Acid Mine Drainage Project Experiment
Getting Started

The first day of the Project Lab will be devoted to planning. You will need to plan not only what you will do, but also how you will divide the work for maximum productivity and efficiency. At the end of the first day, you will submit a design proposal. In your proposal, you will need to specify the variable you will investigate and detail the procedures you will use. You may also wish to identify which group members will take the lead role in various aspects of the project. Who is the best at experimental design? Who is the best at theoretical modeling? Who is the best communicator? Who is the best writer that will maintain the primary documentation of the group's work? These people will not have exclusive responsibility for a particular part of the project; rather they can act as the leader for that aspect. All group members are expected be productive, contribute significantly, and understand all parts of the project.

Your design proposal is the starting point for your experiments. Once your experiments are underway, you will likely need to adjust and refine your procedures. Experiments rarely work the first time exactly as planned! Problems occur and new questions arise. You will modify your experiments to solve the problems and/or answer the questions that arise. This process will be repeated multiple times over the course of the project.

Design Proposal

You will be given ~1.5 g of the mineral arsenopyrite with which to conduct your experiments. Keep in mind that you will conduct multiple dissolution reactions during the project. You will take aliquots from your reaction vessels over the course of a week. Initially you will be measuring the iron concentration in these aliquots using spectrophotometry. Later you will also analyze for arsenic and sulfur concentrations. Many of the issues you need to consider while planning your experiments are discussed in the introductory project handout (especially pp. 8-9). Below are some additional issues that you should consider.

1) Your initial sample will not be clean. Metal oxides and elemental sulfur have formed on the surface. You'll need to clean the mineral. How will you do it?

2) Define the conditions in your reaction vessel. That is, consider the amount of sample, the desired pH, and what reagents you need including volumes and concentrations. Take care that any chemicals you add to the vessel do not contain sulfur, since that will most likely swamp out the subtle changes of sulfur concentration that evolve due to the dissolution of the mineral.

3) Define your sampling method including frequency, aliquot size, and how you will store them.

4) You should estimate the concentration of iron you expect to obtain in the reaction vessel. (You did a similar calculation in HW#2 for pyrite.) The results of your calculation will be different depending on the reaction volume you choose and your sample size. Assume a particle size of 50 μm. Consult the research literature to find the density and dissolution rate of arsenopyrite. Will your procedures above give you a measurable amount of Fe in solution? If not, you will need to revise the details in (2) and (3) above.

5) The spectrophotometric measurement of Fe concentration is a separate experiment that you need to define. The details of this experiment should be spelled out similarly to those in the Spec. Fe lab you have already done. But the details are DIFFERENT! You should consider using phenanthroline as the complexing agent. Your concentration ranges will be different and the optimum pH will depend on the complexing agent. Again, you may need to revise the details in (2) and (3) so that you have a measurable amount of iron in solution.

Points will be deducted for unclear or missing procedural components. Your proposal should be terse (1-2 pages), yet with enough detail that someone else could set up your experiment without asking a lot of questions.

Starting Your Experiments

When your proposal is complete you can begin your actual experiments! You should start your first reaction for the dissolution of the mineral. If it is necessary to leave the reaction vessels under a hood or someplace OTHER than your lab drawers, be sure to clearly mark the flasks and experimental area with your names, an email address a phone number, and your TA's name just in case there is an issue you need to be alerted to. Cover your samples with parafilm to minimize evaporation and prevent dust and other contaminants from settling into the solutions.

Once the reaction is underway, you can begin testing and refining your procedure for the spectrophotometric measurement of iron for the specific conditions defined by your procedure. You've already used a positive control in two experiments this semester. A positive control is a sample similar in quantity, matrix, and concentration to the actual unknown you'll be measuring. A positive control has a known concentration, and is used to test the accuracy and precision of the experimental method. Your group should verify the experimental design you've developed with an experimental control.

Why do you need to do this? Even though the TECHNIQUE you'll use is similar to the Spec. Fe Lab, the samples will be significantly different in volume, pH, and speciation. In general, your samples will be much smaller; on the order of a few mL vs. the 25 mL you pipette from the flask in the Spec. Fe Lab. They will be much more acidic too, since the samples you analyzed previously were simply dissolved in water and not initially buffered in any way. Finally, the solution will have a mixture of Fe^{2+} and Fe^{3+}. Your group should consider ways to either oxidize or reduce the sample such that the final analysis is for only one oxidation state of the iron, depending on the complexing agent you decide to use to create the colored complex.

For your design proposal you predicted the concentration of iron that should evolve in the reaction vessel based on a literature value for the rate of oxidative dissolution of arsenopyrite. Use this prediction as a starting point for making a control. You can use any iron salt, such as ferric chloride or ferrous chloride, to make a stock solution of iron. Then from this stock solution, you can use aliquots to add to a buffered matrix, similar in composition to the solution covering the arsenopyrite samples. Make sure your final solution is approximately the same pH and similar in concentration to that which you predicted in your design proposal. Using the same sample volumes you plan to use for the real analysis, try to develop good color as you complex the control sample with the complexing agent. If you use phenanthroline, make sure you let the complex "age" for about 20 minutes before taking a transmittance measurement. If you don't see color when you develop the complex for the control, consider the following:

- Is the final pH correctly chosen for the complexing agent?
- Was the concentration of Fe calculated correctly? (Some students forget to convert to moles or ppm of Fe vs. of reagent).
- Are you using the correct units?
- Did you add enough complexing agent to form the complex? (It should be present in excess in solution.)
- Could there be an interfering anion that is tying up iron, preventing it from complexing with the intended agent in solution?

Be sure to keep good records on the development of the experimental control and how it was used to verify the experimental procedure. In the next progress report, your group will be asked to include the analysis of your control along with a calibration plot and preliminary results from your samples.