

Chemistry 141 Laboratory
Spectrometric Determination of Iron
Experiment Lecture Notes
Dr. Abrash

- I. Background
- a. What is the purpose of this experiment?
 - i. We're going to learn another way to quantify the amount of material in an unknown sample, to add to our arsenal of gravimetric determination (weighing) and titration. This new technique is called spectrometry.
 - b. What is Spectrometry?
 - i. Spectrometry is a technique that uses the amount of light absorbed by a substance to determine its concentration. In this experiment we will be using the amount of visible light absorbed by an iron complex as a way of determining the amount of iron in an unknown.
 - c. What do we actually measure in spectrometry?
 - i. We measure two characteristics of the light that's absorbed. One is called the wavelength of the light absorbed. The wavelength of the light is the distance in nanometers (10^{-9} meters) between the peaks of the waves.

The second is called the intensity of the light absorbed. The intensity of the light absorbed is simply how much of the light is absorbed.

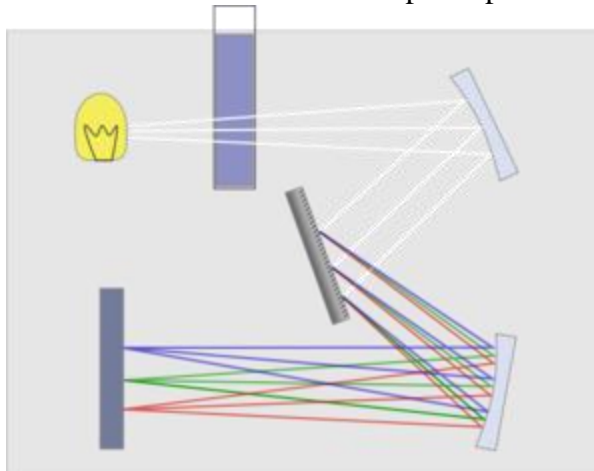
- d. What is visible light?
 - i. Visible light is that portion of the electromagnetic spectrum that we can see. Other portions include the infrared (longer wavelengths than red light), and ultraviolet (shorter wavelengths than violet light), which are invisible. Visible light has wavelengths between about 650 nm and about 420 nm. Visible light when passed through a prism or bounced from a grating can be broken down into its component colors, the colors of the rainbow. The order of these colors from longest to shortest wavelength is given by the mnemonic device

ROY G BIV

for Red Orange Yellow Green Blue Indigo Violet. One of your tasks today will be to assign wavelength ranges for each of these visible colors.

- e. How are frequency and intensity measured?
 - i. With a device called a spectrophotometer. A spectrophotometer has the following components
 1. A light source.

2. A light dispersion device (a prism or grating)
3. A pair of slits (to select among the dispersed wavelengths)
4. A sample cell
5. A detector of some sort.
6. Here's a crude schematic of a spectrophotometer:



- ii. How does a spectrometer work.
 1. It works by comparing the amount of light detected when the sample is there (I) with the amount of light detected when the sample is not there (I^0). A small computer within the spectrometer then converts this to either Transmittance (%T) or absorbance (A).

$$\%T \text{ is defined as } \%T = \frac{I}{I^0} \times 100\%$$

$$A \text{ is defined as } A = \log\left(\frac{I^0}{I}\right).$$

- f. How does this help us find the concentration of our unknown?
 - i. concentration is related to absorbance by the Beer-Lambert law:

$$A = \epsilon bc$$

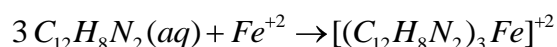
In this equation, c is the concentration of the substance in question, b is the path length of the sample, usually set to one cm, and ϵ is a number called the extinction coefficient, which depends on wavelength, and says how strongly a molecule absorbs at that wavelength.

Therefore at a given wavelength, the higher the absorbance, the higher the concentration.

- g. How do we decide what wavelength to use?
 - i. We take a spectrum, and choose the wavelength where the absorbance is highest, called λ_{\max} .

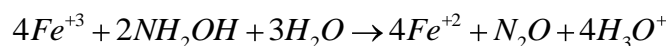
A spectrum is a graph of wavelength vs absorbance or wavelength vs transmittance. We will measure both absorbance and transmittance for all parts of the experiment.

- h. What do we do if we don't know the extinction coefficient ϵ ?
 - i. We create a standardization curve, by measuring the absorbance of several solutions of known concentration, and then plotting them on a graph. We then measure the absorbance of our unknown, and by comparing to our standard curve, determine the concentration.
- i. What if our unknown is colorless (has no visible absorption)?
 - i. Then we mix it with a substance that will react with it in such a way that the product is both soluble and colored. Such a substance is called a complex. In this experiment, the complex is formed between Fe^{+2} and an organic molecule called ortho-phenanthroline, $C_{12}H_8N_2$. The reaction between them is:



The resulting solution has a red-orange color.

- j. The reaction you just wrote is for Fe^{+2} only, but iron can be either Fe^{+2} or Fe^{+3} , and we're supposed to measure the total iron? How do we deal with this problem?
 - i. The problem is resolved by putting a solution in your reaction mixture that ensures that all the iron is reduced to Fe^{+2} . In this case we use, hydroxylamine hydrochloride. The balanced reaction is



II. Experimental Tips

- a. Make your solutions for parts II and III first. This will allow them to develop while you're doing other parts of the experiment.
- b. Measure your solutions directly into your volumetric flasks.
- c. While the color is developing do the chalk experiment. **DO NOT THROW THE CHALK AWAY.**
- d. After the chalk experiment, determine λ_{max} for one of your standard solutions. It is not necessary to wait the full 30 minutes before doing this. To form your spectrum, take measurements of absorbance and % transmittance every 20 nm beginning with 380 nm and going to 640 nm.
- e. Collect data for your standard curve after setting your wavelength to λ_{max}
- f. When doing your write up, you **MUST** create the graphs for your standardization curves on your own.

III. Lab Report

- a. For your lab report you need to turn in the following:
 - i. Carbon copies of your lab notebook pages (today)
 - ii. Your prelab (today)

- iii. A color line for your data in part b (described on p. 44)
- iv. A plot of absorbance vs. wavelength for the data in part C. From this determine λ_{max} and enter in on your report sheet.
- v. A calibration curve for your standard solutions. Include the equation for the line and your R^2 value.
- vi. A determination of the ferrous concentration in your unknown. Calculate your average absorbance, and use this average with your calibration curve to determine the concentration of the ferrous. Record this on your Data Report sheet.
- vii. Use the concentration above, along with the initial volume of the unknown iron solution you pipetted into the volumetric flask to determine the initial concentration of iron in your unknown.
- viii. Sample calculations.
- ix. Brief comments on the accuracy of the data you used in determining your calibration curve and any sources of error you encountered in the experiment. Also comment on whether your data in D are consistent with Beer's law. Why or why not?

IV. Honor Stuff

- a. All experimental work will be collaborative. You can do all work involving your standard solutions together, including the plotting and regression analysis on the calibration curve.
- b. Each of you will have a different unknown, so you'll have to do the calculation of the ferrous concentration in your unknown, and the original iron concentration in your unknown individually. The discussion questions in section viii on page 45 need to be answered individually. Finally, the color lines should be done individually.