

Experiment 7: Adsorption Spectroscopy I, Determination of Iron with 1,10-Phenanthroline

Procedure

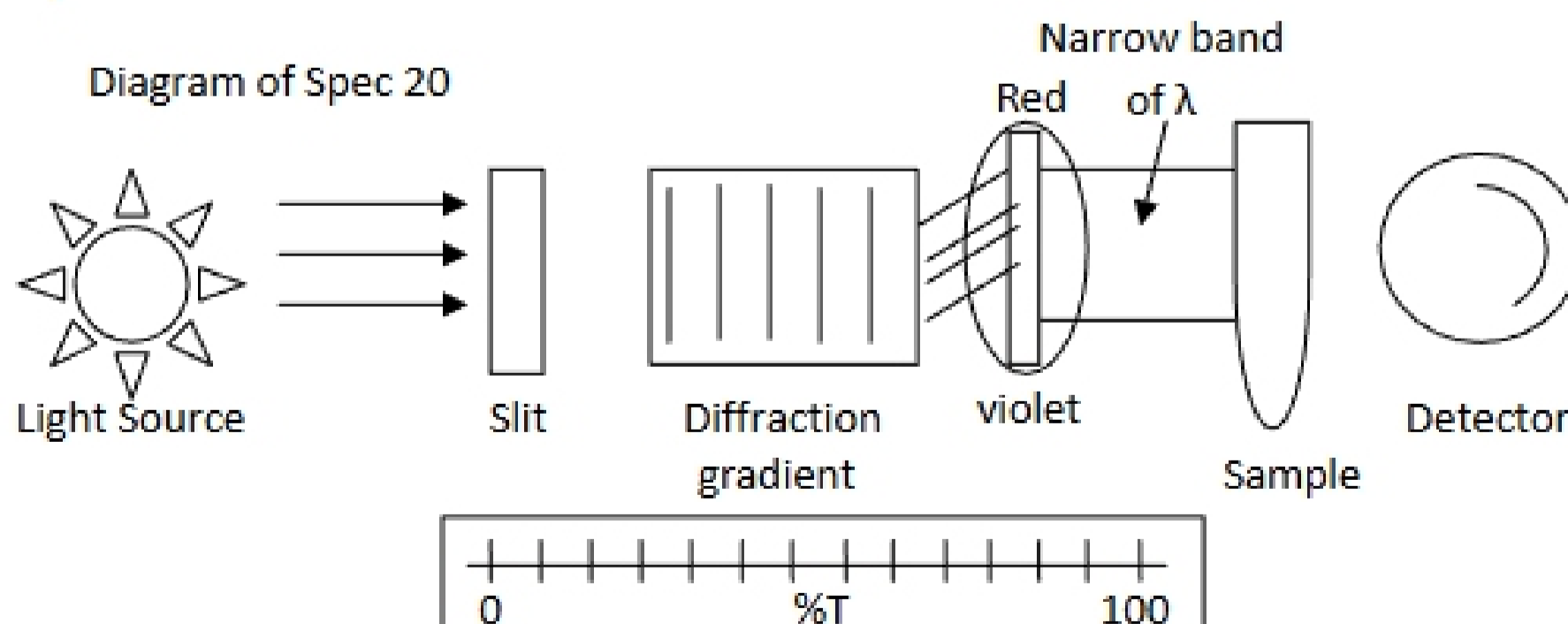
In this experiment, we aim to determine the Na concentration of an unknown sample by measuring an emission intensity of unknowns and comparing the measurements to a calibration curve created from solutions of known concentrations.

First, turn in a 100mL volumetric flask to TA for unknown at least one day before doing the experiment. Weigh out about 0.07g of ferrous ammonium sulfate, dissolve in water, and transfer to a 1000mL volumetric flask. Add 2.5mL of concentrated sulfuric acid and dilute to the mark. Now, prepare the standards. Using a pipet place 1, 5, 10, 20, and 35ml of standard iron solution into five 100mL volumetric flasks. Also, place 50mL of H₂O in a sixth flask (for blank). Treat all samples (including unknown) in the following manner: pipet 1mL of hydroxylamine solution, 10mL of 1,10-phenanthroline solution and 8mL of sodium acetate solution into each flask and dilute to the mark. Mix and allow to stand for 10 min. Now, on the Spec 20 set the %T to zero with no cuvette in the compartment, using the disk on the left front. Set the %T to 100 using the right front dial, with the blank in the compartment. Recheck these settings frequently. Set the wavelength at 505nm (instructed to do so by Shawn) and record the %T within 0.2% for each of the standards and the usual unknown. Calculate the absorbance using $A = -\log(\%T/100)$ and the concentration for the standard. Plot a graph of absorbance vs. concentration of the standards using Tablecurve or Excel.

For detailed procedure refer to: Handout, Experiment 7, Absorption Spectroscopy I: Determination of Iron with 1,10-phenanthroline.

For further information on adsorption spectroscopy refer to:
Skoog, Holler, and West, Chapters 20,21, and 22. Pp. 383-439.

Theory



In preparing the standards and the unknown the reaction of the ferrous iron with 1,10-phenanthroline yielded a red complex that served as a good sensitive method for determining iron:



The molar absorptivity of this complex is 11,100 at 508nm. Prior to the development of the red-orange color, hydroxylamine is added as a reducing agent to put the iron in the ferrous state:



The sodium acetate is added to adjust the pH to between 6 and 9. The color intensity remains relatively constant between pH 2 and 9.

During the analysis of the samples, the spec 20 is calibrated by using the blank sample which contained all species except the iron solution. By doing this, we hoped to minimize the effects caused by reflection, absorption, etc. due to the presence of the other elements (ie, the cuvette and solvent). Failure to use a blank would have caused inaccurate % transmittance readings. Now, by placing the subsequent standards in the spec 20, the decreased %T was due only to the presence of the iron solution. In passing through the cuvette the power of the light beam is decreased as a result of the interactions between the photons and absorbing particles (the iron solution). The %T reading on the spec 20 results from the following ratio:

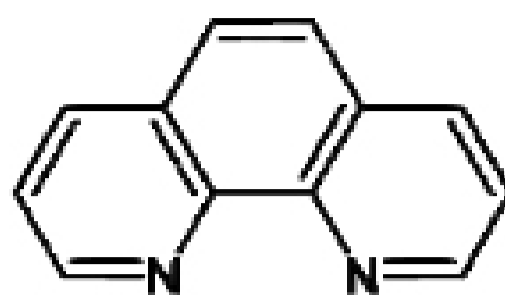
$$\%T = \frac{P}{P_0} \times 100\%$$

Where P is the power of the beam prior to entering the cuvette and P_0 is the power after passing through. The absorbance was calculated from the %T using the equation:

$$A = -\log\left(\frac{\%T}{100}\right)$$

It was not read from the scale on the Spec 20. Beer's law states that "absorbance is linearly related to the generation of the absorbing species, c , and the path length, b , of the radiation in the absorbing medium." (Skoog, 405). In my graph of absorbance vs. concentration, I used Tablecurve. In asking it to draw a curve that best fit my data points, it produced an exponential curve as its first choice, which obviously is not a linear graph. I went and showed the graph to Dr. Holler (After Shawn told me to get some assistance in making it a linear curve). He, at first, started to help me redo the graph. Then, he thought since the exponential graph was in fact the best fit, he said solve for c and use it. So, technically my graph is not linear, which would suggest it did not obey Beer's Law. However the curve to the exponential is very slight, so essentially Beer's Law was obeyed.

Structure of 1,10-phenanthroline



Data

Amount of ferrous ammonium sulfate: 0.0724g

%Transmittance at $\lambda=505\text{nm}$

Conc(ppm)	%T
0.103	92.8%
0.515	76.6%
1.03	57.1%
2.06	32.3%
3.61	15.1%
Unknown	34.0%

Absorbance at 505nm

Conc(ppm)	Abs
0.103	0.0324
0.515	0.116
1.03	0.243
2.06	0.491
3.61	0.821
Unknown	0.468

Calibration Curve