DETERMINATION OF IRON IN WATER

LAB VIS 5
From Juniata College SIM

INTRODUCTION
Spectrophotometers measure the amount of light absorbed by a sample. All colored solutions absorb visible light and can, therefore, be analyzed with a spectrophotometer. This experiment uses a spectrophotometer to determine the concentration of iron(II), or Fe$^{+2}$, ions in an unknown sample after analyzing the absorption values for a series of iron(II) standards, solutions of known concentration. Although solutions containing iron(II) ions are colorless, the addition of ortho-phenanthroline complexes the iron(II) ions, immediately forming an orange species. More iron(II) ions in a sample result in a deeper orange color that will absorb more light.

PURPOSE
The purpose of this experiment is to determine the concentration of iron(II) in an unknown sample.

MATERIALS
- 100 ppm Fe$^{+2}$ solution
- 0.25% ortho-phenanthroline solution
- graduated 10 mL pipet
  - or 1,2,4, and 6 mL pipets
- 6 – 100 mL volumetric flasks
- 6 cuvettes

Kimwipes
spectrophotometer
unknown or collected water sample
deionized water
10 mL graduated cylinder
small funnel

SAFETY
- Always wear goggles and an apron in the lab.
PROCEDURE

Part I: Preparation of standards
1. Label 5 100 mL volumetric flasks: 0 ppm (or blank), 1.0 ppm, 2.0 ppm, 4.0 ppm, 6.0 ppm.
2. Use pipets to deliver 0.0 mL, 1.0 mL, 2.0 mL, 4.0 mL, and 6.0 mL of the 100 ppm Fe$^{+2}$ solution to the appropriate 100 mL flasks.
3. Add 5 mL of 0.25% ortho-phenanthroline solution, measured in a graduated cylinder, to each flask. Add enough deionized water to each flask to dilute to the 100.0 mL mark. These are the standard solutions.
4. Label five cuvettes: 0 ppm (or blank), 1.0 ppm, 2.0 ppm, 4.0 ppm, 6.0 ppm.
5. Fill each cuvette about ¾ full with the corresponding standard solution.

Part II: Forming the standard curve
1. Ensure that the spectrophotometer has warmed up for at least 20 minutes. Use the same instrument for the entire experiment.
2. Adjust the wavelength to 510 nm.
3. Using a Kimwipe, wipe off the cuvette containing the blank (0 ppm). Do not touch the clear sides of the cuvette. Place the cuvette in the sample compartment with the triangle on the cuvette facing the front of the instrument. Close the lid.
4. Press 0 ABS 100%T
   Record the absorbance of the blank solution in the Data Table.
5. Remove the cuvette from the instrument.
6. Wipe and align the 1.0 ppm sample cuvette in the same manner. Close the lid. Record the absorbance of this solution in the Data Table.
7. Repeat step 6 to obtain absorbance readings for each of the other standard solutions.
8. Make a graph of absorbance (y-axis) vs. concentration (x-axis), this is the standard curve.
Part III: Preparation and analysis of the unknown

1. Obtain an unknown sample from the instructor or prepare your own unknown using a collected water sample and the instructions given in step 2. If the unknown is provided, skip to step 5.

2. Prepare your own unknown by adding 5 mL of 0.25% ortho-phenanthroline solution to a 100 mL volumetric flask. Dilute to mark with the collected water sample.

3. If using a collected water sample, fill a cuvette about ¾ full with the sample. This will be the blank. Insert the blank cuvette into the spectrophotometer and press 0 ABS 100%T.

4. Remove the blank cuvette from the instrument.

5. Fill a cuvette about ¾ full with your unknown, either prepared or provided by your instructor.

6. Record the absorbance of the unknown solution in the Data Table.

7. From the standard curve, determine the concentration of iron in this unknown sample. To do this, locate the absorbance of the unknown solution on the y-axis of the standard curve graph. Find the point where this absorbance intersects the standard curve. The corresponding x-axis value is the predicted concentration of the unknown sample. The concentration of the unknown can also be found using the slope of the graph.
DETERMINATION OF IRON IN WATER

DATA TABLE

<table>
<thead>
<tr>
<th>Concentration</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0 ppm (blank)</td>
<td></td>
</tr>
<tr>
<td>1.0 ppm</td>
<td></td>
</tr>
<tr>
<td>2.0 ppm</td>
<td></td>
</tr>
<tr>
<td>4.0 ppm</td>
<td></td>
</tr>
<tr>
<td>6.0 ppm</td>
<td></td>
</tr>
<tr>
<td>unk #____</td>
<td></td>
</tr>
</tbody>
</table>

Concentration of iron in unknown: __________

QUESTIONS

1. Why was ortho-phenanthroline added to the solutions?

2. What is the purpose of preparing and analyzing standard iron solutions?

3. What other items could be analyzed using this method?