



# Albuquerque Bernalillo County Water Utility Authority

WATER RECLAMATION DIVISION  
4201 2ND STREET SW, ALBUQUERQUE, NEW MEXICO 87105

## WATER QUALITY LABORATORY STANDARD OPERATING PROCEDURE APPROVAL FORM

### WQL SOP 205 Anions

CURRENT VERSION # 7

Implementation Date: 8/20/09

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Out of Service By: \_\_\_\_\_ Date:  / /  
Reason:

**History of Revision** This table lists the revision history and effective dates of this procedure.

<b>Revision</b>	<b>Date</b>	<b>Description of Changes</b>
6		Revised to address comment in A2LA audit on manual baseline changes and data backup. Also updated SOP to address change to Chromeleon software. Changed procedure to provide more stepwise instructions.
7		Revised to address addition of Dionex, ICS-2000. Revised Anions Maintenance Log.

## 1.0 SCOPE AND APPLICATION

- 1.1. This SOP covers the determination of the following inorganic anions: Bromide, chloride, nitrate, nitrite, orthophosphate, and sulfate. This method is not applicable to quantification of fluoride. The range is specific to each analyte as in the table below. Ranges can be extended with sample dilution.

Chloride	0.2ppm-80ppm
Nitrite	0.05ppm-6ppm
Bromide	0.1ppm-10ppm
Nitrate	0.05ppm-6ppm
Phosphate	0.1ppm-8ppm
Sulfate	0.5ppm-100ppm

- 1.2. This method is applicable to drinking surface and saline waters, domestic and industrial wastes, solids (after extraction) and leachates (when no acetic acid is used).

## 2.0 SUMMARY OF METHOD

- 2.1. A water sample is injected into a stream of carbonate-bicarbonate eluent and passed through a series of ion exchangers (guard column, and separator column). The anions of interest are separated on the basis of their relative affinities for a low capacity, strongly basic anion exchanger (separator column). The separated anions are directed through a micro-membrane suppressor. In the suppressor the separated anions are converted to their highly conductive acid forms and the carbonate-bicarbonate eluent is converted to weakly conductive carbonic acid. The separated anions in their acid forms are measured by conductivity. They are identified on the basis of retention time as compared to standards. Quantification is by measurement of peak area.

## 3.0 DEFINITION OF TERMS

- 3.1. QA SOP-007- Reference for general terms related to quality and technical procedures, which applies to all standard operating procedures within WQL.

## 4.0 INTERFERENCE

- 4.1. Any substance that has a retention time coinciding with that of any anion to be determined and produces a detector response will interfere. For example, relatively high concentrations of low-molecular-weight organic acids interfere with the determination of chloride and fluoride by isocratic analyses. A high concentration of any one ion also interferes with the resolution, and sometimes retention, of others. Sample dilution overcomes much interference. To resolve uncertainties of identification or quantitation use the method of known additions. Spurious peaks may result from contaminants in reagent water, glassware, or sample processing apparatus. Because small sample volumes are used, scrupulously avoid contamination. Modifications such as pre-concentration of samples, gradient elution, or re-injection of portions of the eluted sample may alleviate some interference but require individual validation for precision and bias.

## 5.0 SAFETY

- 5.1. **Health Hazards**

5.1.1. For specific hazards, consult the MSDS for compounds listed in section 7.0 of this SOP [MSDS on file in WQL Conference Room].

5.1.2. Use, store, and dispose of chemicals in accordance with WQL Chemical Hygiene Plan (CHP-Section 5- Revision December 2005).

5.2. **Protective Equipment**

5.2.1. Wear appropriate Personal Protective Equipment (PPE) in accordance with WQL CHP (Section 5.1 – Revision December 2005)

5.3. **Spills and Contamination**

5.3.1. Clean up spills immediately in accordance with WQL CHP (Section 5.11- Revision December 2005).

**6.0 APPARATUS AND EQUIPMENT**

6.1. All analytical equipment requirements for availability, installation, out-of-service, and record keeping (identification, manufacture, serial #, model #, and date of purchase) will follow WQL Quality Assurance Manual (QAM) procedures (Section 5.5).

6.2. Dionex, ICS-2000

6.2.1. RFIC

6.2.2. Automated sampler, AS40

6.2.3. 0.45 um syringe filter

6.2.4. Dionex AS40 and ASM 5 mL Vials and Filter Caps

6.3. Dionex, DX500

6.3.1. LC20

6.3.2. Automated sampler, AS40

6.3.3. Conductivity Detector, CD20

6.3.4. Isocratic pump, IP25

6.3.5. 0.45 um syringe filter

6.3.6. Dionex AS40 and ASM 5 mL Vials and Filter Caps

6.4. Chromeleon Software with dongle

**7.0 REAGENTS AND STANDARDS**

7.1. **Chemicals/Reagents** - All chemicals and reagents transport and storage requirements will follow WQL QAM procedures (Section 5.6.4).

7.2. **Deionized water**, free from interference at the minimum detection limit of each constituent, filtered through a 0.2 um filter to avoid plugging columns, and having a conductance of less than 1.0  $\mu\text{S}/\text{cm}$ .

7.3. **Eluent solution**

7.3.1. Dionex, ICS-2000:

7.3.1.1. Purchase EluGen cartridge, EGCIK<sub>2</sub>CO<sub>3</sub>

7.3.2. Dionex, DX500:

7.3.2.1. **0.5M sodium bicarbonate**- Dissolve 41.7g sodium bicarbonate in DI water and dilute to one liter in a one liter volumetric flask.

7.3.2.2. **0.5M sodium carbonate**- Dissolve 52.8g sodium carbonate in DI water and dilute to one liter in a one liter volumetric flask.

7.3.2.3. **0.5M sodium bicarbonate-sodium carbonate eluent**- prepare solutions by addition of 7mL stock 0.5M sodium carbonate and 2mL stock 0.5M sodium bicarbonate stock solution dilute to one liter in a one liter volumetric flask.

7.4. **Standard anion solutions, 1000 mg/L:** Stock standards are purchased. The alternative if purchased standards are unavailable is to prepare the stock standards following the table below.

Prepare a series of standard anion solutions by weighing the indicated amount of salt, dry salt at 105°C for one hour, cool in a desiccator to a constant weight and dilute, to one liter with DI water in a one liter volumetric flask. Bring sodium nitrite to constant weight in a desiccator, **DO NOT** oven dry!

<u>Anion</u>	<u>Salt</u>	<u>Amount g/L</u>
Chloride	NaCl	1.6484
Bromide	NaBr	1.2876
Nitrate(N)	NaNO <sub>3</sub>	6.0679
Nitrite(N)	NaNO <sub>2</sub>	4.9257 <b>DO NOT</b> oven dry, place in desiccator
Phosphate(P)	KH <sub>2</sub> PO <sub>4</sub>	4.3937
Sulfate	K <sub>2</sub> SO <sub>4</sub>	1.8141

7.5. **Working anion solutions**

**Calibration and warm up standard**

The following combined Working Standard is a 100%, prepare by addition of Stock Standard for each analyte in a one liter volumetric flask and diluted to volume with DI water to obtain the following concentrations:

<u>Anion</u>	<u>mL Stock Std.</u>	<u>Conc. ppm</u>
Chloride	40	80
Bromide	5	10
Nitrate(N)	3	6
Nitrite(N)	3	6
Phosphate(P)	4	8
Sulfate	50	100

Take an aliquot from the 100% calibration standard for the following standards; 10%, 25% and 50%(The 50% is used as a CCVS). Take an aliquot for 25% LCS, LCSD.

**ICVS solution**

Combine the following working standards from a second source or second lot as a 50% ICVS in a one liter volumetric flask and diluted to volume with DI water to obtain the following concentrations.

<u>Anion</u>	<u>Conc. ppm</u>
Chloride	40
Bromide	5
Nitrate(N)	3
Nitrite(N)	3

Phosphate(P)	4
Sulfate	50

**Matrix Spike**

The following is a spike solution used with a sample selected for the Matrix sample and Matrix sample duplicate.

<u>Anion</u>	<u>Conc. ppm</u>
Chloride	5
Bromide	5
Nitrate(N)	5
Nitrite(N)	5
Phosphate(P)	5
Sulfate	5

**8.0 QUALITY ASSURANCE/ QUALITY CONTROL**

- 8.1. **Analyst Training** - Analysts must follow the steps outlined in the DOC Training Program for WQL SOP's. Follow requirements in QA SOP-004.
- 8.2. **Quality Control Requirements** – Follow requirements in QA SOP-005. The Quality Control Requirements section covers the following topics: 1) Quality Control Limits 2) Quality Control - Instrument Performance 3) Laboratory (Method)
- 8.3. **Data Evaluation**- Follow requirements in QA SOP-005. The Data Evaluation section covers the following topics: 1) Internal Audits 2) Control Charts Procedures 3) Performance Audits 4) Method Detection Limit Procedures
- 8.4. **Calibration Requirements** - The calibration curve must be produced using three or greater ICALs. The calibration curve generated must have a regression analysis performed on each analyte.
  - 8.4.1. A correlation coefficient, resultant from the regression analysis, must be greater than or equal to 0.995 for each analyte.
  - 8.4.2. A mid range ICVS must be run to validate the calibration curve. The percent difference must be no greater than 10%.
  - 8.4.3. If the acceptability limits are not met for the correlation coefficient and/or the ICVS, then a new calibration curve must be generated which meets the acceptability limitations.
- 8.5. **Manual Integrations** -
  - 8.5.1. Manual integrations or baseline adjustments are not allowed. In the case where baseline noise interferes with the analysis or quantitation, the affected samples must be reanalyzed.

**9.0 PROCEDURE**

- 9.1. **Sample Handling**
  - 9.1.1. **Preservation** - 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.
  - 9.1.2. **Sample Holding Time** - Sample preservation and holding times for the anions that can be determined by this method are as follows:

Analyte	Preservation	Holding time
Bromide	None	28 days
Chloride	None	28 days
Nitrate	Cool to 4 <sup>o</sup> C	48 hours
Nitrite	Cool to 4 <sup>o</sup> C	48 hours
O-Phosphate	Cool to 4 <sup>o</sup> C	48 hours
Sulfate	Cool to 4 <sup>o</sup> C	28 days

9.1.3. **Storage** - In a given sample, the anion that requires the most preservation treatment and the shortest holding time will determine the preservation treatment. Generally, all samples should be refrigerated or kept cool to 4<sup>o</sup>C and analyzed within forty eight hours, if the analysis includes any of the following analytes: Nitrate, Nitrite or O-Phosphate.

9.1.4. **Sample Preparation** –Bring samples to room temperature. All samples must be filtered to remove particles larger than 0.2 µm with a Dionex AS40 filter vial cap. For solid samples the following extraction should be used. Add an amount of DI water equal to ten times the weight of dry solid material taken as a sample. This slurry is mixed for ten minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45 µm membrane syringe filter.

9.2. **Dionex, ICS-2000 Equilibration:**

9.2.1. Perform daily instrument maintenance (See Section 11.3).

9.2.2. Prime if DI water has been added.

9.2.2.1. Open door and verify that the primary valve on the primary pump head is closed (turn clockwise).

9.2.2.2. Open waste valve on secondary pump head one-half turn counter clockwise.

9.2.2.3. Press the Prime button Chromeleon control panel or on ICS-2000 ICS touch-screen home.

9.2.2.4. Prime until all air or previous are purged and no air bubbles are exiting the wasted line.

9.2.2.5. Press pump off and close waste valve. Do not over tighten.

9.2.3. In the Chromeleon software go to the Dionex Control Panel. In the system window connect the ICS-2000 system and click on the connected tab. Wait for the connection to be established.

9.2.4. Select the start up button at the top-right corner of the page.

Note: This will initiate the pump to start pumping at 1.2 ml/ min and the suppressor to run. It will take a few minutes for the instrument to equilibrate and eluent to begin mixing (EGC-eluent generator cartridge).

9.2.5. Allow the instrument to run while you set up run.

Note: Wait for the conductivity of the eluent, which is on the right hand upper corner of the detector screen, to stabilize. This may take 30 minutes or more. You can use the real-time display on the Chromeleon software to provide a graphical representation of the conductivity.

9.3. **Dionex, DX 500 Equilibration:**

9.3.1. Perform daily instrument maintenance (See Section 11.2).

- 9.3.2. Prepare Eluent if necessary See Section 7.3). Record in Ion Chromatography reagent log.
- 9.3.3. Prime if using new eluent or when the instrument has not been used.
  - 9.3.3.1. Open door on LC20 and panel door below IP25 display.
  - 9.3.3.2. Open pressure transducer waste valve' by turning valve ~1.5 rotations counter clockwise (valve located above and between pump pistons).
  - 9.3.3.3. Press the Prime button on IP25 control panel.
  - 9.3.3.4. Allow pump to prime ~2 minutes. Prime for about 5 minutes if the DX500 was off. Press Prime button a second time to stop priming.
  - 9.3.3.5. Close the priming block, by turning the valve clockwise until semi-tight to the touch.
- 9.3.4. In the Chromeleon software go to the Dionex Control Panel. In the system window connect the CD20 and IP25 click on the connected tab. Wait for the connection to be established.
- 9.3.5. Select the start button at the top center of the page.

Note: This will initiate the pump to start pumping at 1.00 ml/ min and the suppressor to run and set it at 50mA. You will see bubbles coming out of the suppressor when this happens it will take a few minutes for the instrument to equilibrate.

- 9.3.6. Allow the instrument to run while you set up run.

Note: Wait for the conductivity of the eluent, which is on the right hand upper corner of the CD20 screen, to stabilize. This may take 30 minutes or more. You can use the real-time display on the Chromeleon software to provide a graphical representation of the conductivity.

#### 9.4. INSTRUMENT CALIBRATION:

- 9.4.1. Instrument must be calibrated every time that new eluent is changed out or as indicated by failure of Calibration verification standards.

Note: Calibration consists of generation of a calibration curve via Dionex Chromeleon software interfaced with the ion chromatograph conductivity cell output module. The calibration curve must be produced using three or greater ICALs. The calibration curve generated must have a regression analysis performed on each analyte. A correlation coefficient, resultant from the regression analysis, must be greater than or equal to 0.995 for each analyte. A mid range ICVS must be run to validate the calibration curve, the percent difference must be no greater than 10%. If the acceptability limits are not met for the correlation coefficient and/or the ICVS, then a new calibration curve must be generated which meets the acceptability limitations.

#### 9.5. Dionex, ICS-2000 and Dionex, DX500 Set up sequence:

- 9.5.1. In the Browser area click on the left hand side of the screen under ICS-2000 or DX500, then sequences.
- 9.5.2. Pick the last acceptable analysis file and save the file with the following format "DDMMYY" using the current date. This is the batch ID.
- 9.5.3. In the **type** field use unknown for all samples and QC. Use standard only for the calibration standards.
- 9.5.4. For the ICS-2000 only: In the **Inj. Volume** field, the volume should be set at 40.0 µL
- 9.5.5. In **Name** field enter the SQL\*LIMS Sample ID for samples or the working standard ID for standards.



- 9.5.6. In the **program** field use Anion for the ICS-2000, or Anion 4 point calibration.pgm for the DX500, for all the samples and shutdown for the shutdown. The program gives commands to the Ion Chromatography system.
- 9.5.7. In the **Method** field use the Anions method for all. The method instructs how the data is calculated.
- 9.5.8. In the **Status** field there is single, finished or interrupted. Single means it has not been run. Finished means it has been run and interrupted means it started but run stopped on that sample.
- 9.5.9. In the field **Inj.** Date and time will be stamped when sample starts running.
- 9.5.10. In the field **Dil. Fact.** Is the dilution factor. Be sure to change this before you start run.
- 9.5.11. In the field **Sample Id.** the User sample ID from SQL\*LIMS which is the sample point or the standard name.
- 9.5.12. In the field **Comment** may be used for a comment and/or analyst initials.
- 9.5.13. When done with the sequence, or if any changes are added, save the sequence. The software will not recognize the change until it is saved
- 9.6. **Setup vials-** right click on the sequence and print sequence. If needed print out method files and/or program files, to setup vials only the sequence is needed.
  - 9.6.1. Every sample needs a vial.
  - 9.6.2. When setting up the autosampler cassette the track needs to be facing toward the front of the autosampler when loaded into the autosampler. Set vials up with the track side of the cassette flipped around and then order the samples from left to right.
  - 9.6.3. Label vials in the cassette and fill with sample to the fill line and place a Dionex AS40 vial filter cap on each vial. Check to see that each filter cap has a filter in it. The filter protects the lines and columns from allowing any debris from entering. Use the filter cap adjustor to align and push the filter cap into vial.
  - 9.6.4. When setting up the autosampler cassette the track needs to be facing you when loaded into the autosampler. So set up the vials with the track side of the cassette flipped around and then order the samples from left to right. When cassette is loaded into the autosampler the first sample should be the next one going into the injector.
  - 9.6.5. The first vial, last vial and after any known high concentration sample place a DI water rinse. Fill vial with DI and align the filter cap but do not push into the vial. Before starting run, ensure that the autosampler is in the run position. If it is not it will not communicate with the Ion Chromatography system/CD20 (ICS-2000/DX 500). At the very beginning of the run allow the rinse to go through by pressing the hold/run button on the autosampler. Do not start run until rinse has completed.

Note: Perform a baseline, by going to the Dionex control panel click on the blue dot on the control bar this is the acquisition tab. Data acquisition window pops up, highlight the ECD device and click ok. Allow the unit to run for a few minutes. Turn acquisition off by clicking on the blue dot again and a window will come up asking if you want to stop acquisition. The acquisition must be off before you can start the run.

- 9.6.6. To start run, go to sequence, then batch, and the start window will come up with sequences not run yet or with samples not run yet. Highlight the one you want

and press start. When the batch has started, the first sample to be run will be highlighted green.

Note: To see the report or the integration click twice on the sample that is running and the integration screen will come up. The report is shown; the QNT editor and calibration setup can be accessed by selecting the tools on the toolbar. During the calibration check to see that all the peaks of interest are being labeled.

9.6.7. When the run is complete, check all the data to ensure that QC has passed and that all samples were analyzed.

9.6.8. Print out report with the calibration correlation coefficient attached to it. Do calculations. File the batch with the sequence report, Sample Summary Report sheet and all reports.

## 10.0 DATA REPORTING

10.1. **Calculations** -Provide a list of calculations used in this method. All calculations are automatic with the Chromeleon software.

10.1.1. Due to the increased volume of standard used for the Matrix spike, the following calculation is used to correct for the volume of standard being used.

$$\text{Correction factor} = \frac{\text{Total volume,mls} - \text{mls standard}}{\text{Total Volume,mls}}$$

$$\% \text{ Recovery} = \frac{\text{MS} - (\text{M} \times \text{Correction factor})}{\text{spike concentration}}$$

10.2. **Log Entry**-Record the batch ID (See 9.4.2) in the Sample Summary log and any comments on the batch.

10.3. **Corrective Actions**- Follow requirements in QA SOP-003 and QA SOP-005. The Corrective Action section covers the following topics: 1) Out of Control Data Procedures and 2) Corrective Action Logbooks.

10.4. **Data Assessments** – Follow requirements in QA SOP-005. The Data Assessments section covers the following topics: 1) Accuracy and Precision 2) Data Validation Procedures 3) Data Reporting Procedures.

10.5. **Data Entry** – Enter results in SQL-LIMS as required.

## 11.0 MAINTENANCE

Note: Use the Anions Maintenance Log to record all maintenance conducted.

11.1. **Dionex, ICS-2000 Instrument run specifications:**

11.1.1. Before starting batch perform ready check. Ready check will tell you how much eluent will be needed.

11.2. **Dionex, DX 500 Instrument run specifications:**

11.2.1. Nitrogen psi leaving the tank needs to be between 80-100psi

11.2.2. Eluent psi needs to be between 40-60psi or 6-9.

11.2.3. Enough eluent for one run would be 1000mLs.

11.3. **Dionex, ICS-2000 Daily Instrument Maintenance (Conducted each time the instrument is used for analysis):**

- 11.3.1. Rinse the piston to remove any crystallization. The crystals can abrade the piston and cause the main seal to leak.
  - 11.3.1.1. Open the pump door and locate the two pump heads.
  - 11.3.1.2. Open waste valve on the left-side pump and attach small syringe to the rinse/prime port on the right-side pump.
- 11.3.2. Check the entire chassis for any leaks from the rinse ports, the eluent manifold connections and valves and eluent reservoirs. Tighten or replace any leaking fittings.
- 11.3.3. Wipe up spills and rinse dried reagents off the pump components with DI.
- 11.4. **Dionex, DX 500 Instrument Maintenance (Conducted each time the instrument is used for analysis)**
  - 11.4.1. Rinse the piston to remove any crystallization. The crystals can abrade the piston and cause the main seal to leak.
    - 11.4.1.1. Open the pump door and locate the two rinse ports on the front of each of the pump heads.
    - 11.4.1.2. Install waste pump tube onto the pump head. And place in a waste beaker. Attach small syringe to the pump head and flush 3 to 4 times with 5-10mLs of DI.
    - 11.4.1.3. Do this for both pump heads.
  - 11.4.2. Check the entire chassis for any leaks from the rinse ports, the eluent manifold connections and valves and eluent reservoirs. Tighten or replace any leaking fittings.
  - 11.4.3. Wipe up spills and rinse dried reagents off the pump components with DI.
  - 11.4.4. Check nitrogen pressure going into the DX500 and tank level. Pressure going into the DX500 needs to be between 80-100psi.
- 11.5. **Dionex, ICS-2000 - Periodically:**
  - 11.5.1. Check all air and liquid lines for crimping. Move or reroute pinched lines: replace damaged lines.
  - 11.5.2. Check liquid lines for leaks and clean up any spills.
  - 11.5.3. Check and replace column beds every 4-6 or when needed. Look for a white or brown crust on the bed surface. Replace the beds only on the influent side of the column.
- 11.6. **Dionex, DX 500 – Periodically:**
  - 11.6.1. Check all air and liquid lines for crimping. Move or reroute pinched lines: replace damaged lines.
  - 11.6.2. Check liquid line from the CD20 to the conductivity cell for leaks and clean up any spills.
  - 11.6.3. Check and replace column beds every 4-6 weeks. The guard column will need it more often. Look for a white or brown crust on the bed surface. Replace the beds only on the influent side of the column.

- 11.6.4. When there is a color change on the eluent filter Change out. The eluent filter should be white.

## 12.0 TROUBLESHOOTING

### 12.1. Dionex, ICS-2000

- 12.1.1. For problems with the Ion Chromatography System, see ICS-2000 Operator's Manual, Ch. 4. Troubleshooting, on Dionex Reference Library Disc.
- 12.1.2. Calibration Peak Identification:
- 12.1.2.1. If there are unidentified peaks in the calibration because peaks drifted past the 0.5second retention time window.
- 12.1.2.2. Go to the integration screen at the top of the chromatograph.
- 12.1.2.3. Select the appropriate peak.
- 12.1.2.4. Slide peak identifier to fit over the unnamed peak and save.
- 12.1.2.5. Indicate changes on the first report and reprint after changes are made.
- 12.1.2.6. Report results from the second report and keep the first report with the batch.
- 12.1.2.7. Recalculate the calibration based on the new retention times and peak identifications.

### 12.2. Dionex, DX500

#### 12.2.1. Low pressure limit violation

- 12.2.1.1. Verify that eluent is present in the channel selected. If the eluent reservoir is empty, refill it. Prime the pump before resuming operation.
- 12.2.1.2. Make sure the waste valve on the pressure transducer is closed by turning the knob on the pressure transducer housing clockwise.
- 12.2.1.3. Make sure there are no liquid leaks in the flow system.
- 12.2.1.4. Place the pump in LOCAL, DIRECT CONTROL. Press off/on to start the pump and verify that the pistons are moving and that you can hear the pump. If there is no sound from the pump, check the LED on the CPU case inside the door to the electronics chassis. A red LED indicates a defective power supply. Replace the power supply. Contact Dionex Technical Support.
- 12.2.1.5. With the pump running, pen the DSP STATUS screen and note whether the left-right pressure varies by more than 3% between strokes. If it does, refer to Section 4.1 of the pump section Ref.14.1. If it does not, either increase the flow rate or reduce the low pressure limit setting and continue operation.

#### 12.2.2. Liquid leaks/Leak Alarm

- 12.2.2.1. Pump head: Leaks from the front rinse ports or rear of the pump head may indicate a defective piston seal. Replace the piston seal and the rinse seal (see section 5.2 of the pump section Ref.14.2). Check all connections between the eluent.

12.2.2.2. Pressure transducer: Inspect the pressure transducer. If the source of the leak is the waste valve, replace the waste valve O-ring (see Section 5.4 of the pump section in the DX500 operator's manual). If the leak is from the rear of the transducer, call Dionex Technical Support.

12.2.2.3. Priming valve: Tighten any leaking fittings just enough to stop the leak. If this does not stop the leak, replace the fittings and/or tubing making the connection. If this does not stop the leak, replace the priming block assembly.

12.2.2.4. Interior mechanical chassis leaks: Inspect the chassis for leaks Tighten any leaking fittings. Replace any damaged parts.

#### 12.2.3. Calibration Peak Identification:

12.2.3.1. If there are unidentified peaks in the calibration because peaks drifted past the 0.5second retention time window.

12.2.3.2. Go to the integration screen at the top of the chromatograph.

12.2.3.3. Select the appropriate peak.

12.2.3.4. Slide peak identifier to fit over the unnamed peak and save.

12.2.3.5. Indicate changes on the first report and reprint after changes are made.

12.2.3.6. Report results from the second report and keep the first report with the batch.

12.2.3.7. Recalculate the calibration based on the new retention times and peak identifications.

### 13.0 WASTE DISPOSAL AND POLLUTION PREVENTION

13.1. All waste disposal procedures will follow the Water Quality Laboratory CHP (Section 5.12-Revision December 2005). Disposal procedure is as follows:

13.1.1. Discard all remaining analyzed samples in an acid sink.

13.1.2. All sample labware must be washed with laboratory soap inside and out followed by multiple rinses with distilled or deionized water.

13.2. Pollution Prevention - Eliminate waste at the source and base the quantity of purchased reagents on expected usage during their shelf life.

### 14.0 REFERENCES

14.1. Standard Methods 18<sup>th</sup> edition of Standard Methods, 4110B

14.2. ICS-2000 Operator's Manual, Dionex Reference Disk

14.3. DX 500Chromatography System Operator's Manuals.

### 15.0 LOGSHEET CONTROL DOCUMENTS

Note: Data for each analytical run is recorded electronically and printed out from the data file. These records are the data. Data are saved on the network, which is backed up nightly. Data review is conducted on the printout of the run file and the sample summary report.

15.1. Anions Maintenance Log