Photometric Determination of Iron

Theory

In photometry a beam of light is used to measure the concentration of a solution. If you shine a beam of light through a coloured solution, some of the light will be absorbed in passing. The more intense the colour, the more concentrated the solution, and the more the light will be absorbed. There is a direct relationship between the concentration of the solution and the amount of light absorbed. The more concentrated the solution, the greater the amount of light absorbed. Since we have a method of determining the amount of light absorbed, we also have a method of determining the concentration of a solution.

You should be aware of the photoelectric effect. When light is allowed to impinge on certain of the alkali metals, the energy is sufficient to liberate electrons from the higher energy levels. These electrons can be caused to flow through a conducting wire to an ammeter where the rate of flow of charge can be measured. This is also proportional to the amount of light absorbed. Thus the measured amperage is proportional to the solution concentration.

To use this concept analytically, we must first measure the light absorbed as it passes through coloured solutions of known concentration of the species we wish to test. We can plot this concentration-absorbance data on a graph and derive a slope. This slope can be used as a standard. We then measure the absorbance of an unknown solution, locate this value on the slope and determine the concentration from this.

You will use a piece of apparatus called a Spectronic-20. This instrument allows for the selection of a specific wave length of light (monochromatic) rather than using all the wave lengths found in white light. This wave length tends to give the best results with the coloured solution used. It also uses a procedure while allows you to eliminate the effects of other added chemicals and the solvent so that all you will be measuring is the concentration of the unknown species as shown by the specific colour complex it forms. This is why you will use a blank which contains the same solvent and all the chemicals except the particular species you are measuring.
**Procedure**

1. Obtain six clean test tubes. Place them in a wooden test tube rack and label their concentrations in parts per million (ppm) according to the table below.

<table>
<thead>
<tr>
<th>Volume Fe³⁺ (aq) mL</th>
<th>Volume H₂O (l) mL</th>
<th>Fe³⁺ (aq) (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>20.00</td>
<td>0.000</td>
</tr>
<tr>
<td>2.00</td>
<td>18.00</td>
<td>0.200</td>
</tr>
<tr>
<td>4.00</td>
<td>16.00</td>
<td>0.400</td>
</tr>
<tr>
<td>6.00</td>
<td>14.00</td>
<td>0.600</td>
</tr>
<tr>
<td>8.00</td>
<td>12.00</td>
<td>0.800</td>
</tr>
<tr>
<td>10.00</td>
<td>10.00</td>
<td>1.000</td>
</tr>
</tbody>
</table>

2. Clean a 50 mL buret and rinse it with two 10 mL portions of the Fe³⁺ (aq) solution.
3. Fill the buret completely with the Fe³⁺ (aq) solution. Make sure there is no air in the tip of the buret and that it reads 0.00 mL. Use an eye dropper, if needed, to make the volume 0.00 mL initially.
4. Obtain a second 50 mL buret. Clean it and rinse with distilled water.
5. Fill the second buret with distilled water. Make sure that there is no air in the tip of the buret and that the volume reads 0.00 mL. Use an eye dropper to ensure that the volume is 0.00 mL initially.
6. Run 20.00 mL of distilled water into the test tube labeled 0.000 ppm.
7. Run 2.00 mL of Fe³⁺ (aq) and 18.00 mL of distilled water into the test tube labeled 0.200 ppm.
8. In a similar fashion, prepare the rest of the set of six standard solutions. You will have to refill the buret as required.
9. Obtain an unknown from stores. It will contain 20.00 mL. All test tubes should contain 20.00 mL. If the heights of the liquid in the test tubes appear to vary, ignore it. This is due to slight differences in the sizes of the test tubes.
10. Add 10 drops 6.0 mol/L HNO₃ (aq) and 40 drops of 2.0 mol/L KSCN (aq) to each test tube. Tap each tube to aid mixing. Do not stir or shake the tubes. Allow to stand for ten to fifteen minutes. You should have a set of seven test tubes at this time each with varying concentrations of the Fe³⁺ solution and drops of HNO₃ and KSCN.
11. Obtain seven cuvettes from stores. They should be clean. However, if there are slight water spots, clean them with the wipes provided. Don’t use regular paper towels as they will scratch the cuvette and distort your results.
12. Arrange in order the seven cuvettes and fill each to within one centimeter of the top with your set of standards and unknown.
13. Use the Spectronic-20 to compare the concentrations in the cuvettes. The general operating instructions are included in this handout.
14. Record the percentage transmittance and the absorbance of each cuvette in a neat data table (prepare before class!).
15. Percent transmittance is recorded to one decimal place. Absorbance is recorded to three decimal places.
**Determination of the Unknown**

Construct a graph of percentage transmittance and absorbance on the vertical axes versus concentration of the iron (III) ion in ppm on the horizontal axis for your set of six standards. This should produce two straight lines or slopes. For the unknown, locate the appropriate percentage transmittance and absorbance values on the lines. Extend these points down to the concentration axis. These should intersect the concentration axis at the same concentration. If they differ, then report the average of the two values as the concentration of the unknown. Please refer to the handout of creating graphs to ensure that you have all the elements required for a proper graph.
Operating Instructions for the Bausch & Lomb Spectronic-20

1. Turn the instrument on by rotating the amplifier control (c) in a clock-wise direction. Allow 5 minutes for the instrument to warm up. (The machines should already be warmed up for you.)

2. Rotate the wavelength control (a) until the desired wavelength is shown on the wavelength scale (b). (We will be using 590; this also should be selected for you.)

3. Adjust the amplifier control (c) with the sample compartment lid (e) closed until the meter needle reads 0 on the % Transmittance scale (d). This is zeroing the meter.

4. Fill a clean cuvette with the 0.000 ppm solution and wipe it with lens paper (or KIMWIPES) to remove liquid droplets, dust and finger prints.

5. Place the 0.000 cuvette in the sample compartment and align the guide mark on the cuvette with the guide mark at the front of the sample compartment. Close the lid.

6. Rotate the light control (f) so that the meter reads 100 on the % Transmittance scale or 0 on the Absorbance scale. This compensates in such a way that any reading you get from following solutions will only be due to the iron (III) ion content.

7. Remove the 0.000 cuvette.

8. Load the first test cuvette in the sample compartment and read the Absorbance and % Transmittance on the dial. Note these readings in your table.

9. Repeat steps 5-9 for each sample.

10. When you are finished, pour all the solutions down the sink. Rinse the cuvettes with water but do not attempt to clean them. They are easily scratched and this makes them unsuitable for use in the Spectronic-20.