

## Experiment #9 Redox Titrations

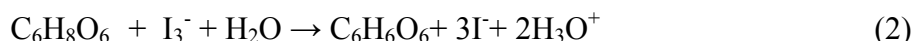
### Iodimetric Titration of Vitamin C<sup>1</sup>

Ascorbic acid (C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>, vitamin C) is a mild reducing agent that reacts rapidly with triiodide

In this experiment, we will generate a known excess of I<sub>3</sub><sup>-</sup> by the reaction of iodate with iodide:



allow the reaction with ascorbic acid to proceed,



and then back titrate the excess I<sub>3</sub><sup>-</sup> with thiosulfate:



#### Reagents:

**Starch indicator:** Make a paste of 5 g of soluble starch and 5 mg of Hg<sub>2</sub>I<sub>2</sub> 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<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O/student.

**Sodium carbonate:** 50 mg Na<sub>2</sub>CO<sub>3</sub>/student.

**Potassium iodate:** 1 g KIO<sub>3</sub>/student.

**Potassium iodide:** 12 g KI/student.

**0.5 M H<sub>2</sub>SO<sub>4</sub>:** 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<sub>2</sub>SO<sub>4</sub>:** 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 Hg<sub>2</sub>I<sub>2</sub> 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<sub>2</sub>S<sub>2</sub>O<sub>3</sub>** by dissolving 8.7 g of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O in 500 mL of freshly boiled water containing 0.05 g of Na<sub>2</sub>CO<sub>3</sub>. Store this solution in a tightly capped amber bottle. **Prepare 0.01 M KIO<sub>3</sub>** by accurately weighing 1.0000 g of solid reagent and dissolving it in a 500-mL volumetric flask. From the mass of KIO<sub>3</sub> (FM 214.00), compute the molarity of the solution.

**3. Standardize the thiosulfate solution as follows:** Pipet 50.00 mL of  $\text{KIO}_3$  solution into a flask. Add 2 g of solid KI and 10 mL of 0.5 M  $\text{H}_2\text{SO}_4$ . 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  $\text{KIO}_3$  solution. From the stoichiometries of Reactions (1) and (3), 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  $\text{H}_2\text{SO}_4$ , 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  $\text{KIO}_3$ . Then titrate with standard thiosulfate as above. Add 2 mL of starch indicator just before the end point.

### **References:**

1. D. N. Bailey, J. Chem. Ed. 1974, 51, 488.
2. Skoog et al., 7<sup>th</sup> ed., Chap. 18
3. D.C. Harris 6<sup>th</sup> ed., Chap. 16