FLUORIDE IN WATER

INTRODUCTION

This week you will use spectrometry to measure the fluoride concentration in water samples.

Fluoride is used at very low levels in some municipal water supplies and in toothpastes to help prevent dental decay and as a treatment for osteoporosis. Fluoride ions are colorless (i.e., do not absorb light in the visible region) in aqueous solutions and therefore cannot be measured directly by absorbance. However, if we react the fluoride ions with a highly colored complex of zirconium ions and a dye (represented as **ZrIn)** we can indirectly get an accurate measurement of the fluoride concentration. The following simplified reaction represents the reaction of the ZrIn complex and fluoride ions:

$$\underset{\text{(red)}}{ZrIn} + F^- \rightarrow \underset{\text{(colorless)}}{ZrF} + In^-$$

When fluoride is added to a solution containing ZrIn, it reacts according to the above equation. **This reaction causes the absorbance of the solution to decrease with increasing fluoride concentration**; this is the expected behavior since you are removing the absorbing species by the addition of the fluoride.

THE "**PARTS PER MILLION**" (ppm) UNIT

When we use percent, we are actually working with parts per hundred (parts) and use the % symbol rather than pph.

When we are able to detect fairly small quantities of solutes in solution, it is often more convenient to use parts per million, ppm, rather than percent.

For liquid solutions, ppm is usually based on mass: a 1 ppm solution contains 1 g of solute per 10^6 grams of solution. Or, for dilute aqueous solutions, 1 ppm = 1 mg (solute)/L (solution).

For example, a 20 ppm aqueous NaCl solution contains 20 mg of NaCl per liter of solution.

PROCEDURE

Since the fluoride concentration in our water samples ranges from 0 to 3 ppm, work very carefully when doing this experiment. Also, glassware has to be scrupulously clean. Your results depend on it. Data that are less than 98% correlated will not be accepted.

1. Double click the "Logger Pro" icon and allow the screen to open.

2. The spectrometer needs to be powered for about 5 minutes before using so do this step before preparing your solutions. Do not use the Go!Link with the spectrometer. Plug the spectrometer via provided USB cable to the computer USB port.

3. Load a 25 mL buret with DI water.

4. Use a measuring pipet to deliver the following volumes of **FLUORIDE STOCK SOLUTION (3.0 ppm).** Deliver the exact volumes of stock solution and water shown in the Table below into large test tubes (TT) marked with the letters A through G.

Test Tube	STOCK (3.0 ppm) F^- SOLN (mL)	DI WATER (mL)	F^- concentration (ppm)
A	0	3.0	0
B	0.5	2.5	
C	1.0	2.0	
D	1.5	1.5	
E	2.0	1.0	
F	2.5	0.5	
G	3.0	0.0	3.0

5. The fluoride concentration in each TT after the addition of water should have been determined and entered in the above table as a Pre-Lab exercise.

6. Using a volumetric pipet, deliver 3.0 mL of an unknown fluoride solution into a TT labeled U for the unknown.

7. Go to the COLOR REAGENT DISPENSER. Gently lift and then depress the plunger to deliver the preset volume (5.0 mL) of reagent into each TT. Thoroughly mix the contents of each tube without spilling.

8. Check for cloudiness and proceed only if the solutions are clear.

9. Calibrate the Spectrometer ; Do not unplug the spectrometer during this experiment or you will have to start over.

    a. Return to the Logger Pro screen on the computer. Click: Experiment ; Calibrate ; Spectrometer:1.

    b. Allow the lamp to warm up for 90 seconds as displayed on the computer screen.

c. Only touch the ridged faces of the cuvette, never touch the clear faces. Rinse and fill one cuvette (about ¾ full) with the blank. In this lab the blank is deionized water without any color reagent. **Gently blot** (don't scratch the sides of the cuvette) off any drips on the outside of the cuvette with a Kimwipe.

d. Place the cuvette containing the blank in the spectrometer so that one of the clear sides is aligned with the white arrow at the top of the cuvette slot. Click: "Finish Calibration" ; OK.

10. Select the Wavelength

a. Rinse and fill a cuvette with the most **concentrated** solution (contents of test tube G including color reagent) in the spectrometer. Click: Experiment ; Data Collection ; Full Spectrum ; Done. Close this box by clicking the "x" in the upper right hand corner of the window.

b. Click the small green triangle in the toolbar labeled "Collect". After the line graph appears on the screen, click the small red square in the toolbar labeled "Stop".

11. Generate the Calibration Curve.

a. In the toolbar click: Experiment ; Data Collection ; choose Events with Entry in the Mode box. Highlight the word Event in the Column Name box and replace it with Concentration. Put ppm in the Units box. Clear "Short Name". OK. You are ready to begin collecting data. Remove the cuvette from the spectrometer but keep it handy.

b. Click the rainbow icon labeled "configure spectrometer" located in the toolbar.

c. Scroll and check the box that has a wavelength closest to 550 nm. OK.

d. Rinse and fill a cuvette with the contents of test tube A (deionized water with color reagent). Place the cuvette in the spectrometer. Click the begin data collection button (triangle) in the toolbar labeled "Collect". When the absorbance reading stabilizes, click the KEEP button located in the toolbar just to the right of the Red Stop Button (be careful that you **don't** accidentally click the stop data collection button (square)). Type in the concentration of the solution that is in the cuvette (don't include units). OK.

Pour your samples back into the appropriate TT after you have measured each absorbance and discard them in the waste container only after you have acceptable data.

e. Working in order of most **dilute** to most **concentrated** of the remaining standard solutions (not the unknown), rinse and then fill a cuvette with the solution that will be analyzed. Don't forget you already have a cuvette containing the most concentrated solution. Place the cuvette in the spectrometer. When the absorbance reading stabilizes, click the KEEP button (be careful that you **don't** accidentally click the stop data collection button (square)), and enter the concentration of the analyzed solution. Repeat until the absorbance of each solution has been determined.

f. When the absorbance of all standard solutions (not the unknown) has been measured, click the stop data collection button (square) located in the toolbar.

g. To determine the equation of the line for your calibration curve click: Analyze ; Linear Fit. Click: Analyze ; Autoscale ; Autoscale. You should see a box with the equation and a correlation.

To receive full credit for this lab your calibration curve must be a good, straight-line graph, with a correlation coefficient of 98% or better (Corr: on the screen reads 0.9800 or greater). You should repeat the experiment until you get this proficiency. Work carefully. If you need to repeat the experiment, you still must be done with the write up and post-lab questions before the end of the lab period.

12. Rinse and fill a cuvette with your assigned unknown. Place the cuvette in the spectrometer. Record the absorbance in your lab report once the reading has stabilized.

13. Each lab partner's report must have a Logger Pro generated printout of the calibration curve attached to it. The printout must show the graph, the information needed to generate the equation (slope and intercept) for the line, and the correlation reading. To do this click: File ; Print. Uncheck the "Print Visible Spectrum on Wavelength Graphs" and change the "orientation" to landscape under properties. Be sure that the names of all lab partners are entered in the "Name" section and that the date box is checked.

14. When you are done, return your bin to Lab Services.

DATA AND ANALYSIS SHEET: FLUORIDE IN WATER

Name: _____ Date _____

Lab Partner _____

Wavelength used: _____

Attach the calibration curve (graph), with curve characteristics to this report sheet.

Equation of line for the calibration curve: _____

Correlation: _____

Unknown Identification: _____

Absorbance of your assigned unknown fluoride solution: _____

Determine the concentration of fluoride in your unknown solution using the calibration curve (not the equation). Mark and identify the point on the printout of the calibration curve.

Concentration: _____

Determine the concentration of your unknown using the equation for the line for **your** calibration curve (show work).

Concentration: _____