

SPECTROPHOTOMETRIC DETERMINATION OF IRON

In this experiment you will determine trace amounts of iron using spectrophotometric methods.

BACKGROUND

In solution ferrous iron combines with 2,2' bipyridyl to form an intensely red-colored complex which has a broad absorption band with a maximum absorbance at 522 nm (Fig. 1 and Fig. 2). Moss and Mellon¹ have shown that the color is developed rapidly and is very stable in the pH 3-5 range. In fact colored solutions prepared under these conditions have been kept for up to three years without any noticeable change in intensity. For a set range of concentrations, Beer's Law will work for this system, and therefore visible spectroscopy is an easy, cheap and effective means to perform a quantitative analysis of Fe.

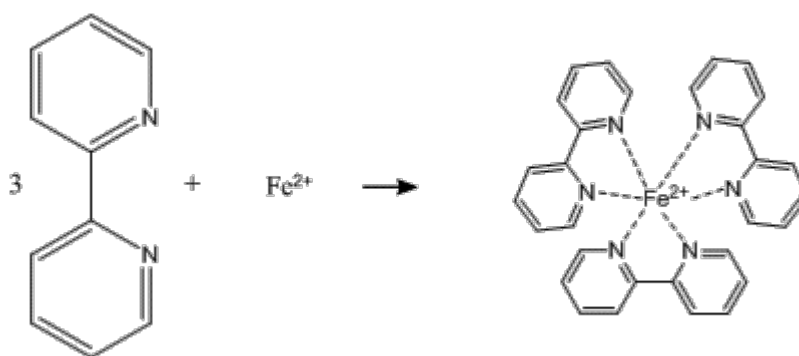


Figure 1. Structure of 2,2' bipyridyl and its complex with ferrous iron

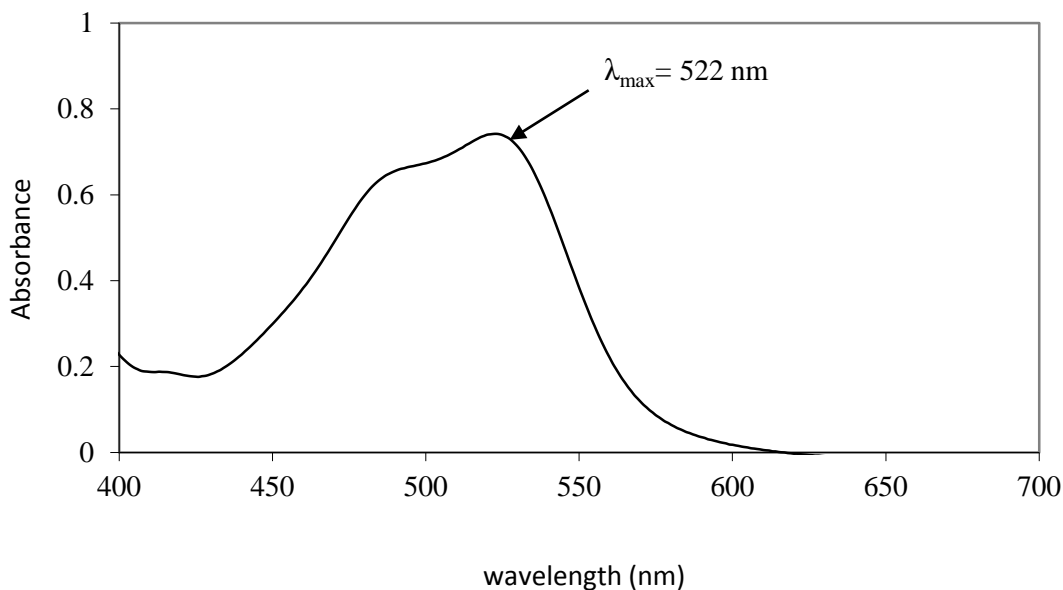
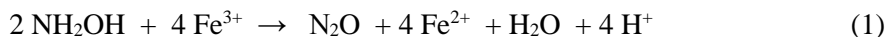


Figure 2. Absorbance spectra for ferrous iron complex with 2,2' bipyridyl.

Fe(II) reduces easily in water to Fe(III). To reduce any Fe (III) back to Fe (II) we add a reducing agent to the solution. Of several reducing agents suitable for this purpose, hydroxylamine hydrochloride has been found to be very effective (Eq. 1).



The success of this method of analysis depends on proper buffering of the solution, presence of an effective reducing agent, and precise laboratory techniques.

EXPERIMENTAL

Equipment Needed:

5, 10, 20, 25, 50 mL pipets
 10 - 100 mL volumetric flasks
 1000 mL volumetric flask
 500 mL volumetric flask
 9 - plastic storage bottles.

1. 100 mg/L Iron Solution. In a 400 mL beaker add 5 mL of 6 M HCL to about 250 mL of water. Dissolve 0.7022 g of ferrous ammonium sulfate ($\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6 \text{H}_2\text{O}$) in in the acidified water. Transfer to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix well.
2. 10 mg/L Iron Solution. Pipet 50 mL of 100 mg/L iron solution into a 500 mL volumetric flask. Dilute to mark with deionized water and mix well.
3. Unknown Solution. Turn in a clean 10 oz. plastic bottle labeled with your name to your laboratory instructor. When you receive your unknown solution quantitatively transfer to a 100 mL volumetric flask, dilute to the mark with deionized water and mix well. **DO THE QUANTITATIVE TRANSFER FIRST.** The resulting 100 mL solution serves as the “prepared sample” in Table 1.
4. Preparation of Standards, Blank and Sample. Label nine 100 mL volumetric flasks according to the following scheme (0.5 mg/L, 1.0 mg/L blank, sample). Prepare the nine solutions following the block outline in Table 1.

Reagents (mL)	0.5 mg/L	1.0 mg/L	2.0 mg/L	3.0 mg/L	4.0 mg/L	5.0 mg/L	7.0 mg/L	Blank	Prepared Sample
Unknown solution	-	-	-	-	-	-	-	-	25
10 mg/L Fe^{2+}	5	10	20	30	40	50	70	-	-
1 M HCl	1	1	1	1	1	1	1	1	1
1% Hydroxylamine	5	5	5	5	5	5	5	5	5
1 M Sodium Acetate	2	2	2	2	2	2	2	2	2
0.1% Bipyridyl	10	10	10	10	10	10	10	10	10
Water	Dilute to the mark.								

Table 1. Preparation of ferrous standards, blank and sample.

BE SURE TO MIX WELL. Visually examine the solutions and place the sample solution next to the standard which appears to have the same intensity of color as the sample.

5. Transmittance Measurements. If you have not previously used a spectrophotometer consult the operating instructions and your laboratory instructor about the operation of the instrument assigned to you.

Rinse a 1 cm square cuvet several times with small portions of the blank and then fill 3/4 full. Set the wavelength at 522 nm. Using the blank solution calibrate the instrument to 100% T. Discard the blank solution and refill the SAME cuvet with the standard or sample solution. Be sure to insert the cuvet in the same orientation as you did the blank. Record your readings in %T, which will be displayed on the LabQuest readout.

Measure transmittance as much as you can in the order of low to high concentrations. Do this by starting with the 0.5 mg/L solution. Rinse the cell several times with 0.5 mg/L solution. Then fill 3/4 full and measure the %T of the solution. Discard the solution in the cell and then rinse several times with small portions of the next (e.g. 1 mg/L) solution, then fill 3/4 full and measure the %T of the solution. Repeat this procedure for the other standard and SAMPLE solutions proceeding from the lower to higher concentrations. Be sure to include the SAMPLE solution. Re-blank your spectrometer several times to check the 100% T setting is retained.

Repeat the transmittance measurements for all eight colored solutions again, starting with the lowest (0.5 mg/L) and proceeding to the next higher concentration.

RESULTS/CALCULATIONS

Hand in the completed answer sheet and carbonless copies of the notebook pages to you TA. Be sure your calculations include the following:

1. Determine the average of the %T values for each of the solutions measured. Then convert these average %T values to absorbance (A) values.
2. Prepare a working curve of average absorbance (A) against exact concentration of iron. Make sure to include the 0.0 A point from the blank you prepared. Include this plot with materials you hand in to your TA.
3. Using the working curve, determine the concentration (mg/L Fe) of the UNKNOWN solution you prepared in step 3.

REFERENCES

- (1) *Moss and Mellon, Ind. Eng. Chem., Anal. Ed., 14, 862 (1942).*
- (2) *R. A. Day, Jr., and A. L. Underwood, pp. 383 392; 396 399.*
- (3) *R. W. Ramette, pp. 138 140; 145 151.*
- (4) *D. A. Skoog and D. M. West, Chap. 20; pp. 539 546.*

Spectrophometric Determination of Iron

Name _____ Course _____ Section _____ Date _____

Mass of ferrous ammonium sulfate _____
Concentration of stock iron solution _____

	Concentration of Iron (in mg/L)	Average Absorbance
Standard 1		
Standard 2		
Standard 3		
Standard 4		
Standard 5		
Standard 6		
Standard 7		
Sample	-----	

Concentration of iron in complex/diluted sample (mg/L) _____

Concentration of iron in original 100 mL solution before dilution (mg/L) _____