Notes on Procedure for Experiment No. 21

We will modify the preparation of the solutions in order to speed the preparation and to decrease the potential for mistakes. The unknowns will be in the form of solutions.

A partial list of the glassware you will use:
- A 5 mL volumetric pipet (for use with the unknown solution only)
- A 5 mL graduated pipet (for use with the known iron solution, 40 ppm, only)
- A 1 mL graduated pipet (for use with the hydroquinone solution only)
- A 2 mL graduated pipet (for use with the o-phenanthroline solution only)
- Two Pasteur pipets (one for citrate and the second for other uses)
- Eight 50 mL volumetric flasks

You first need to determine the number of drops (#) of sodium citrate to buffer the solutions to a pH of ~3.5. Using the volumetric pipet, transfer 5 mL of your unknown into a small beaker. Get about 5 mL of sodium citrate solution and about a 3 to 4-inch strip of pH paper. Use the first Pasteur pipet to add the citrate solution dropwise to the iron solution. Hold the pipet completely vertical so that the drops will be of consistent size. If the pipet is tilted, the drops will be different sizes. Start by adding 3 or 4 drops each time; mix and place a drop on the pH paper using the glass stirring rod. Repeat. As you approach a pH of 3.5, add fewer drops each time.

Complete the table on page 450 of the lab manual. Add a line for the unknown solution. Fill in the third column with the number of drops of citrate. For Known 1, Blank, and Unknown, use the number (#) of drops that you counted. Known 2 will be 4/5 of # (or # x 0.8), Known 3: 3/5 of # (or # x 0.6), Known 4: 2/5 of # (or # x 0.4), Known 5: 1.5/5 of # (or # x 0.3), and Known 6: 1/5 of # (or # x 0.2). For the second column in the table (vol of Fe²⁺), the blank will get no iron solution of either kind, and for the unknown solution, you will use 5 mL of your unknown sample (with no 40 ppm solution). The unknown will use 1.0 mL of hydroquinone and 1.5 mL of o-phenanthroline.

Get eight 50 mL volumetric flasks. Rinse them with distilled water using the plastic distilled water bottle at your desk. Get each solution as you need it. About 20 mL of the known iron solution (40.00 ppm), 8 to 10 mL of hydroquinone, and 15 mL of o-phenanthroline.

Pipet the appropriate iron solution into each flask; count the appropriate number of drops of sodium citrate into each flask; pipet the hydroquinone solution into each flask, and finally add o-phenanthroline to each flask. (Because the color of the final solution in all of the flasks will look very similar, a technique to help keep track of which flasks have been completed is to line up the flasks on the bench and then move each one back an inch or two after you have finished adding all solutions to it. You will then have an indication of which ones you have completed). Finally, fill each flask close to the graduation line with distilled water and finish accurately using the second Pasteur pipet. Mix by slowly inverting each flask at least 10 times.

Get 8 cuvettes and a holder. Rinse each cuvette with the appropriate solution by using the second Pasteur pipet to fill the cuvette with the solution. Then dump into your waste beaker and refill. Repeat with the next solution and cuvette. Note that you are rinsing not only the cuvette but also the pipet. There is no reason to use more than one pipet for this. When you have filled each of the eight cuvettes with one of the solutions, take them to the UV-VIS spectrophotometers and collect your data. If you do not remember the procedure for using these instruments from last semester, check with your T.A.

Finally, dump the used solutions into the waste and rinse the flasks and cuvettes with distilled water. Do not use a brush to clean the cuvettes because it may scratch them. Do not use soap to clean the volumetric flasks. Return all items to the places where you got them.