Experiment 2: Spectrophotometric Determination of Iron

Unknown #3

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**Purpose:**

The goal of this experiment is to collect the absorbance of solutions with varying iron concentrations and create an absorbance vs. concentration graph. This graph will then be used to determine the concentration of an unknown iron solution based on its absorbance.

**Materials:**

Equipment:

* Parafilm
* 10 mL graduated cylinder
* 25 mL volumetric flasks
* Pipet
* 50 mL burette
* 100 mL beaker
* Cuvette and cover
* Genesys 20 Spectrophotometer
* Kimwipes

Chemicals:

* Nano-pure water
* 0.0001803 M Iron Solution
* 1.44 M Hydroxylamine hydrochloride
* Sodium acetate solution
* Orthophenanthroline
* Unknown # 3

**Procedure:**

Begin by adding the appropriate amount of iron solution into each volumetric flask. Then add, in this order, hydroxylamine hydrochloride, sodium acetate solution, and orthophenanthroline. Fill the remainder of the flask with nano-pure water. Invert solution using parafilm. Let the solutions sit for at least 5 minutes. While waiting, begin warming up the spectrophotometer. Run for at least 5 minutes before initial use. Place the blank solution into a clean cuvette. Clean with a kimwipe. Place the cuvette into the spectrophotometer. Set to appropriate wavelength for optimal absorbance. Press the 0 ABS 100% T button and make sure the read out is set to 0.000 A. Dispose of the blank solution into a waste breaker. Rinse the cuvette with the least concentrated solution and place into the waste beaker. Fill with the same solution and clean the cuvette with a kimwipe. Place cuvette into the spectrophotometer and record its absorbance value into data table 1. Dispose of the solution into a waste beaker and again fill the cuvette with blank solution. Set the absorbance so that the readout is once again 0.000 A. Repeat the above process for the remaining four iron solutions being sure to use the least concentrated solution first. Using the data from data table 1, create an absorbance vs. concentration graph (figure 1).

For the unknown determination, obtain an unknown iron solution. Fill the cuvette with blank solution and place into the spectrophotometer. Make sure the absorbance readout is 0.000A before placing the unknown solution into the cuvette. Rinse the cuvette with the unknown iron solution and dispose of into a waste beaker. Refill the cuvette with the unknown solution and place into the spectrophotometer. Record the absorbance value and place in data table 1. Using the graph, determine the concentration of the unknown iron solution.

**Results:**

Table 1: Absorbance Values for Various Concentrations of Iron

|  |  |  |
| --- | --- | --- |
| mL Iron in solution | Absorbance Value | Concentration of Iron (M) |
| 0.00 | 0.000 | 0.00 |
| 2.50 | 0.198 | 1.80 x 10-5 |
| 5.00 | 0.406 | 3.61 x 10-5 |
| 7.50 | 0.573 | 5.41 x 10-5 |
| 10.00 | 0.750 | 7.21 x 10-5 |
| 12.50 | 0.625 | 5.97 x 10-5 |
| Unknown # 3 | 0.722 | 6.92 x 10-5 |

*Determination of concentrations:*

M1V1=M2V2

Blank: (0.0001803 M) (0.00 mL) = M2 (25.00 mL)

M2= 0.00 M Iron

2.50 mL: (0.0001803 M) (2.50 mL) = M2 (25.00 mL)

M2= 1.80 x 10-5 M Iron

5.00 mL: (0.0001803 M) (5.00 mL) = M2 (25.00 mL)

M2= 3.61 x 10-5 M Iron

7.50 mL: (0.0001803 M) (7.50 mL) = M2 (25.00 mL)

M2= 5.41x 10-5 M Iron

10.00 mL: (0.0001803 M) (10.00mL) = M2 (25.00 mL)

M2= 7.21x 10-5 M Iron

12.50 mL: (0.0001803 M) (12.50 mL) = M2 (25.00 mL)

M2= 5.97x 10-5 M Iron

Figure 1: Absorbance of Iron Complex

*Determination of Unknown Iron Concentration:*

Y= mx + b {y= unknown absorbance; x= unknown iron concentration; m= molar absorptivity}

Y= 10218x +0.0148

0.722= 10218x +0.0148

0.7072= 10218x

X= [concentration unknown # 3] = 6.92 x 10-5 M Iron

The absorbance values for varying iron concentrations were determined and recorded in data table 1. As the concentration of the iron increased, so did the absorbance value. The unknown iron solution’s concentration was calculated using the equation generated by the graph depicted in figure 1. The data shows a fairly linear correlation. Unknown # 3 was determined to have a concentration of 6.92 x 10-5 M Iron.

**Conclusion:**

The absorbance values of solutions with various iron concentrations were recorded and used to create a graph of absorbance vs. concentration. As the concentrations of the solutions increased, so did their absorbance values. The line depicted in the figure shows a linear correlation between absorbance and concentration. An equation was generated based on the data. The resulting equation was y= 10218x + 0.0148. This equation was used to determine the concentration of iron in the unknown solution. The absorbance of the unknown was placed in the equation for y and x, the unknown iron concentration, was solved for. The concentration of the iron unknown solution was calculated to be 6.92 x 10-5 M.

No experiment is 100% accurate and thus an analysis of error must be conducted. Volumetric flasks can be rather difficult to use when trying to create a precise solution. One extra drop to the standard could produce an incorrect concentration. If there was a drop more than the 25 mL mark, the concentration would be more dilute and the solution would give a slightly lower absorbance reading that expected. This reading would adjust the equation line regarding the graph and would affect the calculated unknown concentration. When using the spectrophotometer, using a cuvette with a scratch or crack it in could result in error. A scratched cuvette might affect the path of light through the solution and thus give an incorrect absorbance reading. This again would affect the graph as well as the equation of the line. Each compound has a wavelength at which the compound reaches its maximum absorbance. For the Iron (II) phenanthroline complex, the maximum absorbance occurs at a wavelength of 508 nm. Failing to record the various absorbance values at the wavelength of 508 nm would result in values that were lower than it should be. As seen before, this would affect the equation of the line and would give a lower concentration for the unknown iron solution.

In order to try and resolve the sources of error there are numerous things that could be done. Squatting down at eye level and using a pipet to get the exact volume will ensure the best results and help to reduce error when using the volumetric flask. Picking a good cuvette and taking care of it will help to ensure that all light will pass through the solution. A good cuvette is one that has no scratches or marks and is thoroughly cleaned with kimwipe after being picked up. After determining the maximum absorbance value for a solution, making sure the spectrophotometer is set at that wavelength is vital. In order to confirm that the correct value is chosen, going up or down a couple wavelengths can help confirm the correct wavelength max was chosen.