Experiment 9

IODOMETRIC BACK TITRATION OF VITAMIN C

Text: Chapter 16.7, 7.1

Ascorbic acid (vitamin C) is a mild reducing agent that reacts rapidly with triiodide (See Section 16-7 in the textbook). In this experiment, we will generate a known excess of I⁻ by the reaction of iodate (IO₃⁻) with iodide (I⁻) (Reaction 16-18), allow the reaction with ascorbic acid to proceed, and then back titrate the excess I⁻ with thiosulfate (S₂O₃²⁻) (Reaction 16-19). The first set of titrations will be utilized to standardize the thiosulfate solutions, then this standardized solution will be used to analyze for Vitamin C in an unknown.

The reaction stoichiometry is a little more complicated than you might be used to. You should be able to work through the calculations, but it may take some time. Your instructor will not work through the calculations for you beforehand. You should figure it out yourself to the extent possible.

Preparation and Standardization of Thiosulfate Solution

1. Prepare 500 mL of 0.07 M Na₂S₂O₃ from Na₂S₂O₃·5H₂O in 500 mL of freshly boiled water containing ~0.05 g of Na₂CO₃. Store this solution in a tightly capped amber bottle.

2. Prepare 500.0 mL of ~0.01 M KIO₃ by accurately weighing the solid primary standard reagent and dissolving it in a 500-mL volumetric flask.

3. Standardize the thiosulfate solution as follows:

   Pipet 50.00 mL of KIO₃ solution into an Erlenmeyer flask. Add ~2 g of solid KI and ~10 mL of 0.5 M H₂SO₄. Immediately titrate with thiosulfate until the solution has lost almost all its color (pale yellow). Then add 2 mL of starch indicator and complete the titration. Upon addition of the starch indicator the solution should become purple. Titrate until the solution is clear. Repeat the titration with two additional 50.00-mL volumes of KIO₃ solution.

Some information to help in your calculations:

A primary standard KIO₃ solution is made, so the known volume of KIO₃ used translates to a known number of moles of IO₃⁻ delivered. The IO₃⁻ is then converted to I₃⁻ by reaction with excess I⁻ in acidic solution, according to the reaction:

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

The I₃⁻ generated from the above reaction is then titrated in a direct titration with thiosulfate to standardize the thiosulfate solution:

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

Determine the number of moles of I₃⁻ generated from the known number of moles of IO₃⁻. Further, determine the number of moles of S₂O₃²⁻ required to react with I₃⁻ during the titration. Using the thiosulfate volume in the titration should provide the molarity of the S₂O₃²⁻ solution.
Analysis of Vitamin C Unknown

Perform the following analysis at least 3 times, and find the mean value (and relative standard deviation) for the mass % Vitamin C in the unknown.

1. Accurately weigh approximately 1.2 g of unknown as obtained (do not dry since the ascorbic acid can oxidize in the oven) in 250.00 mL of 0.3 M H₂SO₄.

2. Titrate 50.00 mL aliquots of this sample by first adding 2 g of solid KI and 50.00 mL of standard KIO₃. Then titrate with standard thiosulfate as above. Add 2 mL of starch indicator just before the end point.

Some information to help in your calculations:

This is a back-titration. Once again a known number of moles of I₃⁻ is generated from the primary standard KIO₃ solution and KI (reaction 16-18 in the text). The Vitamin C (ascorbic acid) dissolved in the solution reacts (oxidizes) the I₃⁻ with a 1:1 reaction stoichiometry as shown on page 343 of the text. This is a back-titration because there is more I₃⁻ present than there is Vitamin C to react with. Therefore some I₃⁻ remains unreacted in the solution even after all of the Vitamin C has reacted. The excess I₃⁻ is then back-titrated with the standardized thiosulfate solution as shown by Equation 16-19 in the text.

Since the number of moles of Vitamin C is needed to find the mass of Vitamin C in the unknown analyzed, it is important to know how much Vitamin C reacted with I₃⁻. At this point all that is known is that there is more I₃⁻ than Vitamin C in the tablet. However you can determine the number of moles of I₃⁻ added initially to the Vitamin C solution. Also, from the titration data, the number of moles of I₃⁻ in excess after reaction with Vitamin C can be determined. Thus the number of moles of Vitamin C reacted with I₃⁻ can be determined by the difference between the number of moles added and the number of moles in excess.

Fill out and turn in the results sheet on the following page.
Vitamin C Back-Titrination Results Sheet

Name: _______________________

KIO₃ mass (g) __________

KIO₃ Molarity ____________

Unknown # ________________

Unknown mass ______________

<table>
<thead>
<tr>
<th>Titration Number</th>
<th>KIO₃ volume (mL)</th>
<th>Na₂S₂O₃ volume (mL)</th>
<th>Na₂S₂O₃ Molarity</th>
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Mean ± Standard Deviation of Na₂S₂O₃ Molarity ______________

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<thead>
<tr>
<th>Titration #</th>
<th>Moles I₃⁻ added</th>
<th>Na₂S₂O₃²⁻ back-titration volume (mL)</th>
<th>Moles I₃⁻ excess</th>
<th>Moles Vit. C</th>
<th>Mass Vit. C</th>
<th>Mass % Vit C</th>
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Average Mass % Vitamin C mass of unknown ± absolute standard deviation ______________

Average Mass % Vitamin C of unknown ± 95% confidence interval ______________