

CH 424 Instrumental Analysis Lab

Determination of Iron by Atomic Absorption Spectroscopy (AAS)

Introduction

Environmental concerns along with a company's desire for sensitive quality control create the need for quick, accurate, and sensitive methods of detecting small quantities of elements in samples. AAS has become one of the most widely used methods for detection of parts per billion (ppb) concentrations of analytes in aqueous or other solutions. This technique involves measurement of the absorption of discrete wavelengths from a line source, such as a hollow cathode lamp, by ground-state atoms in the gas phase. The technique of AAS can be sub-divided into four categories, namely flame, furnace (electrothermal), vapor generation, and glow discharge AA. Each type of atomizers used in AAS has unique features and provides distinct advantages for certain applications.

Furnace (electrothermal) AA is one of the most sensitive techniques for metal analysis. In this experiment, you will use the furnace AAS in Dr. Thrasher's lab to analyze iron in an aqueous sample. The furnace causes the sample to be atomized off a sample platform inside a graphite tube. The element to be studied absorbs wavelengths of light generated by a specific hollow cathode lamp.

Procedure (at all times follow the instructions of TA/operator)

- 1) Adjust the flow of the inert gas (in this case Argon) to 40-60 psi, and the water to above 10 psi. The water is for cooling the instrument. The graphite furnace is always on; do not otherwise adjust it unless instructed to do so.
- 2) Choose the correct lamp for the analysis that you are to about to perform. Each element has its own lamp which emits light in the wavelength that it absorbs.
- 3) Align the lamp. This many times requires the use of a dental mirror. Once it is aligned sufficiently enough to get a reasonable signal to the computer you may stop and begin to calibrate the injection using the needle.
- 4) Calibrate the injection. In a fully automated sample loader you need only prepare a minimal number of solutions and the computer-controlled device can do the proper dilutions for a standard curve. However the injection arm must be aligned such that it goes the proper distance into the sample and injects at the correct distance from the sample platform in the graphite furnace. This can be checked visually with the aid of the dental mirror.

- 5) Next you will need to align the lamp with the aid of the computer.
 - 5.1 Under the windows menu choose align lamps.
 - 5.2 Choose the lamp that you wish to align.
 - 5.3 Make sure the voltage and other setting are correct by cross-checking the settings with those of the lamp information material (box label).
 - 5.4 After you have aligned the lamp with the power bar you may then close out of this dialog box.
 - 5.5 Next go to the window menu and choose element parameters, then either select the file or make a new one. This file will hold the information about your experiment, concentrations of your standard plot points and the location of each, etc. It also will control the order in which you take sample, diluent, and matrix modifier.

- 6) Go to the window menu to the ID weight parameter. This is where you will name your samples. Normally we will not worry about the actual weight of the sample because the solutions will be so dilute that they will have the same density as water.

- 7) Next click on Auto. This will give you a menu through which you may calibrate the instrument and then run your samples.

Wet Procedure

- 1) The Iron standard solution was ordered from Fisher Scientific, and was carefully calibrated to 1000 ppm. This is far more concentrated than this instrument will accurately detect. Therefore dilutions must be made.

- 2) I'll not go into the exact procedure for the dilutions. They will need to be diluted down to the 1-10 ppb range. These dilutions should be done in 2% Nitric Acid. The acidic nature of the solution helps to keep the Iron in solution. Make your own standards for the range 2.5-50 ppb. The instrument will also make these standards automatically. Compare the results.

- 3) Solutions should be mixed completely with a magnetic stirrer. This is to insure that the solution is homogenous.

Write full laboratory reports, discussing the iron analysis results obtained by this method and that of the UV-Vis experiments for a given unknown sample. This means that for these two experiments (AAS and UV-Vis for iron) you will prepare **one** full length report, comparing and contrasting the two methods. The paper should be in a format suitable for publication in the scientific literature. Chose any style you wish (follow the editor's submission guidelines associated with the chemistry journal of your choice). Note that the ACS publishes a complete *Style Guide*. You should tell me which journal style you are following.