13. Iodimetric Titration of Vitamin C[13]

Ascorbic acid (vitamin C) is a mild reducing agent that reacts rapidly with triiodide (See Section 16-3 and Box 16-2 in the textbook). In this experiment, we will generate a known excess of I_3^- by the reaction of iodate with iodide (Reaction 16-20), allow the reaction with ascorbic acid to proceed, and then back titrate the excess I_3^- with thiosulfate (Reaction 16-21 and Color Plate 12).

Reagents

Starch indicator: Make a paste of 5 g of soluble starch and 5 mg of Hg_2I_2 in 50 mL of distilled water. Pour the paste into 500 mL of boiling distilled water and boil until it is clear.

Sodium thiosulfate: 9 g $Na_2S_2O_3 \cdot 5H_2O$/student.

Sodium carbonate: 50 mg Na_2CO_3/student.

Potassium iodate: 1 g KIO_3/student.

Potassium iodide: 12 g KI/student.

0.5 M H_2SO_4: 30 mL/student.

Vitamin C: Dietary supplement containing 100 mg of vitamin C per tablet is suitable. Each student needs six tablets.

0.3 M H_2SO_4: 180 mL/student.

Preparation and Standardization of Thiosulfate Solution

1. Prepare starch indicator by making a paste of 5 g of soluble starch and 5 mg of HgI_2 in 50 mL of water. Pour the paste into 500 mL of boiling water and boil until it is clear.
2. Prepare 0.07 M $Na_2S_2O_3$[14] by dissolving ~8.7 g of $Na_2S_2O_3 \cdot 5H_2O$ in 500 mL of freshly boiled water containing 0.05 g of Na_2CO_3. Store this solution in a tightly capped amber bottle. Prepare ~0.01 M KIO_3 by accurately weighing ~1g of solid reagent and dissolving it in a 500-mL volumetric flask. From the mass of KIO_3 (FM 214.00), compute the molarity of the solution.

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13 D. N. Bailey, *J. Chem. Ed*. **1974**, *51*, 488.

14 An alternative to standardizing $Na_2S_2O_3$ solution is to prepare anhydrous primary standard $Na_2S_2O_3$ by refluxing 21 g of $Na_2S_2O_3 \cdot 5H_2O$ with 100 mL of methanol for 20 min. Then filter the anhydrous salt, wash with 20 mL of methanol, and dry at 70˚C for 30 min. [A. A. Woolf, *Anal. Chem*. **1982**, *54*, 2134.]

3. Standardize the thiosulfate solution as follows: Pipet 50.00 mL of KIO_3 solution into a flask. Add 2 g of solid KI and 10 mL of 0.5 M H_2SO_4. 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. Repeat the titration with two additional 50.00-mL volumes of KIO_3 solution. From the stoichiometries of Reactions 16-20 and 16-21, compute the average molarity of thiosulfate and the relative standard deviation.

Analysis of Vitamin C

Commercial vitamin C containing 100 mg per tablet can be used. Perform the following analysis three times, and find the mean value (and relative standard deviation) for the number of milligrams of vitamin C per tablet.

1. Dissolve two tablets in 60 mL of 0.3 M H_2SO_4, using a glass rod to help break the solid. (Some solid binding material will not dissolve.)

2. Add 2 g of solid KI and 50.00 mL of standard KIO_3. Then titrate with standard thiosulfate as above. Add 2 mL of starch indicator just before the end point.