WHAT’S IN THE WATER: DETERMINATION OF IRON

INTRODUCTION

Iron can create serious problems in public water supplies. Along with manganese, iron can interfere with laundering operations and can impart stains to plumbing fixtures. Iron and manganese can also cause problems in the water-distribution system by supporting the growth of bacteria. Iron imparts to water, a taste that is detectable at very low concentrations. Because of this, Health and Welfare Canada recommends that public water supplies not contain more than 0.3 mg/L of iron or 0.05 mg/L of manganese.

In this experiment, you will play the role of a quality control chemist and determine the amount of iron in a sample of water. You will use a colorimetric method to find the concentration of iron (III) ions in the parts per million (ppm) range. The principle behind this method is to find a chemical agent or agents that react specifically with iron (III) ions, so that a coloured solution results. The intensity of the colour depends quantitatively on the amount of iron ions in the sample. Such chemical agents do exist for iron (III) ions. A substance called potassium thiocyanate (KSCN) combines with Fe$^{3+}$ ions to produce a distinctive red colour. This red colour intensifies as the iron concentration increases.

The colorimetric procedure involves making up a series of known iron solutions and treating them with potassium thiocyanate. At the same time, we also treat two water samples (whose iron concentrations are to be determined) with the KSCN. After the colour develops in the solutions, we compare the colour of the water sample with the colours of the known iron solutions.

A person can, of course, compare colours by visual observation, but this method is not always reliable, especially if the colours are very similar in intensity. A much better method is to use a device called a colorimeter, an instrument that measures the intensity of the colour electronically.

The colorimeter does this by measuring the absorbance of a specific wavelength of light as it passes through the coloured sample. Each of the known iron solutions absorbs a different amount of light. The more iron in the solution, the more light it absorbs. The observer reads the absorbance of each sample on the colorimeter meter and then draws a graph, plotting the concentration of each known iron solution against its absorbance. A sample graph is shown below. The procedure should give a straight line, called a calibration curve. The calibration curve enables one to determine the iron content of the water samples by finding the absorbance of the unknown solutions on the calibration curve, and reading off the corresponding concentration of iron in ppm.
Figure 5-1. Graph of absorbance versus iron concentration. The calibration curve is plotted from absorbance of solutions of known iron concentration.

**Colourimetric Determination of Iron Concentration in Unknown #5**

Absorbance

Concentration Iron [ppm]

\[ y = 0.0814x + 0.0058 \]

\[ R^2 = 0.9946 \]
1. The following table contains all of the data necessary for preparing five solutions of known iron content, and for preparing one blank to serve as a reference solution. The stock solution contains 0.05 mg of iron/mL of solution. Sample calculations follow after the table.

<table>
<thead>
<tr>
<th>Test Tube #</th>
<th>Iron Content (mg)</th>
<th>Concentration of Iron (ppm)</th>
<th>Volume Required of Stock Iron Solution (mL)</th>
<th>Volume Required of Water (mL)</th>
<th>TOTAL Volume (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 BLANK</td>
<td>0.00</td>
<td>0</td>
<td>0.0</td>
<td>10.0</td>
<td>10.0</td>
</tr>
<tr>
<td>2</td>
<td>0.01</td>
<td>1</td>
<td>0.2</td>
<td>9.8</td>
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</tr>
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<td>3</td>
<td>0.6</td>
<td>9.4</td>
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</tr>
<tr>
<td>4</td>
<td>0.05</td>
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<td>1.0</td>
<td>9.0</td>
<td>10.0</td>
</tr>
<tr>
<td>5</td>
<td>0.07</td>
<td>7</td>
<td>1.4</td>
<td>8.6</td>
<td>10.0</td>
</tr>
<tr>
<td>6</td>
<td>0.10</td>
<td>10</td>
<td>2.0</td>
<td>8.0</td>
<td>10.0</td>
</tr>
<tr>
<td>7</td>
<td>unknown</td>
<td>unknown</td>
<td></td>
<td></td>
<td>10.0</td>
</tr>
<tr>
<td>8</td>
<td>unknown</td>
<td>unknown</td>
<td></td>
<td></td>
<td>10.0</td>
</tr>
</tbody>
</table>

SAFETY ALERTS!

**Hydrochloric acid 4 M** (corrosive) - causes severe burns to eyes and skin, avoid contact with clothing. Eye contact: flush eyes with water for 15 minutes, seek medical advice. Skin contact: wash affected areas with warm running water for 15 minutes.

**Potassium thiocyanate** - causes irritation to eyes and skin, avoid contact with clothing. Eye contact: flush eyes with water for 15 minutes. Skin contact: wash affected areas with soap and warm water.

**Hazardous waste** - Dispose of the red-coloured iron-thiocyanate complex and thiocyanate solution in the waste containers provided.
Sample Calculations Using Test Tube #2.

(i) Concentration of Iron (ppm)

The unit of ppm can be defined as mg solute per L solution. Test tube #2 contains 0.01 mg iron in 10.0 mL solution. What is the iron concentration in ppm?

First, convert volume to L. \( 10.0 \text{ mL} \times \frac{1 \text{ L}}{1000 \text{ mL}} = 0.0100 \text{ L} \)

Then calculate the concentration in ppm.

\[
[\text{Fe}^{3+}] \text{ in ppm} = \frac{0.01 \text{ mg iron}}{0.0100 \text{ L solution}} = 1 \text{ ppm}
\]

(ii) Volume Required of Stock Iron Solution (mL)

The stock solution contains 0.05 mg of iron/mL of solution. If 0.01 mg of iron is needed for your standard solution, let \( X \) be the volume of stock solution required.

\[
\frac{X \text{ mL}}{0.01 \text{ mg iron}} = \frac{1.00 \text{ mL iron solution}}{0.05 \text{ mg}}
\]

\[
X \text{ mL} = \frac{0.01 \text{ mg} \times 1.00 \text{ mL}}{0.05 \text{ mg}} = 0.2 \text{ mL}
\]

(iii) Volume Required of Water (mL)

The total volume for each test tube is to be 10.0 mL, and a volume of 0.2 mL of stock iron solution has already been added to test tube #2. Therefore the amount of water required is:

\[
10.0 \text{ mL} - 0.2 \text{ mL} = 9.8 \text{ mL}
\]

2. Plug the colorimeter into the electric socket at your bench and allow the machine to warm up.

3. You have been provided with eight dry screw cap tubes in a test tube rack which should be labelled #1 through to #8. Each tube contains 1 mL of 4.0 M HCl and 1 mL of 10% Potassium Thiocyanate solution. Safety Reminder: If you spill any of this solution on your skin or your clothes, tell the instructor or your teacher immediately.

4. Obtain 15 mL of stock iron solution in a clean dry beaker.

5. Add to tubes #1 through #6, the required volume of distilled water as calculated. Use a 10 mL graduated pipette for your additions. Touch the tip of the pipette to the side of the test tube while draining the water.
6. Using a graduated 2 mL pipette, add to tubes #2 through #6, the required amount of stock iron solution. *Take care that there is no contamination of tube #1 with iron.* This is the blank solution that you need to standardize your colorimeter.

(NOTE: Those who have difficulty using the pipettes can choose to measure out their water and iron solution using a graduated cylinder.)

7. Hand in tubes #7 and #8 to your instructor and obtain two different unknown iron solutions of 10 mL each.

8. Tap each tube carefully to mix the solutions, taking care not to spill any.

9. Cap each tube and invert 5 times to mix the solutions.

10. Based upon the color of the known and unknown samples, estimate the concentration of the unknown samples. Record your guesses on the Report Sheet.

11. Your instructor will demonstrate how to operate the colorimeter. Directions have also been included on page 6 of this booklet.

12. Obtain the readings for all your solutions, going from the *lowest* iron concentration to the *highest* concentration. The darker the solution, the higher the content of iron. **Note:** Before each reading, rinse the cuvettes with a little of the solution to be tested before filling it for the reading. This will prevent contamination by the previous solution. Dry the outside of the cuvette with Kimwipe before inserting it into the colorimeter. Pour the contents of each solution back into the test tubes after you are finished. This will allow you to recheck any measurement.

Record all your results in a table on the Report Sheet. Plot a graph of absorbance (calculated using the equation below) versus iron concentration (ppm) and draw the line-of-best-fit. This is your calibration curve. Plot the absorbances of your two unknowns on it, and determine the iron concentration (ppm) of the two unknown solutions. State the concentration of the unknowns on the graph.

Absorbance = log (1 / T), For example: where % T = 88.1 % (88.1 / 100),

\[
\text{Absorbance} = \log \left( \frac{1}{0.881} \right)
\]
Spectronic 20 Instructions

Key

1. Sample Compartment
2. Pilot Lamp
3. Wavelength control
4. Transmittance / Absorbance control (100%T / 0 A)
5. Power switch / Zero control
6. Filter lever (Note: Only the 20+ models have a filter lever)

Operation

1. Turn on the instrument by turning the Power switch (#5) clockwise. Allow the spectrophotometer to warm up for at least 15 minutes to stabilize.
2. After the warm-up period, set the desired wavelength (540 nm for iron analysis) with the Wavelength control knob (#3).
3. Set the filter lever (#6, if equipped) to the left
4. Remove the cuvette from the sample compartment (#1), close the cover, and adjust the Zero control (#5) so that the meter reads 0% T.
5. Fill the cuvette ¾ full with the blank solution (from test tube #1) and wipe the cuvette with a Kimwipe to remove liquid droplets, dust, and fingerprints.
6. Place the cuvette into the sample compartment, close the cover, and adjust the 100%T using the Transmittance / Absorbance control (#4) so that the meter reads 100%T.
7. Remove the cuvette and empty the contents into waste.
8. Rinse the cuvette twice with small volumes of the solution to be measured and fill it ¾ full with the solution to be measured.
9. Wipe the cuvette with a Kimwipe, insert the cuvette into the sample compartment (#1) and close the lid.
10. Read the %T value from the meter
11. Remove the cuvette from the sample compartment and repeat steps 7-10 for the remaining solutions
12. When all measurements are complete, turn off the spectrophotometer by turning the Power Switch / Zero Control counter clockwise until it clicks.

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REPORT SHEET

Partner #1: ______________________________ date: ______________

Partner #2: ______________________________

1. TUBE #7: Unknown Sample Number ______
   Estimated Iron Concentration: _______ ppm

2. TUBE #8: Unknown Sample Number ______
   Estimated Iron Concentration: _______ ppm

3. Results.

<table>
<thead>
<tr>
<th>Tube #</th>
<th>Iron Content (mg/10 mL)</th>
<th>Iron Concentration (ppm)</th>
<th>Absorbance reading</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.00</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>2</td>
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