Chem 122 **Instrumental Analysis** **Fall 2010**

Laboratory #1
Estimation of Iron in a Water Sample

Your first laboratory assignment is to estimate the concentration of iron in a water sample that will be provided. Iron occurs naturally in water in the environment. Water sitting in iron pipes or containers also collects iron. The aqueous and redox chemistry of iron have been well studied. You must familiarize yourself with the dependence of iron species in aqueous medium with respect to pH. You also should study the bioinorganic importance of iron as a micronutrient for microorganisms and therefore the control of microorganism growth in water by the availability of iron.

The goal of this experiment is to understand the reaction between a ligand and the Fe^{2+} ion and investigate by spectrophotometry. The divalent oxidation state of iron is required for this reaction; thus a reductant must also be used. The complexation reaction leads to a colored solution. First, determine the molar absorptivity of the complex. The concentration of iron in the unknown water sample may then be calculated using Beer's law.

Scrutiny of the coordination chemistry of iron will further indicate the principles behind this experiment. This experiment will also allow you to become familiar with the spectrophotometer and the principles of spectrophotometry. The class lectures will give you insight into the methods of handling your data and the TAs will assist you with the experiment. You should try to understand both the merits and limitations of the technique. Also determine the lower detection limit of this method by diluting your samples volumetrically. These experiments should be completed during the week of September 27th (see schedule handout).

Before you begin Laboratory #2, you will need to gather more insight into the technique of spectrophotometry. The second handout will have directions in this area.

You must attend the first lab (Sections A and B: Monday, September 27th; Sections C and D: Tuesday, September 28th) for lab check-in and safety training, and find a lab partner. The written report for this experiment is due Friday, October 8th at noon. E-mail your report (individual, not in pairs; Word or RTF format) to your TA, including figures. Details of the requirements and expectations for the report will be given during lecture. Before beginning this experiment, you will first be checked by the TA for the following:

(i) A one paragraph abstract in your lab notebook. Include the overall goals, method, techniques, calculations and toxicity of all reagents (see MSDS link on the 122 website).

(ii) Goggles and proper attire are required at all times in the lab.

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Laboratory #1 Handout

This lab studies the determination of iron in an unknown solution by colorimetry using 1,10-phenanthroline.

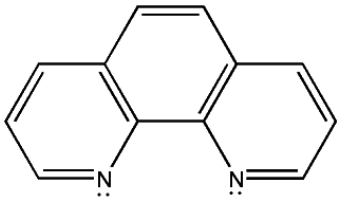
The following stock solutions have been prepared by the stockroom:

Mohr's Salt $(NH_4)_2Fe^{2+}(SO_4)_2 \cdot 6H_2O$ FW 392.14
0.380 g brought to 2000 mL vol. flask with MilliQ water with 5.0 mL conc. Sulfuric acid.
4.85×10^{-4} M

Sodium Acetate CH_3CO_2Na FW 82.03
200. g brought to 2000 mL vol. flask with MilliQ water. 1.22 M

Hydroxylamine hydrochloride $NH_2OH \cdot HCl$ FW 69.49
20.7 g brought to 200 mL vol. flask with MilliQ water. 1.49 M

1,10 Phenanthroline $C_{12}H_8N_2 \cdot H_2O$ FW 198.23
1.015 g dissolved in 50 mL ethanol, then brought to 1000 mL vol. flask with MilliQ water.
5.12×10^{-3} M



Instructions for preparing samples and standard curve:

Prepare all samples and standards in 100 mL volumetric flasks with MilliQ (ultra-pure) water. For standards, add to each flask 10 mL acetate, 1 mL hydroxylamine, 5 mL 1,10-phenanthroline, and 0.5 to 10 mL Mohr's salt. Fill to the line with water. Prepare three to five standards and also prepare a blank with all reagents except Mohr's salt. For the unknown sample, use all reagents except Mohr's salt. You will need to determine the amount of unknown solution to add in order for the absorbance to fall within the range of your standards. Once you have found that amount, prepare three identical unknown samples. These will be used to determine the unknown concentration and error. When calculating the unknown concentration, don't forget to account for the dilution factor. When all samples have been measured and the data recorded, you're done. Discard stock solutions in the waste container. Samples and standards can be discarded in the sink.