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LAB GROUP: _____

Spring 2007 Geochemical Characterization of Natural Systems

Dr. J.T. McGuire

LABORATORY 5: Introduction to Capillary Electrophoresis (CE)

The objectives of this week are to familiarize yourself with 1) capabilities of CE; 2) methods of operation; 3) theory of how CE works. These lessons will build upon prior lessons (e.g., proper lab procedures, instrument calibration, error sources, proper use of blanks, etc.) meaning that you should include these elements in your reports (as appropriate) in addition to this week's objectives.

Questions to consider as you write your reports:

Introduction:

1. Briefly, how does a CE work? (How are chemicals separated and detected?) Hint: there may be more than one option while still using CE!
2. In general, what analytes can be quantified using CE?
3. In what media (soil, water, rock, etc.)?
4. What are some advantages and disadvantages of this technique?

Methods:

1. What analytes did you measure?
2. On what types of samples (media)?
3. What standards did you use? Why?
4. Instrument parameters used:
 - a. buffer (electrolyte) solution
 - b. column type and length
 - c. type of injection & duration
 - d. type of detector
 - e. direct or indirect detection
5. Data retrieval techniques

Results:

1. Considering your data table of standards, sample, and field blanks:
 - a. assess the quality of your standard curves
 - b. report the anion concentrations in your samples and blanks
2. Any Irregularities?
3. Sources of error? (DO NOT WRITE HUMAN AND INSTRUMENT =)

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TASKS-Wednesday (class time)

- 1) **Turn on instrument and initialize the system.** This will turn on lamp and prepare instrument.
- 2) **Prepare your column.** The CE works best when the column has been flushed with the buffer solution you will use prior to you analysis (20 minute minimum but days is best).
- 3) **Pipette your sample (+ standards) into CE vials.** Ensure that your sample is well mixed, filtered and that the vial contains NO AIR BUBBLES.
- 4) **Pipette some buffer solution into your “home” and “Flush” vials.** This has been done for you. The home vials contain the buffer that will experience the electrical charge and the flush vial will contain fresh buffer just to rinse in between samples.
- 5) **Pipette Nanopure water into a rinse vial.** This has been done for you.
- 6) **Set Parameters in Method.** We will go through this stage again in lab. You may want to print your method at that time. Record your method and which home vials you used here:

Method name: _____ Home Vials: _____

- 7) Set up sequence table, save sequence parameters and START!!!!
- 8) Your data should be ready to process by lab.

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TASKS-Laboratory

- 1) Ask any questions about method development-print out method used for your samples. This will be a good reference when writing up your report.
- 2) Manual integration of standards, sample, and field blank from your site.
 - a) Open Instrument-Offline
 - b) View→Data Analysis
 - c) Select integrate mode from buttons on right
 - c) Open File→ex. 014-0801 (014=vial location, 08=line#, 01=injection)
*I recommend loading 3 samples at once, if run looks good you need only load the top signal for each sample.
 - d) Manual Integration:
 - 1) select auto-integrate button (fairy wand)
 - 2) delete the auto-integrations with X button
 - 3) load one sample at a time, zoom in and integrate
 - 4) load all
 - e) Print your results (you will have 2 sets of 3):
 - 1) print→ integration results (this will print the data table)
 - 2) print→ selected window (this will print graphs)
 - 3) label your print-outs with samples before you leave
- 3) Using the “spiked” samples, identify your peaks. Are all your peaks accounted for in your standards? If not then can you guess what your mystery peaks might be?
- 4) These data can then be used to plot concentration vs. area to obtain calibration curves for Cl, Br, NO₂, SO₄, NO₃, F, and PO₄ (all in your standards). Remember that the order is always the same even if peaks shift. * Make all 7 calibration curves even if your sample does not contain 7 peaks. You will want to discuss the quality of each curve in your report.
- 5) Calculate the concentrations of your sample(s) using your calibration curves.