Experiment 9: Determination of Iron with 1,10-Phenanthroline

In this experiment, the amount of iron present in a sample is quantitated by first reacting the iron with 1,10-phenanthroline to form a colored complex and then measuring the amount of light absorbed by this complex. Beer's law can then be used to determine the concentration relative to absorption: \( A = ebc \).

To form a complex, the iron must be first reduced to its ferrous state. This is done by reacting the iron with hydroxylamine hydrochloride by the following reaction:

\[
2 \text{Fe}^{3+} + 2 \text{NH}_2\text{OH} + 2 \text{OH}^- \rightarrow 2 \text{Fe}^{2+} + \text{N}_2 + 4 \text{H}_2\text{O}
\]

Once a colored complex is formed, the wavelength of light which is most strongly absorbed is found by measuring the absorbance at various wavelengths between 400 - 600 nm. After the most suitable wavelength is determined, a series of iron standards is measured at this wavelength and a calibration plot of absorbance vs. concentration is prepared. The absorbance of the unknown sample is measured and the calibration curve is used to calculate the concentration of iron in the sample.

Procedure:

NOTE: All iron solutions should be discarded into a Heavy Metals waste container.

1. Obtain your unknown in a 100-mL volumetric flask turned in to the TA the previous week. **You should provide a clean, labeled 100-mL volumetric flask to the TA for your unknown one week prior to this experiment.**

2. Obtain 100 mL of stock ferrous ammonium sulfate solution from the teaching assistant in a clean erlenmeyer flask. Be certain to note the mass of ferrous ammonium sulfate used by the TA to make the solution and the final volume of the solution prepared by the TA.

3. Label five additional 100-mL volumetric flasks as Std. 1, Std. 5, Std. 10, Std. 25 and blank.

4. Into the standard volumetric flasks, pipette 1, 5, 10 and 25 mL aliquots of the standard iron solution.

5. To each of the 6 flasks, add 1 mL of ~1.4 F hydroxylamine hydrochloride, 10 mL of ~5 mF 1,10-phenanthroline, and 8 mL of ~1.2 F sodium acetate (a buffer). DO NOT PUT YOUR PIPETTES INTO THESE SOLUTIONS; pour a small amount into your beaker and pipette from this.

6. Fill each flask to the mark with deionized water, mix well and allow to stand for 10 minutes.

7. Find a matching set of cuvettes; fill one cuvette with the analytical blank and fill the other with Std. 10.

8. Set the wavelength at 400 nm and zero the instrument.

9. Measure the absorbance of the standard in 20 nm increments in the range of 400 to 600 nm; you must re-zero the instrument every time you change the wavelength.

10. Determine the wavelength in this range which resulted in the maximum absorption. From this wavelength, measure the absorbance at +/- 15 nm in 5 nm increments.

11. Set the wavelength to the where the maximum absorbance was observed and measure the absorbance of the remaining standards and the unknown.
Report: (Submit plots and answer the questions on a separate sheet of paper)

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<thead>
<tr>
<th>Name</th>
<th>Date</th>
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<tbody>
<tr>
<td>Title</td>
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<tr>
<td>Unknown #</td>
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<tr>
<td>Concentration of unknown (ppm iron)</td>
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<tr>
<td>Estimated uncertainty in concentration (from $s_m$ and $s_b$)</td>
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<tr>
<td>The two reactions that are important for this experiment</td>
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Plot absorbance vs. wavelength (from steps 9-10) and indicate where $\lambda_{max}$ occurs.
Plot absorbance vs. concentration. Report the slope, intercept, and $c_{\lambda_{max}}$.
Answer the following questions:

1. Suppose you had used a water blank instead of the analytical (reagent) blank. How would your data have been affected?

2. In addition to its selective complexation of iron, what physicochemical property of 1,10-phenanthroline makes it suitable for this analysis?

Notes:
1. For the calibration curve, use a best-fit line for your data and do not force the line through the origin.

2. To determine ppm iron, you need to know that ferrous ammonium sulfate is \( \text{Fe(NH}_4\text{)}_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O} \) and has a molecular weight of 392.1 g/mol.