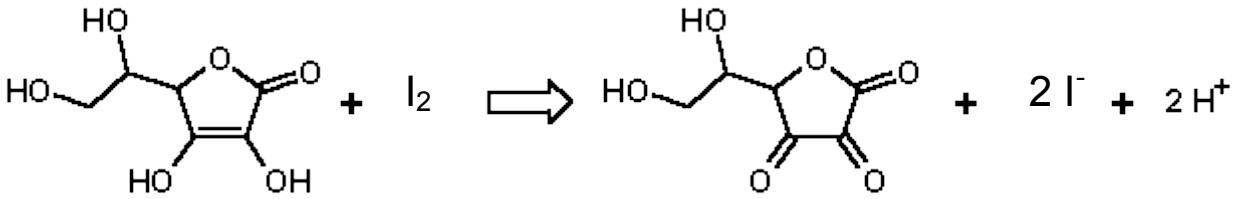
Determination of Ascorbic Acid in Vitamin C Tablets

Introduction:
Ascorbic acid, $C_6H_8O_6$, is a reducing agent and can be oxidized to form dehydroascorbic acid by iodine via the following reaction (1):



The iodine used in the titration is generated by adding an excess amount of KI to an acidified solution of potassium iodate according to the following reaction:

$$IO_3^- + 5\,I^- + 6\,H^+ \rightleftharpoons 3\,I_2 + 3\,H_2O \qquad (2)$$

Sodium thiosulfate solutions can be standardized by direct titration of the I_2 generated in the KIO_3 reaction using the starch-iodine complex as the indicator (remember that the iodine is actually in the form of the triiodide ions in the presence of excess KI due to the $I_2 + I^- \rightleftharpoons I_3^-$, equilibrium.)

In the determination of ascorbic acid, the KIO_3 method employs a **back titration** with a standard thiosulfate solution of the **excess** I_2 generated by adding a precisely known amount of a primary standard KIO_3 solution to an ascorbic acid solution. The excess iodine (what is left behind after reaction 1 has occurred) is back-titrated with standard sodium thiosulfate.

$$I_2 + 2\,S_2O_3^{-2} \rightleftharpoons 2\,I^- + S_4O_6^{-2} \qquad (3)$$

Ascorbic acid can undergo air oxidation requiring that the procedure be performed with minimal delay.

Before coming to lab:

- Read chapter 13, sections 13A-13C in your textbook.
- You'll need to know how to do the calculations for a back titration. See Chapter 13 pp. 348-349 for examples. **This is different** from previous (direct titration) laboratory calculation.
- Write reactions 1, 2, and 3 (above) in your lab notebook.
- Record the molecular weight for potassium iodate in your notebook.
- Record the molecular weight for ascorbic acid in your notebook.
- List all of the reagents (formulas and names) in your notebook. Familiarity with the reagents will help you avoid costly mistakes during the experiment.

Reagents:

- Potassium iodide
- Potassium iodate
- 0.5 M H_2SO_4
- starch solution

Preparation of a 0.1 M Sodium Thiosulfate Solution

Only prepare this solution if you did not do so last week.
Make this solution in your 1 L plastic bottle and label it with tape. Dissolve about 25 g of sodium thiosulfate pentahydrate crystals in 1 liter of distilled water. Add about 0.2 g of sodium carbonate as a preservative. Shake to ensure complete dissolution and mixing.

Preparation of Standard 0.010 M Potassium Iodate

Weigh approximately 2.1 g (to the nearest 0.1 mg) of dried and cooled reagent grade KIO_3. Transfer quantitatively to a 1000.0 mL volumetric flask. Initially, dissolve the KIO_3 in about 200 mL of distilled water. Dilute to the mark and mix thoroughly. Calculate the concentration of the KIO_3 solution.

Standardization of 0.10 M sodium thiosulfate (not necessary if you standardized your solution last week and achieved good results)

1. Pipette 50.00 mL of the KIO_3 solution into a 250 mL Erlenmeyer flask.
2. Add 2 g of solid KI and 10 mL of 0.5 M H_2SO_4.
3. Immediately titrate with the thiosulfate solution until the solution has lost its initial reddish-brown color and has become pale yellow.
4. Add 2 mL of starch indicator and complete the titration.
5. Repeat the titration twice or until the concentration of the thiosulfate agrees to within ±1%.

Determination of Ascorbic Acid in Vitamin C tablets (at least three replicates)

6. Record your unknown number. Record the number of tablets and the total weight of the tablets to the nearest 0.1 mg before grinding the sample.
7. Grind all of the tablets thoroughly using a clean mortar and pestle. Transfer the ground powder to a clean, dry weighing bottle.
8. Prepare three flasks in the following way. Weigh (to the nearest 0.1 mg) a 0.25 g sample into a 250 mL Erlenmeyer flask. Dissolve sample in 20 mL of 0.5 M H_2SO_4. Some solid binding material in the tablet formulations may not dissolve completely.

At this point, you should prepare and titrate your samples one at a time.

9. Add approximately 2 g KI and exactly 50.00 mL of standard KIO_3 to each sample.
10. Immediately titrate with thiosulfate until the solution has lost its initial reddish-brown color and has become pale yellow although it may be difficult to see these colors with other components of the tablets. When in doubt, add the starch early in the titration.
11. Add 2 mL of starch indicator and complete the titration. If starch binder has been used to manufacture the tablets, the characteristic blue color of the starch-iodine complex will appear upon the addition of KI.

Typically about 8 to 14 mL of the thiosulfate solution will be needed for the first sample. Using a **smaller** amount of unknown sample will **increase** the number of mL of thiosulfate required.

Because this is a back titration, the 50.00 mL of KIO3 will produce the same number of moles of I_2 in the unknown solution as in the standardization titration. The I_2 from the KIO_3 will react with ascorbic acid so that the amount of I_2 actually titrated will be the difference between the moles of I_2 generated from the KIO_3 and the moles of ascorbic acid reacting with the I_2, such that:

I_2 reacting with ascorbic acid = I_2 generated - I_2 titrated

Calculate the **% ascorbic acid** in your unknown at this point. Report the result in terms of the percentage of ascorbic acid in your unknown. The range of unknown values should be 50% to 90%.

SAVE YOUR STANDARDIZED THIOSULFATE SOLUTION FOR THE NEXT EXPERIMENT