Analysis of Vitamin C Using Iodine

Introduction

Vitamin C (ascorbic acid) is oxidized to dehydroascorbic acid using a mild oxidizing agent such as iodine. The oxidation is a two-electron process, following the reaction:

\[
\begin{align*}
\text{OH} & \quad \text{OH} \\
\text{H} & \quad \text{OH} \\
\text{OH} & \quad \text{OH} \\
\end{align*}
\]

(ascorbic acid)

\[
\begin{align*}
\text{OH} & \quad \text{OH} \\
\text{H} & \quad \text{OH} \\
\text{OH} & \quad \text{OH} \\
\end{align*}
\]

\[+ \text{I}_2 \quad \rightarrow \]

(dehydroascorbic acid)

Vitamin C in water solution is readily oxidized by dissolved oxygen; therefore samples should be dissolved immediately before analysis. Long-term storage of vitamin C solutions must be avoided. During titration, a small amount of oxidation from oxygen already dissolved in the solution is unavoidable, but is usually not significant. However, continuous shaking of an open flask will bring about enough absorption of oxygen to cause a significant error.
Solutions needed for this experiment:

<table>
<thead>
<tr>
<th>Solutions prepared by the student</th>
<th>Solutions provided by the instructor</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0300 N Iodine titrant</td>
<td>Concentrated hydrochloric acid</td>
</tr>
<tr>
<td>5% sodium hydroxide</td>
<td>Starch Indicator Solution</td>
</tr>
</tbody>
</table>

Specialized equipment needed:

No specialized equipment is used in this experiment.

Experimental Procedure

Preparation and standardization of iodine titrant:

1. Using the top loading balance, weigh approximately 20.0 g of reagent grade potassium iodide. **Make sure to record the exact weight of potassium iodide, to two decimal places, in your notebook.** Dissolve the potassium iodide in approximately 25 mL of DI water. Weigh approximately 3.80 g of iodine, record the exact weight of iodine, to two decimal places, in your notebook, and add the iodine to the potassium iodide solution. (**NOTE: do NOT weigh the iodine in the analytical balance!!**) Stir the solution carefully to dissolve all of the iodine. (Iodine dissolves slowly, so this step may take some time.) When all of the iodine has dissolved, analytically transfer the solution to a 1.000 L volumetric flask and QS with DI water. Clean a brown glass bottle with soap and water, rinse 2-3 times with small portions of DI water, followed by 2-3 rinsings with small portions of your iodine titrant. After rinsing the brown bottle with iodine titrant, store the rest of your iodine titrant in the brown bottle. Label the bottle with your name (or initials) and date. All waste materials from this step may be flushed down the sink with water.

2. Weigh ~0.3700 grams of arsenic (III) oxide on the analytical balance and transfer into a 250 mL beaker. Add ~20 mL of 5% sodium hydroxide solution and stir until the solid dissolves. Add 50 mL of deionized water and 2 mL of 12 M hydrochloric acid (concentrated hydrochloric acid). Transfer this solution quantitatively to a 250.0 mL volumetric flask and QS with DI water.

The chemical reactions involved in this step are:

$$\text{As}_2\text{O}_3(s) + 2\text{OH}^- + 2\text{H}_2\text{O} \rightarrow 2\text{H}_2\text{AsO}_3^- + 2\text{H}_2^+ \rightarrow 2\text{H}_3\text{AsO}_3$$
Therefore, 0.3700 g arsenic (III) oxide produces 0.001870 moles of arsenic (III) oxide, which produces 0.003741 moles of arsenious acid. When diluted to 250.0 mL, the resulting solution is 0.01496 M, or 0.02993N. Of course, a different mass of arsenic (III) oxide produces a different normality of solution. Calculate the normality of your solution based on the mass of arsenic (III) oxide that you used.

3. Pipette 25.00 mL of the arsenious acid into each of three Erlenmeyer flasks (~125 mL size). Add 25 mL of DI water and 3.5 grams of sodium bicarbonate (a solid material) to each flask. Check the pH of each solution with pH paper (NOT litmus paper). If necessary, add additional small amounts of sodium bicarbonate until the pH is between 7 and 8. If you overshoot pH 8, add small amounts of hydrochloric acid to reduce the pH.

pH Test Paper color chart:

4. Add 5 mL of starch indicator to each flask and titrate with iodine solution until the first, stable, blue starch-triiodide color is achieved. Calculate the average normality of the iodine solution. Normality is calculated below. Label the iodine titrant with the normality, and record the normality in your notebook.

\[
N_{I_2} = N_{H_3AsO_3} \times \frac{mL_{H_3AsO_3}}{mL_{I_2}}
\]
Titration of vitamin C unknown:

1. Weigh ~0.5 grams of your unknown vitamin C sample to 4 decimal places, record the mass, and transfer to a 100.00 mL volumetric flask. QS with DI water and mix by inversion several times. (NOTE: a small amount of binder present in the vitamin C will not dissolve, and gives the solution a slightly cloudy appearance. This binder does not affect the results.)

2. Pipette 25.00 mL of the vitamin C solution into a 250 mL Erlenmeyer flask and add 5 mL of starch indicator. Titrate the unknown with iodine titrant rapidly but accurately, to the first appearance of a stable blue starch-triiodide color. The entire contents of the Erlenmeyer flask should be the same blue color, and should maintain its color for at least 10 seconds.

3. Analyze 2 additional 25.00 mL aliquots of vitamin C solution.

4. Your instructor will provide you with details concerning how to report the vitamin C concentration.

Lab report

A sample report is shown at the end of this procedure.

WASTE DISPOSAL: All solutions containing arsenic must be disposed of in the aqueous metals waste container. All other solutions may be poured down the sink.
Vitamin C
Unknown #20098762435
Robert "Bud" Fisher

Standardization of Iodine titrant:

<table>
<thead>
<tr>
<th></th>
<th>1st</th>
<th>2nd</th>
<th>3rd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vol. Arsenious acid, mL</td>
<td>25.00</td>
<td>25.00</td>
<td>25.00</td>
</tr>
<tr>
<td>N Arsenious acid</td>
<td>0.03010</td>
<td>0.03010</td>
<td>0.03010</td>
</tr>
<tr>
<td>Volume Iodine titrant, mL</td>
<td>25.05</td>
<td>25.07</td>
<td>25.00</td>
</tr>
<tr>
<td>N Iodine titrant</td>
<td>0.03016</td>
<td>0.03018</td>
<td>0.03010</td>
</tr>
</tbody>
</table>

N Iodine titrant (average) = 0.03014

Titration of vitamin C unknown:

Mass of vitamin C sample: 0.5978 grams

<table>
<thead>
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<th>2nd</th>
<th>3rd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vol. Vitamin C solution, mL</td>
<td>25.00</td>
<td>25.00</td>
<td>25.00</td>
</tr>
<tr>
<td>Vol. Iodine titrant, mL</td>
<td>4.85</td>
<td>4.75</td>
<td>4.80</td>
</tr>
<tr>
<td>Normality, vitamin C</td>
<td>0.00584716</td>
<td>0.0057266</td>
<td>0.00578688</td>
</tr>
<tr>
<td>Molarity, vitamin C</td>
<td>0.00292358</td>
<td>0.0028633</td>
<td>0.00289344</td>
</tr>
<tr>
<td>Moles, vitamin C</td>
<td>0.000292358</td>
<td>0.00028633</td>
<td>0.000289344</td>
</tr>
<tr>
<td>mg, Vitamin C</td>
<td>51.49009096</td>
<td>50.4284396</td>
<td>50.95926528</td>
</tr>
</tbody>
</table>

Average mg. Vitamin C in sample: 50.95926528
Vitamin C in Sample: 85.2 mg/g sample

NOTE: results are not rounded until the final answer!!

Sample calculations shown on following pages.
Sample calculations:

Normality of iodine:

\[ N_{\text{iodine}} = \frac{(mL_{\text{arsenious acid}} \times N_{\text{arsenious acid}})}{mL_{\text{iodine}}} \]

\[ = \frac{(25.00mL \times 0.03010N)}{25.04mL} = 0.03005N \]

Normality of vitamin C:

\[ N_{\text{VC}} = \frac{(mL_{\text{iodine}} \times N_{\text{iodine}})}{mL_{\text{VC solution}}} \]

\[ = \frac{(4.85mL \times 0.03005N)}{25.00mL} = 0.0058297N \]

Molarity vitamin C:

\[ M_{\text{VC}} = \frac{N_{\text{VC}}}{2} \]

\[ = \frac{0.0058297N}{2} = 0.00291485M \]

Moles vitamin C in sample:

\[ moles_{\text{VC}} = M_{\text{VC}} \times V_{\text{VC solution}} \]

\[ = 0.00291485M \times 0.10000L = 0.000291485\text{moles} \]
mg. Vitamin C in sample:

\[ mg_{VC} = moles_{VC} \times 176.12 \frac{\text{grams}}{\text{mole}} \times 1000 \frac{mg}{g} \]

\[ = 0.000291485 \times 176.12 \frac{\text{grams}}{\text{mole}} \times 1000 \frac{mg}{g} = 51.3363382 mg \]

Vitamin C in sample:

\[ = \frac{mg_{VC,\text{average}}}{mass_{VC,\text{sample,grams}}} \]

\[ = \frac{51.3363382 mg}{0.5978 g} = 85.9 \frac{mg}{g} \]

Note: in final answer, volume of iodine titrant was known to 3 SF, sample mass was known to 4 SF, therefore 3 SF governs number of SF in answer.