Dionex IC Protocol Version A 10/17/2007

- 1. System Startup
 - 1.1. Open Chromeleon software from desktop. Active window will default to window last opened from previous user.
 - 1.2. Go to 'Browser' window and select correct instrument panel from directory tree depending on the instrument configuration to be used (with or without Eluent Generation)
 - 1.3. On instrument panel, check for system connections
 - 1.4. Check eluent levels and prep solutions as needed
 - 1.4.1. Fill reservoir for ICS 2000 with Nanopure. Eluent generator will produce carbonate/bicarbonate eluent per instrument panel and program settings
 - 1.4.2. Fill reservoir for ICS 1000 with a solution of 4.05 mM Sodium Carbonate and 0.72 mM Sodium Bicarbonate (18 ml each of 450mM and 80mM stock solutions, respectively in 2 L flask. QS with nanopure water).

NOTE: Starting eleuent levels must be entered under 'Pump Settings'; Chromeleon calculates eluent usage via program parameters, not by sensing volume. Warnings will appear at \leq 200 ml. (See STATUS page on instrument or ready check prior to start of batch processing)

- 1.5. Check waste container and empty if needed
- 1.6. Click 'Startup' on instrument panel window
 - 1.6.1. Monitor pump pressures; if pumps have difficulty reaching or holding pressure, prime pumps

NOTE: WASTE VALVE MUST BE OPEN BEFORE PRIMING TO PREVENT COLUMN DAMAGE

- 1.6.1.1. Click 'Shutdown' then 'Pump Settings' for appropriate pump
- 1.6.1.2. Open waste valve (left knob with no hole) on pump and click 'Prime'
- 1.6.1.3. Prime for several seconds then **stop pump** and close waste valve
- 1.6.1.4. IF eluent lines have been changed or lines are dry, prime the lines with syringe via right knob with hole before following above procedure. See manual section B.13 for details.

1.6.1.5. Repeat 'Startup'

- 1.6.2. Suppressors are set to turn on 30 seconds after system startup
- 1.6.3. Baseline can be monitored by clicking blue 'Acquisition On/Off' circle icon in tool bar. Monitoring must be stopped before batch analysis can be started

2. Instrument Conditions

2.1. ICS-2000

Pressure ~ 2000 psi Column Temp = 35 °C Cell Temp = 35 °C Suppressor Current (4mm ASRS) = 25 mA Total Conductivity ~ 2 uS Flow Rate = 1 mL/min Sample Loop = 250 uL for low level analysis

2.2. ICS-1000

Pressure ~ 1500 psi Suppressor Current (4mm ASRS) = 25 mA Total Conductivity ~ 20 uS Flow Rate = 1 mL/min Sample Loop = 25 uL for high level analysis N2 head pressure ~ 6-9 PSI

- 3. Sample (Batch) Processing
 - 3.1. Build a Sequence (run)

NOTE: Each sequence requires at least one analytical program (.pgm; hardware method), quantitation file (.qnt; integration parameters), and shutdown program ('Shutdown.pgm' or similar).

- 3.1.1. Select 'New' on toolbar or menu
- 3.1.2. Select 'Sequence Using Wizard'
 - 3.1.2.1. Follow wizard dialogue boxes to select number of samples (unknows, QC's, and one STOP) and standards (calibration levels), and select program and method.
 - 3.1.2.1.1. For routine LTER analysis select 'Simultaneous injection on ASRS w EGC.pgm program and 'lowlevel_WMP_ADP.qnt method

- 3.1.2.2. Confirm each line in sequence is using the correct method and program (pull down menus). These can also be selected from template folders and copied to sequence folder for use in the run
- 3.1.2.3.Copy and paste shutdown.pgm to the sequence folder and assign to STOP sample at end of sequence.
- 3.2. Prepare samples
 - 3.2.1. Fill 5 mL sample vials and cap; caps should be flush with vial for samples and left raised above vial tops for rinses. Start each run with 1-2 rinses and end with a rinse (or a blank)
 - 3.2.2. Fill grey racks with samples and place into autosampler with black dot in lower right corner, grooved side forward
 - 3.2.3. Press 'Run/Hold' on front of autosampler to process rinse samples and flush injector. The autosampler will then hold for an inject signal from Chromeleon.
- 3.3. Check background on instrument panel. If OK, stop manual acquisition (blue dot on tool bar).
- 3.4. Select 'Start/Stop Batch' icon from toolbar or 'Batch → Edit' from menu. Dialog box will appear with current sequence listed. Add sequence if necessary.
 - 3.4.1. Highlight sequence and select 'Ready Check'. Dialog state amount of eluent needed, run time, and data storage space needed for this analysis
- 3.5. If all Ready Check information is acceptable then select 'Start'. Instrument will begin analysis.
- 4. Shutdown
 - 4.1. Shutdown program assigned to STOP sample will stop instrument and shutdown pumps after final analytical sample injection.
 - 4.2. When run is completed remove sample vials from rack. Remove caps and rinse vials with nanopure. Then place vials in nanopure bath for later use.
 - 4.3. Log run in database. If both channels were used remember to count each sample twice.