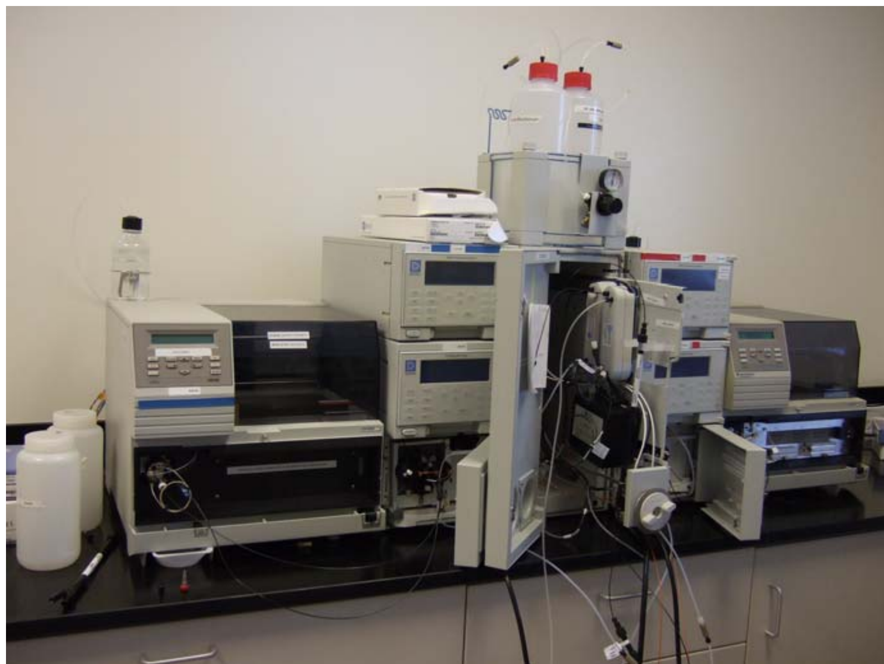
EXPERIMENT #5

Introduction to the Ion Chromatograph: Calibration and Blanks



OVERVIEW

The purpose of this lab is to become familiar with the Dionex DX-500 Ion Chromatograph. This instrument is used to perform the analyses for several other lab exercises (filter samples, impactors, denuders, precipitation sampling). The first step in running the ion chromatograph (IC) is to prepare standards of known concentration and construct a calibration curve. In this exercise each group will prepare standards and their own calibration curve.

EQUIPMENT NEEDED

Lab Notebook, Safety Glasses, Lab Coat
Latex gloves
Ion Chromatograph
Balance
Weighing boats
Teflon spatula
Deionized water from water system
Wash bottle with deionized water
1 l volumetric flask
100 ml volumetric flask
Pipettes and pipette tips
Inorganic Salts: Sodium Chloride, Sodium Nitrate, Sodium Sulfate
Autosampler vials
Plastic bottles for storing standards
Single component standards (provided by teaching assistant)
Mystery Sample (provided by teaching assistant)

PROCEDURE

Preparing a blank sample

You should probably wear latex gloves for any of the procedures where you might contact the samples or standards. While the gloves are not particularly clean, your hands probably have lots of salts on them, especially right after lunch. Wash the gloves with DI water after putting them on.

In order to check that the IC is operating properly, we will run a blank sample and single component standards. The teaching assistant will have started the IC in the morning so it should be ready to work by the lab period.

The autosampler vials are small plastic vials with screw-type caps. The caps have a septum in them. As a group we will prepare one blank sample vial, and one single component standard vial each for chloride, nitrate, and sulfate. Fill each vial with approximately 0.6 ml of the appropriate solution (Nanopure water or a single component standard) and place a cap on it. To avoid confusion label each vial appropriately with a permanent marker.

Preparation of Standards

To save time during this laboratory session, we will only use four standards to prepare a calibration curve. For research analytical work we generally use six or more standards. The standards will contain known amounts of nitrate, sulfate and chloride.

A stock solution is a convenient tool for preparing the standards. A reasonable amount of salt is weighed out for a 1 liter stock solution which is then diluted to the concentrations you need. The concentration of the stock solution depends on what levels you expect to find in your samples. Since you probably don't have a good idea of what these concentrations are right now (and won't have time in this laboratory period to experiment too much), choose a stock solution of :

0.01 Normal Na_2SO_4
0.01 Normal $NaNO_3$
0.005 Normal NaCl

The lowest of the four standards you should prepare for this exercise is 5 μN Cl^- / 10 μN NO_3^- / 10 μN SO_4^{2-}, and the highest should be 10 times those amounts. Choose two intermediate values for your other two standards.

Prepare the stock solution by weighing the amount of salts you need and then adding DI water to fill a 1 liter volumetric flask. You can make the dilutions using a 100 ml volumetric flask and the micropipettes.

Place approximately 0.6 ml of each standard into an autosampler vial. Do the same for the "mystery sample" and a blank sample of Nanopure water. Label each vial appropriately and give them to the TA.

Running the IC

The teaching assistant will show you how to place the autosampler vials into the autosampler. In order to save time, for this lab exercise the teaching assistant will set up the method (instructions for controlling the IC) and the schedule (list of samples the autosampler will inject into the column). Setting these up isn't very difficult, but the software takes some getting used to.

Constructing a calibration curve

Although the Dionex data processing software can prepare calibration curves, for this lab each group will construct their own calibration curves. You need to prepare a calibration curve for each ion in the standards you've prepared.

For each chromatogram, the IC software determines the area under each peak. The teaching assistant will print out a report for each chromatogram. The report shows a picture of the chromatogram and lots of statistics on the peaks, including area.

The calibration curve is then created by constructing a linear regression between the areas of the peaks and the concentrations of the standards. The area is the x-coordinate and the concentration is the y-coordinate.

Once you have the calibration plot and the linear regression between area and concentration, you can then use the equation to find the ion concentrations of any sample from its peak areas. This is the technique needed to find the "mystery sample" concentration.

DISCUSSION/LAB WRITE-UP

Plot the calibration data for your standards and find the equation for the linear regression between concentration and area for each anion. Use the relationship you've found above to calculate the concentrations of your standards (i.e., plug the peak areas for your standards back into the calibration equations to predict concentrations). Are they close to their nominal values?

What were the ion concentrations in the "mystery sample"?

Comment on your calibration curve. Discuss the intercept value. Would more points improve your curve? Discuss the scatter of your standard concentrations about the calibration curve.

What ion concentrations did you obtain from the peak areas found for your blank sample?

Discuss how errors in preparing standards would affect the concentrations of samples you would measure. What if you were making your standards with the 100 ml volumetric flask and you accidentally overfilled the flask on the first standard? Would you be able to get a decent calibration curve if you just overfilled the flask the same amount for the other standards? Would the concentrations you get for samples be accurate?

Suppose an unidentified peak showed up in your chromatograms. How might you decide what this peak was?