

DETERMINATION OF ASCORBIC ACID IN VITAMIN C TABLETS BY REDOX TITRATION

Background reading: Harris, 7th ed., Chap. 16. Skoog et al., 7th ed., Chap. 18;

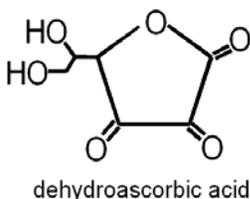
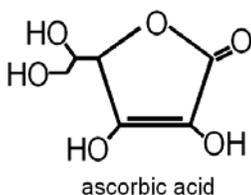
Introduction

Iodine is a versatile redox reagent because its potential falls in the middle of the range of potentials observed in aqueous solutions. Thus in the presence of strong oxidants, such as dichromate, iodide is oxidized to iodine; in the presence of reducing agents, such as As (III), iodine is reduced to iodide.

Solid I_2 is only slightly soluble in water, but in the presence of excess iodide it forms the soluble triiodide ion, I_3^- , and it is in this form that it is used for redox titrations. Reducing agents are determined by direct titration with standard I_3^- . For the determination of oxidizing agents it is not feasible to titrate directly with standard iodide, because a high concentration of iodide is needed to form the I_3^- complex. Instead excess iodide is added to oxidizing agents, and the excess I_3^- formed is titrated with a standard solution of a reducing agent, thiosulfate, $S_2O_3^{2-}$.

An advantage to all of these analyses is the ready availability of a specific indicator, starch. I_3^- reacts with starch to form an intense blue color that is visible even at very low I_3^- concentrations. In direct titrations with I_3^- the endpoint is signaled by the appearance of the blue color when the first trace of I_3^- is produced after the equivalence point. In titrations of triiodide with thiosulfate, the endpoint is signaled by the disappearance of the blue color. Care must be taken, however, to add the starch after most of the I_3^- has already reacted. In the presence of large I_3^- concentrations a rather stable complex forms, and the blue color persists beyond the equivalence point.

In this exercise you will determine the weight percent of ascorbic acid, Vitamin C, in Vitamin C tablets. Ascorbic acid, $C_6H_8O_6$, (MW = 176.12) is oxidized by iodine, to dehydroascorbic acid, $C_6H_6O_6$. The I_3^- will be generated *in situ* by adding a known volume of a standard iodate, IO_3^- solution to a solution of ascorbic acid and iodide. The iodate oxidizes the iodide to form I_3^- , which reacts in turn with the ascorbic acid. The excess I_3^- is titrated with a standard solution of $S_2O_3^{2-}$. From the moles of I_3^- produced, calculated from the volume of standard IO_3^- solution added, and the moles of excess I_3^- , calculated from the volume of standard $S_2O_3^{2-}$ used, the moles of ascorbic acid can be calculated.



The concentration of the standard iodate solution can be calculated directly from the weight of KIO_3 used. The thiosulfate solution must be standardized. The standardization is carried out against the standard iodate solution. A known volume of the standard iodate solution is added to an excess of

iodide, generating a known amount of I_3^- . The I_3^- is titrated with the $S_2O_3^{2-}$ to a starch endpoint.

Care must be taken in preparing and storing thiosulfate solutions. Although sodium thiosulfate solutions are resistant to air oxidation, they do tend to decompose to give sulfur and hydrogen sulfite ion. Variables that influence the rate of decomposition include pH, the presence of microorganisms, the presence of Cu (II) ions, and exposure to sunlight. To minimize the need for restandardization of the thiosulfate solution, it will be prepared under reasonably sterile conditions, at a pH between 9 and 10, and stored in the dark.

Procedure

Preparation of standard 0.02 M KIO₃ solution. Obtain from the instructor a weighing bottle containing dry KIO₃.

Accurately weigh by difference about 2.0 g of the solid into a small beaker and transfer it quantitatively to a 500 mL volumetric flask. Add water and stir to dissolve solid. Dilute to the mark and mix thoroughly.

Calculate and record the molar concentration of the solution.

Preparation and standardization of 0.1 M Na₂S₂O₃. Boil about 500 mL of distilled water for 10 to 15 min. Allow the water to cool to room temperature; then add about 12 g of Na₂S₂O₃·5H₂O and 0.05 g of Na₂CO₃. Stir until the solid has dissolved. Transfer the solution to a clean glass or plastic bottle and store in a dark place.

Pipet 25 mL of the KIO₃ solution into each of three 250 mL Erlenmeyer flasks. From this point treat each of the flasks separately until the titration is finished.

Add 2 g of solid KI and 10 mL of 0.5 M H₂SO₄ to the first flask and immediately begin titrating with the Na₂S₂O₃ solution. When the titration solution has lost almost all of its color (only a pale yellow remains), add 2 mL of starch indicator. Continue titrating until the blue color disappears.

Repeat for the other two flasks. If the range of titration volumes is greater than about 0.20 mL perform another standardization titration.

Calculate and record the molar concentration of the solution.

Determination of Ascorbic Acid. Crush a Vitamin C tablet and accurately weigh about 100 mg of the powder into each of three Erlenmeyer flasks. Treat each individually as follows.

Add 40 mL of 0.5 M H₂SO₄ and 20 mL of water to dissolve the tablet. (Use a stirring rod to break up any large particles but some solid material used as a binder in the tablet may not dissolve.)

When the tablet is dissolved, add 2 grams of KI and, using a transfer pipet, 25 mL of the KIO₃ solution.

Titrate with the thiosulfate solution just as you did in standardizing the thiosulfate solution. The samples may be colored, making it somewhat more difficult to judge when to add the starch indicator. From the first titration and the weight of sample calculate how much thiosulfate can be added to each of your remaining samples before the indicator is added.

Repeat for the remaining samples.

For each sample find the weight percent of ascorbic acid in the tablet.

Report

Report the weight percent ascorbic acid for each of the three determinations. Report the average and standard deviation for the three determinations. Report your best estimate of the weight percent ascorbic acid in your unknown.