/N 055346)  
IonPac AG16 Guard; 4x50 mm (P/N 055377)
  - 8.6.2. Flow Rate: 1.0 mL/min. (may vary with different suppressors)
  - 8.6.3. Sample volume: 1000 µL
  - 8.6.4. Detection: Suppressed conductivity, ASRS (4mm),  
AutoSuppression external water mode
  - 8.6.5. System Backpressure: 600-1900 psi
  - 8.6.6. Background conductance: below 5 µS
- 8.7. Results Reporting
- 8.7.1. Create a batch in ELMNT prior to loading the instrument. (it can always be edited later if something changes).
  - 8.7.2. Create a bench sheet in ELMNT. Enter the initial sample volumes and the final volumes. Enter the QC information: initial and final volumes, spike source, sample source for MS/MSD.
  - 8.7.3. Use data entry to manually transfer the data into ELMNT.
  - 8.7.4. Verify all sample results and QC results were entered correctly and verify QC acceptance prior to updating the status in ELMNT to analyzed.
- 8.8. Preventative Maintenance and Troubleshooting
- 8.8.1. Low pressure: Check for air bubbles and prime the system with isopropyl alcohol to correct for low pressure.
  - 8.8.2. High pressure: Most likely the column is dirty. Replace the frits and guard discs. Clean the column for two hours with 10x concentrated eluent. Be sure to read Dionex instructions before attempting to clean the column.
  - 8.8.3. Tailing of peaks: The column is dirty. Replace the frits and guard discs. Clean the column for two hours with 10x concentrated eluent. Be sure to read Dionex instructions before attempting to clean the column.
  - 8.8.4. High display value: Be sure that the ASRS-II is clean, make sure the eluent is prepared correctly, and that the correct current is being applied to the ASRS-II.
  - 8.8.5. Noisy baseline/unable to read low end standards: Reprime the system and/or replace the suppressor. Note: The suppressor flow rate should be 10ml/min in and 3.5ml/min out.
  - 8.8.6. Shifted retention times: Recalibrate the instrument.



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- 8.8.7. Difficulty in reading the 4.0 ppb standard within  $\pm 25\%$ : Make new eluent using a new bottle of 50% w/w NaOH.
- 8.8.8. Record all performed maintenance in the instrument maintenance logbook.
- 8.8.9. If an instrument is unusable or has limitation to its use, it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution, and verification of proper operation into the instrument maintenance logbook.

**9. QUALITY CONTROL**

- 9.1. LCS/ICV/CCV. Analyze this second source calibration check standard after the ICP check and after every 10 samples. The recovery must be 85-115%
- 9.2. ICCS 4ppb check standard. Recovery must be between 75-125%.
- 9.3. ICP. Verify the calibration curve by analyzing an IPC check standard on each working day, or after changing the anion eluent. If the response or retention time for any analyte varies from the expected values by more than +10%, re-analyze using a fresh IPC standard. If the results are still more than +10%, prepare a new calibration curve.
- 9.4. IPC-MA. For samples with conductances greater than the MCT level. The following QC criteria must be met by the IPC-MA:
  - 9.4.1. PD (A/H) between the LFB and the IPC-MA must be less than 25% based on the formula in section 8.2.4.
  - 9.4.2. The perchlorate concentration in the IPC-MA must be within 20% of the true value.
  - 9.4.3. The retention time window for perchlorate should not shift more than 5% from the previous calibration. Once the retention time varies by more than 20% from the originally determined time, the column(s) should be cleaned or replaced.
  - 9.4.4. If any of conditions are not met for the IPC-MA, the MCT must be repeated or the source of problem must be determined and the IPC reanalyzed.
- 9.5. Analyze a method blank for every batch of twenty samples, or less at the beginning of the sequence. The method blank must be free of contamination below the reporting limit, or the system must be cleaned and/or recalibrated and samples reanalyzed. (For water samples, the ICB/CCBs are reported as method blanks).
  - 9.5.1. If the method blank is greater than the reporting limit, but the samples are N.D., the results may be reported and submit a corrective action report.
  - 9.5.2. If the method blank is greater than the reporting limit, but the samples are greater than 10 times the method blank, flag the results with a 'B1' qualifier and submit a corrective action report.

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- 9.6. Analyze a matrix spike (MS)/matrix spike duplicate(MSD) every batch of twenty samples or less. Control limits of 80%-120% are to be used. If results are outside of control limits, verify that the ICV/LCS is in control. If the system is in control then sample matrix is deemed the problem. If matrix is the problem, flag the MS/MSD with the appropriate 'M' qualifier. If the problem is not matrix related, fill out a corrective action report and describe the problem and corrective actions taken.
- 9.6.1. The 10ppm intermediate solution is also used to prepare the matrix spike solution.
- 9.6.2. For sample analyzed undiluted, 10mL of sample is combined with 50uL of the 10ppm standard. The final concentration is 50ppb.
- 9.6.3. For samples analyzed at a dilution of 1:2, 1:4, 1:5 (the final concentration is 50ppb):
- 9.6.3.1. Spike 50uL of 10ppm onto 10mL of straight sample. Mix and dilute to appropriate level.
- 9.6.4. For samples analyzed at a dilution of 1:10 or more(final concentration is 500ppb)
- 9.6.4.1. Spike 500uL of 10ppm onto straight sample. Mix and dilute to appropriate level.
- 9.7. An MDL Study must be performed semi-annually or when there is a major system change; e.g. new eluent concentration, different calibration levels, new analyst, etc. On a calibrated instrument, analyze the low calibration standard 7 times. The MDL calculated as follows:  
MDL = (3.14) x the standard deviation (SD) of the 7 replicates.
- 9.8. Perform an Initial Demonstration of Capability (IDOC) by preparing and analyzing 4 LCS samples with an average % recovery of between 90-110% and an RPD < 20%. If this is not achieved, repeat the IDOC.

**10. CALCULATIONS**

- 10.1. Consider all of the dilution factors when calculating an anion concentration.

**11. PAPERWORK FLOW**

- 11.1. Daily
- 11.1.1. Query ELMNT for the desired analysis codes to determine what needs to be analyzed.
- 11.1.2. Complete the sequence file as samples are analyzed. Include on the logsheet the QC batch number, method number, the analyzed date, the analyst's initials, the sample numbers, sample pH, dilution factors, and the pertinent client codes. Record the Standard ID number for each standard used in the analytical sequence.
- 11.1.3. Attach a printout to its corresponding logsheet after reporting results and then file it in the appropriate cabinet.

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11.2. Monthly

11.2.1. Record the monthly calibration curve and file in an appropriate folder.

11.3. As Needed

11.3.1. Complete a Corrective Action Report whenever any non-matrix related quality control sample result is outside of acceptance limits, and notify the QA Officer or Laboratory Manager. Notify the Project Manager immediately if any of the results must be qualified (e.g. missed holding times) so the client can be informed as soon as possible.

11.3.2. Enter the standards information into ELMNT. Print out a copy of the standard information, have it reviewed and place in the standard binder. Store the solution in the refrigerator.

**12. POLLUTION PREVENTION**

12.1. Samples with high concentrations of perchlorate should be segregated and disposed of as hazardous waste.

**13. WASTE MANAGEMENT**

13.1. See 'Laboratory Safety Plan'.

**14. METHOD PERFORMANCE**

14.1. See attached analysis information for information regarding reporting limits, control limits, and method detection limits.

**15. METHOD REFERENCES**

15.1. EPA Method 314.0 Determination of Perchlorate in Drinking Water Using Ion Chromatography, Revision 1, November 1999.

15.2. Dionex Application Note 121 1-13-98 Draft.

**16. REFERENCE VARIANCES**

16.1. Sodium perchlorate is used as the stock standard in place of Potassium Perchlorate for safety reasons.

16.2. For samples with relatively low initial conductivity (less than 2 times the MCT), the laboratory does not measure the conductivity after pretreatment.

16.3. The daily determination of the  $PD_{A/H}$  is based on the responses of the IPC and IPC-MA run on the same day and not from the IPC in the previous analysis batch.

Approved By: \_\_\_\_\_ Date: \_\_\_\_\_

**Laboratory Approval**

Approved By: \_\_\_\_\_ Date: \_\_\_\_\_

**Quality Assurance Approval**