Ion Chromatography Operating Instructions
Version 2.1
9/1/10

1) If the software is not running, double-click the Chromeleon icon to start it. The software will open to the default panel tabsets for the instrument. There are two separate, complete systems in the ICS-5000. System 1 is configured for anion analyses only, and system 2 is configured for cation analyses only. The currently selected system is highlighted with a red box. Both systems may be run simultaneously.
2) Make sure there is enough eluent for the run(s). The normal flow rate is 1.0 ml/min for the cation column, and 1.2 ml/min for the anion column. Multiply the flow rate by the run time per sample (add 1 minute for auto-sampler functions), and then by the number of samples to determine the amount of eluent needed. Make sure at least 300 ml is left in the eluent bottle at the end of the run. If more eluent is needed, see Appendix 1 for instructions. Anion eluent needs to be fresh. Discard the old eluent and make up a new batch if it is over a week old. Cation eluent is good for several weeks.
3) If the pump pressure is unstable or if fresh eluent was made, the line(s) need to be purged. If the suppressor is on, turn it off at this point. Open the door to the pump unit, open the purge valve on the top right side of the pump, and select "Prime" on the Home tab. When the prime is finished, close the purge valve, turn the pump on or verify that it is already running, and turn the suppressor back on.
4) The installed sample loops are 10 µl. Other sample loops and their approximate ranges are as follows: 5 µl (1-500 ppm), 10 µl (1-250 ppm), 25 µl (1-150 ppm), 100 µl (0.1-50 ppm), and 500 µl (0.01-1 ppm). If another loop is desired, unscrew the 2 nuts holding it in place (make sure the ferrules come out with the nuts) and attach the nuts for the new loop. Run a blank at the beginning of a sequence to check the loop for leaks.
5) A stable baseline needs to be established before running samples. This generally takes about 15-20 minutes. To start flow, click *Startup* for the respective system. The flow rate will increase to 1.0 ml/min and the auto-sampler will flush (this takes 1-2 minutes) before the suppressor current is turned on. After the start-up procedure is complete, click *Control→Acquisition On* from the top drop-down menu to see the on-line plot. Set the y-axis scale to a 2 µS range by right-clicking the mouse in the Detector window and turning off *Auto-Autoscale*, then by choosing *Axis/Decoration→Online Signal Plot Properties*. This scaling is helpful for monitoring the baseline. The *Autozero* button can be clicked, if necessary, to bring the baseline back to zero.
6) If not already done, filter samples through 0.45 µm syringe filters into auto-sampler vials. Load the samples into the auto-sampler rack, in the following order: low concentration to high concentration standards, e-pure water blank, and then samples. Intersperse rinse samples as desired.
7) Go to the sequence window by hitting the "Browser" icon, using the "Window" drop-down menu, or by using <Ctrl-Tab>. Create a sequence file for the samples. This can be done by copying an old sequence file and then saving it to your new

sequence name, or starting from scratch with a new sequence by using the Sequence wizard. To edit areas of the sequence, right-click the mouse and use the menu options or simply type over existing text. Enter sample names and whether they are Standards or Unknowns. Enter the sample positions in the sequence to match the positions of the samples in the auto-sampler rack. Enter the desired injection volume for each sample. Select the desired program for each sample. Commonly used programs are "anion_14_min.pgm" or "cation_28_min.pgm". The 28 minute program is usually for $BaCl_2$-extracted samples. Make sure the last line in the sequence uses the "shutdown" program so the flow and suppressor current shut off when the sequence is finished. Select the desired quant method for each sample. Record sequence information in the logbook.

8) Once the baseline is relatively flat, go to *Control→Acquisition Off* to stop the on-line plot. If this isn't done before starting a sequence, an error message will be generated.
9) To run the sequence, select *Batch→Edit→Add*(select proper sequence)*→Open→Start* on the computer. **Make sure the proper system is selected before doing this**. Once the sequence is running, be careful about opening the door to the auto-sampler. The safety interlock has been defeated by a magnet, so it is advisable not to open the door if the syringe or anything else is moving. *Auto-Autoscale* can be re-selected to watch the on-line plot by right-clicking in the Detector window and selecting it from the pop-up menu. The order of peaks in the anion standard solution is fluoride, chloride, nitrite, bromide, nitrate, phosphate, and sulfate. The order of peaks in the cation standard solution is sodium, ammonia, potassium, magnesium, and calcium.
10) Monitor the first run or two to make sure all of the peaks are eluting before the end of the program. If peaks are being cut off or not eluting at all before the end of the program, stop the batch by selecting *Batch→Stop*. A longer program needs to be selected (or created) to run the samples. Save the sequence as a new sequence (with a new name) to reset any samples that have been run. Modify the new sequence to include the new program and click *Save*. Click *Batch→Edit→*(select the old sequence)*→Remove→Close* to remove the old sequence. Repeat Step 9 to start the new sequence.
11) When the sequence is completed, empty the waste bottle into the sink for anion waste or into the acid waste for cation waste. Open the sequence file and click on the quant method used in the sequence to modify it (or click on the quant method for each sample if multiple quant methods were used). Click *OK* if the pop-up message "Unassigned standards detected" is displayed. In the "Edit Amount Columns" window, click *New*, enter the name of the unassigned standard, and click in the white area to save the name. Repeat this for all of the unassigned standards (listed on the right panel of the window). Next, click *Unassigned* in the left panel of the window, and drag the appropriate standards from the right panel to the proper standard name in the left panel. Click *OK*. If the "Unassigned standards detected" pop-up is not displayed, click the "Amount Table" tab. Right-click and select *Autogenerate Peak Table*.
12) To adjust the start of the baseline in the quant method, click the "Detection" tab, and enter the desired time in the table next to "Inhibit Integration". If all peaks

are not being detected, edit the minimum area. To adjust the retention time(s) and/or retention time window(s), click the "Peak Table" tab and modify the values. Scroll through the standards and samples (by using the scroll icons) to ensure that all of the peaks are being identified properly. Further adjust the integration and retention time parameters, if necessary. To edit individual peaks, delete the peak by right-clicking the mouse and then hitting *Delete Peak*. Click the Peak Tool icon and manually draw a new baseline. Save changes.

13) In either the "Amount Table" or "Peak Table" tab, edit the amount columns to match the standard values and click *Save*. The calibration curve is graphically displayed.
14) To set the calibration so it is not forced through zero, click the "Peak Table" tab. Type "LOff" in the "Cal. Type" column for each ion and click *Save*.
15) To view acquired data, double click on a finished sample in the sequence file. Click the "Summary" tab, right-click and select *Load Report Definition*, hit the up folder icon twice, click *Other Templates*, and select the appropriate template (*28 min cation* will work for all cation analyses and *anion_report* will work for all anion analyses). This template provides a data table with ion concentrations in parts-per-million. If "n.a." is displayed for any tabular data, the detection and/or integration parameters may need to be slightly modified. The data table can be copied and pasted into Excel or other spreadsheet programs, if desired.

Appendix 1

Making Eluent for Anion Analysis

Stock solutions: 0.1 M Na_2CO_3 (5.3 g in 500 ml e-pure water)
0.1 M $NaHCO_3$ (4.2 g in 500 ml e-pure water)

AS22 column
Eluent solution concentrations: 4.5 mM Na_2CO_3, 1.4 mM $NaHCO_3$
Eluent solution preparation: Combine 45 ml 0.1 M Na_2CO_3 and 14 ml 0.1M $NaHCO_3$ and bring to 1 L in e-pure water.

Making Eluent for Cation Analysis

Stock solution: 1.0 M MSA (methanesulfonic acid)

CS12A column
Eluent solution concentration: 20.0 mM MSA
Eluent solution preparation: 20 ml of 1.0 M MSA and 980 ml of e-pure water to make 1 L of eluent.