Photometric Determination of Fe(II) using Flow Injection Analysis
2005

In this experiment you will determine the concentration of Fe(II) in an unknown solution using flow injection analysis (FIA) with photometric detection. Iron is a ubiquitous compound found in reactively high concentrations in many lakes, rivers, and water systems. In water containing dissolved oxygen, Fe(III) is the thermodynamically stable form of iron. However, significant concentrations of Fe(II) are observed in many natural waters due to photochemical reduction of Fe(II) to Fe(III). In order to investigate iron reduction, a rapid analytical technique for Fe(II) analysis is desirable. The ligand Ferrozine forms very stable colored complex with Fe(II) but not Fe(III). If several solutions containing increasing concentrations of Fe(II) are reacted with excess ferrozine, a standard curve can be generated relating solution absorbance to concentration (Abs = εb[Fe(II)]. The concentration of Fe(II) in unknown samples can be determined from the absorbance of the unknown solution and the standard curve. The concentration of Fe(III) in the sample can also be determined if a reducing reagent is added to reduce all the Fe(III) to Fe(II).

In the classical procedure, reagents are added to each sample, allowed to react completely, and the absorbance is measured. In the FIA method, the sample is injected into a flow stream of reagent which is then pumped through a photometer. The photometer measures the absorbance of the solution. In FIA, it is not necessary that the reaction between the sample and reagents goes to completion, only that the reaction time is constant for all samples and standards. Under these conditions, the amount of colored complex formed will be proportional the amount of Fe(II) in the sample. Figure 1 is a diagram of the FIA system that we will use in this experiment.

Figure 1.0  FIA system for Fe(II) Analysis.
Procedure:

The FIA system shown in figure 1 will be assembled for you. Carefully check all the tubing and fittings of the system to make sure they are properly connected. The instructor will review with you the operation of the spectrometer.

Standard Solution Preparation:

You will be given a stock solution of Fe(II). From the stock solution prepare four Fe(II) standard solutions with concentrations of $3 \times 10^{-6}$, $1 \times 10^{-5}$, $2 \times 10^{-5}$, and $4 \times 10^{-5}$ by diluting the stock with the phosphate buffer at pH 6. The reagent solution is $1 \times 10^{-4}$ M Ferrozine in phosphate buffer at pH 6. The carrier solution is pure water.

You will also be given an unknown solution in a 100 ml flask. Bring this solution to the mark with phosphate buffer and mix well.

Operating Procedure

Turn on the spectrometer and let it warm up for at least 15 minutes. Blank the instrument using a pure water sample. Record the instrument response to each solution. (Do triplicate injections of each solution.) To fill the sample loop with a sample or standard solution set the injector lever in the "load" (upright) position. Put the sample inlet line into the appropriate solution and let the pump draw up solution for about 10 seconds. This will flush out traces of any previous sample solution and fill the sample loop. Then push the injector lever to the "inject" position.

Be sure to note whether the instrument response is in %T or absorbance units. If it is in %T you will need to convert to absorbance before plotting your calibration curve.

Report

Turn in a brief summary of the experimental procedure, your data, sample calculations, your calibration curve, and a statement of the concentration of Fe(II) determined to be in the unknown sample and an estimate of the error in the determination. Discuss any peculiarities. Include a photocopy of at least one example of the instrument response to a sample or standard solution injection.