REDOX TITRATION

PURPOSE

To determine the concentration of a sodium thiosulphate (Na\(_2\)S\(_2\)O\(_3\)) by a redox titration with the I\(_2\) generated in a reaction with KI\(_3\) using the starch-iodine complex as the indicator.

INTRODUCTION

In a reaction with the thiosulphate ion (S\(_2\)O\(_3^2-\)), iodine (I\(_2\)) is reduced to iodide (I\(^-\)) and the thiosulphate is oxidized to the tetrathionate ion (S\(_4\)O\(_6^{2-}\)). Iodine is only slightly soluble in water, but in the presence of excess iodide ion, it forms the soluble tri-iodide ion (I\(_3^-\)) that is used in redox titrations: I\(_2\) + I\(^-\) → I\(_3^-\). The actual reaction that occurs in the redox titration is then between the tri-iodide ion and the thiosulphate ion.

In this experiment, the thiosulphate is titrated against a known volume of a standard iodate in the presence of excess iodide. The endpoint is signaled by the disappearance of a blue color, due to a starch indicator, when enough thiosulfate has been added to consume the iodine.

PRE-LAB QUESTIONS

1. Write balanced net ionic equations for the reaction of:
   a) Iodate ion (IO\(_3^-\)) with iodide in an acid solution to form I\(_3^-\)

<table>
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<td>IO(_3^-) + 8 I(^-) → 3 I(_3^-)</td>
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   b) I\(_3^-\) and the thiosulfate ion to form the iodide ion and tetrathionate ion

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2. Calculate the concentration of an iodate solution that contains 1.9853 g of KIO₃ in a 1000 mL volumetric flask.

**Steps**

Find the number of moles of KIO₃ are in 1.9853 g.

\[
\text{n}_{\text{KIO}_3} = \frac{1.9853 \text{ g}}{214 \text{ g/mol}}
\]

\[
\text{n}_{\text{KIO}_3} \approx 0.00928 \text{ mol}
\]

Calculate a molar ratio of iodate ions in KIO₃.

\[
\frac{1}{0.00928 \text{ mol}} = \frac{1}{x}
\]

\[
x = 0.00928 \text{ mol}
\]

Use the formula Concentration = \( \frac{\text{Mol}}{\text{Volume (L)}} \) to calculate the concentration.

\[
C = \frac{n}{V}
\]

\[
C = \frac{0.00928 \text{ mol}}{1 \text{ L}}
\]

\[
C = 9.28 \times 10^{-3} \text{ mol/L}
\]

3. Calculate the molar concentration of a thiosulfate solution given that 25.00 mL of 0.0195 mol/L KIO₃ solution in a flask containing 2.00 g of KI and 10 mL of 0.500 mol/L H₂SO₄ required 34.81 mL of thiosulfate solution to reach the starch endpoint.

**Reaction Between IO₃⁻ and I⁻**

\[
\text{IO}_3^- + 8 \text{ I}^- + 6 \text{ H}^+ \rightarrow 3 \text{ I}_3^- + 3 \text{ H}_2\text{O}
\]

Mol of IO₃⁻ reacted = 0.00049 mol

Number of mol of I₃⁻ released = 3 × 0.00049 mol = 0.00147 mol

\[
C_{\text{KIO}_3} = \frac{n_{\text{KIO}_3}}{V_{\text{KIO}_3}}
\]

\[
0.0195 \text{ M} = \frac{n_{\text{KIO}_3}}{0.025 \text{ L}}
\]

\[
0.00049 \text{ mol} = n_{\text{KIO}_3}
\]

**Reaction Between I⁻ and S₂O₃²⁻**

\[
2 \text{ S}_2\text{O}_3^{2-} + \text{ I}_3^- \rightarrow 3 \text{ I}^- + \text{ S}_4\text{O}_6^{2-}
\]

1 mol of I₃⁻ reacts with 2 mol of S₂O₃²⁻ \( \rightarrow 2 \times 0.00147 \text{ mol} = 0.00294 \text{ mol} \)

Concentration of S₂O₃²⁻ solution \( \rightarrow 0.08 \text{ mol/L} \)

\[
C = \frac{n}{V}
\]

\[
C = \frac{0.00294 \text{ mol}}{0.0348 \text{ L}}
\]

\[
C \cong 0.08 \text{ mol/L}
\]
DEFINITIONS

Titration
The precise addition of a solution in a burette into a measured volume of a sample solution

Titrant
The solution in a burette during a titration

End Point
The point in a titration at which a sharp change in a measurable and characteristic property occurs (usually a color change)

Equivalence Point
The measured quantity of a titrant recorded at the point at which chemically equivalent amounts have reacted

Burette
A graduated tube of glassware that has a stopcock at its bottom end. It is used to dispense precise volumes of liquid reagents

MATERIALS

KI\textsubscript{2}O\textsubscript{3} (aq) \text{________ M}  Erlenmeyer flasks  Na\textsubscript{2}S\textsubscript{2}O\textsubscript{3} solution  starch solution

Beakers  graduated cylinders  0.5 M H\textsubscript{2}SO\textsubscript{4} solution

wash bottle  burets and clamps  solid KI

distilled water  balance  retort stand

PROCEDURE

1. Assemble the equipment.
2. Pipette 25.0 mL of the standard KI\textsubscript{2}O\textsubscript{3} solution into a flask. Add 2.000 g of solid KI and 10 mL of 0.500 mol/L H\textsubscript{2}SO\textsubscript{4} to the flask
3. Properly fill a burette with the thiosulfate solution.
4. Titrate with the thiosulfate until the solution has lost its reddish-brown color and has become orange.
5. Add 2 mL of starch indicator and complete the titration.
6. Note the initial and final burette readings to at least one decimal place.
7. Repeat the titrations at least twice more until the concentration of the thiosulfate agrees to within 10%.
OBSERVATIONS
Concentration of KIO₃: 4.305g/2L → 0.01 mol/L

<table>
<thead>
<tr>
<th>Na₂S₂O₃</th>
<th>Rough</th>
<th>Trial #1</th>
<th>Trial #2</th>
<th>Trial #3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final Burette Reading (mL)</td>
<td>19.0</td>
<td>21.0</td>
<td>40.8</td>
<td>46.6</td>
</tr>
<tr>
<td>Initial Burette Reading (mL)</td>
<td>0.0</td>
<td>0.0</td>
<td>21.0</td>
<td>25.0</td>
</tr>
<tr>
<td>Volume Used (mL)</td>
<td>19.0</td>
<td>21.0</td>
<td>19.8</td>
<td>21.6</td>
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CALCULATIONS
1. What is the concentration of KIO₃?

   The concentration of KIO₃ was 0.01 M.

2. Write the balanced equation for the reaction between iodate and iodide ions (see pre-lab)

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3. Calculate the moles of iodate used in each titration and the moles of I₃⁻ produced in each reaction with the iodate ion.

   \[ C_{IO₃} = \frac{n_{IO₃}}{V_{KIO₃}} \]

   \[ 0.01 M = \frac{n_{IO₃}}{0.025 L} \]

   \[ 0.00025 mol = n_{IO₃} \]

   \[ 10I₃⁻ + 8 I⁻ + 6 H⁺ → 3 I₃⁻ + 3 H₂O \]

   For every 1 IO₃⁻ ion, 3 I₃⁻ are produced. Therefore, $3 \times 0.00025 \text{ mol of } IO₃⁻ = 0.00075 \text{ mol of } I₃⁻$ produced.

4. Write the equation for the reaction of the tri-iodide ion and the thiosulfate ion (see pre-lab)

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5. Calculate the moles of thiosulfate in each titration and the concentration of the thiosulfate solution.

\[2 \text{S}_2\text{O}_3^{2-} + \text{I}_3^- \rightarrow 3 \text{I}^- + \text{S}_4\text{O}_6^{2-}\]

1 mol of \(\text{I}_3^-\) reacts with 2 mol of \(\text{S}_2\text{O}_3^{2-}\) \(\rightarrow\) 0.00075 mol of \(\text{I}_3^-\) \(\times\) 2 = 0.0015 mol of \(\text{S}_2\text{O}_3^{2-}\)

<table>
<thead>
<tr>
<th>Trial #</th>
<th>Moles of (\text{S}_2\text{O}_3^{2-}) (mol)</th>
<th>Volume of (\text{S}_2\text{O}_3^{2-}) used (L)</th>
<th>Concentration of (\text{S}_2\text{O}_3^{2-}) (mol/L)</th>
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<tr>
<td>Rough</td>
<td>0.0015</td>
<td>0.0190</td>
<td>0.093</td>
</tr>
<tr>
<td>1</td>
<td>0.0015</td>
<td>0.0210</td>
<td>0.084</td>
</tr>
<tr>
<td>2</td>
<td>0.0015</td>
<td>0.0198</td>
<td>0.089</td>
</tr>
<tr>
<td>3</td>
<td>0.0015</td>
<td>0.0216</td>
<td>0.082</td>
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