

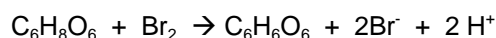
EXPERIMENT 6 DETERMINATION OF ASCORBIC ACID IN VITAMIN C TABLETS BY TITRATION WITH POTASSIUM BROMATE

A. Prelab

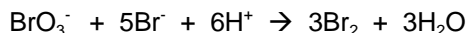
- Read Skoog et al. sections 27I (pp. 755-757) and 18C-4 (pp. 461-465) for background information. Come to lab familiar with the procedure and theory behind the procedure
- Perform the experimental portion of the lab with a partner.
- You have two weeks to complete the experimental portion of the lab.

B. Experimental Overview

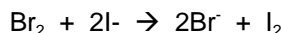
The quantitative determination of ascorbic acid (mw=176.12 g/mol) is based on the following reaction:



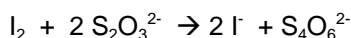
In the above reaction ascorbic acid is oxidized to dehydroascorbic acid by bromine. The production of a known amount of bromine is achieved by adding an excess of KBr to an acidified solution of the sample and titrating with standard KBrO_3 . This titration produces Br_2 according to the following reaction:



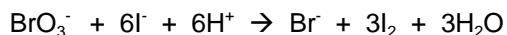
As it is produced, the Br_2 is consumed by reaction with the ascorbic acid until all of the ascorbic acid has reacted. Excess Br_2 is indicated by a faint yellow color. The amount of excess Br_2 can then be determined by using the following reaction to stoichiometrically convert an excess of I^- to I_2 :



The amount of I_2 produced is back-titrated with standard sodium thiosulfate according to:



The elimination of all I_2 is indicated by a change in color using a starch indicator. This second titration requires the standardization of a sodium thiosulfate solution. This is performed beforehand with primary standard potassium bromate according to:



The wt-% ascorbic acid in the sample and mass of ascorbic acid in an average tablet can then be calculated.

C. Procedure

Prepare 0.015 M Potassium Bromate

- Dry ~ 1.5 g of KBrO_3 (mw=167.001 g/mol) at 110°C for 1 hr and cool in a dessicator. Weigh (to the nearest 0.1 mg) 1.25 g KBrO_3 into a 500 mL volumetric flask. Dissolve the KBrO_3 in ~200 mL of water, dilute to the mark, and mix thoroughly.

Prepare ~ 0.1 M Sodium Thiosulfate

- Boil ~250 mL of distilled water for 10 min. Allow water to cool to room temperature; then add 6.25 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ (mw=248.186 g/mol) and 0.1 g of Na_2CO_3 . Stir until the solid has dissolved. Transfer to a clean glass container and store in a dark place.

Standardize Sodium Thiosulfate against Potassium Bromate

- Pipet 25-mL aliquot of standard KBrO_3 solution into a 250-mL Erlenmeyer flask and rinse the vessel wall with water. Treat each sample separately beyond this step.
- Add 2-3 g of KI and 10 mL 1.5 M H_2SO_4 and swirl to dissolve solid. Immediately titrate with your sodium thiosulfate standard until the solution is pale yellow. At this point add 5 mL of starch indicator and continue to titrate with constant stirring until the disappearance of the blue color.
- Perform triplicate standardization procedures
- Calculate the mean molarity of the sodium thiosulfate solution.

Weigh and Prepare Sample

- Individually weigh and record the mass of 3 vitamin C tablets.
- Grind and homogenize the 3 vitamin C tablets in a mortar and transfer the powder to a dry weighing bottle.
- Weigh individual 0.4 g samples (to the nearest 0.0001 g) into dry 250-mL Erlenmeyer flasks. Treat each sample individually beyond this step.

Titrate with Potassium Bromate

- Dissolve the sample in 50 mL of 1.5 M H_2SO_4 ; add ~5 g KBr and titrate immediately with standard KBrO_3 solution to the first faint yellow due to excess Br_2 . Record the volume of KBrO_3 solution used.
- Add 3 g KI and 5 mL of starch indicator; back titrate with 0.1 M Sodium Thiosulfate.
- Perform triplicate determinations
- Calculate the average mass in mg of ascorbic acid.

D. Lab Report

- Title Page (include lab title, your name, your section, and unknown #)
- Purpose (be brief, 1 paragraph)
- **Method** (describe the method in terms of the chemical reactions used, ~1 page)
- Data
- Calculations (show representative calculation of each type)
- Result

This report will be due one week after completion.