Chem.243-Exp.8 (2019)

Determination of ascorbic acid in vitamin C tablets

Introduction:

Many chemical reactions proceed by an electron transfer from one reactant to another. These electron-transfer reactions are referred to as oxidation-reduction or redox reactions. Oxidation is defined as the part of a redox reaction in which a species loses electrons and increases in oxidation number. Reduction is the part of the redox reaction in which a species gains electrons and decreases in oxidation number. In any reaction in which oxidation occurs, reduction must also occur. An oxidizing agent is a species that oxidizes another species and is itself reduced. A reducing agent is a species that reduces another species and is itself oxidized.

Ascorbic acid (Vitamin C) is a relatively cheap, structurally simple, water soluble organic acid--best known for being commonly found in citrus fruits such as oranges, limes, and lemons. The molecular formula for ascorbic acid is $C_6H_8O_6$, It occurs as a white or slightly yellow crystal or powder. To determine the quantity of Vitamin C (ascorbic acid) found in commercially available Vitamin C tablets, an oxidation-reduction titration usually conducted, in which an oxidizing agent is used to oxidize the ascorbic acid.

A standard solution of iodine usually used as an oxidizing agent for the determination of ascorbic acid, a standard solution of iodine can be prepared by weighing the iodine exactly on an analytical balance or by standardizing it against primary standard arsenic (III) oxide. Since it's difficult to handle iodine without losing some of it, it is usually weighed out approximately and standardized. The arsenic (III) oxide is first dissolved in base and then neutralized to arsenious acid:

$$As_2O_{3(s)} + 2OH^- + H_2O \rightarrow 2H_2AsO_3^- + 2H^+ \rightarrow 2H_3AsO_3$$

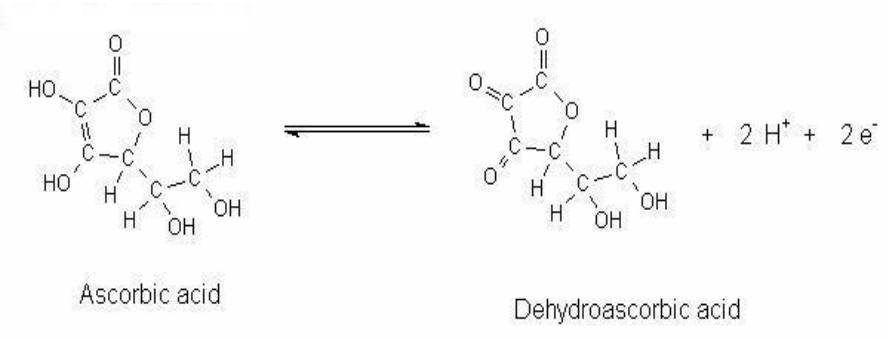
The arsenious acid is then oxidized by the iodine titrant to arsenic acid in a solution buffered at about pH 8 with sodium bicarbonate:

$$I_2 + H_3AsO_3 + H_2O \rightarrow 2I^- + H_3AsO_4 + 2H^+$$

The end point is detected by the formation of the deep blue starch-triiodide color. Since iodine exists as the triiodide (I_3^-) ion in aqueous solution, the first excess of iodine titrant added will form the starch-triiodide complex.

To analyze for ascorbic acid the iodine standard solution is used to oxidize the ascorbic acid quantitatively to dehydroascorbic acid:

$$I_2 + C_4H_6O_4(OH)C{=}COH \rightarrow 2I^- + 2H^+ + C_4H_6O_4C(=O){-}C{=}O$$



Similarly starch solution is used as an indicator and the end point is detected by the formation of starch-triiodide complex.

Note: Since oxygen is a stronger oxidizing agent (E° = 1.23 V) than iodine (E° = 0.535 V), dissolved oxygen oxidizes ascorbic acid also, but much more slowly. Therefore, the samples should be titrated immediately after being dissolved and the flask should be covered to minimize absorption of oxygen from the air.

In this experiment you will determine the amount of ascorbic acid in a vitamin C tablet and calculate the percentage, a standard solution of iodine will be used as the oxidizing agent and starch solution will be the indicator.

Procedure:

Part I: Preparation and standardization of Iodine:

1. Prepare 0.015M iodine for vitamin C analysis by weighing about 2.3 g of reagent grade iodine on a top scale and transfer it to a 100-ml beaker containing 10 g of potassium iodide dissolved in 20 ml of water. Stir carefully to dissolve all the iodine, and pour the entire contents into a glass stoppered amber liter bottle. Rinse the beaker with 25 ml of distilled water and pour this into the bottle. Dilute the mixture to about 500 mL using distilled water and shake several times.
2. Prepare 0.015 M arsenious acid solution by weighing exactly 0.150 g of primary standard arsenic (III) oxide into 250 ml beaker. (Avoid touching this chemical or ingesting it; wash your hands if you have any reason to believe you have touched it.). Add a solution of 0.5 g of NaOH pellets freshly dissolved in 10 ml of distilled water. Swirl until the

oxide dissolves completely, warming if necessary. Add 25 ml of water and 1 ml of 12 M HCl. Transfer quantitatively to a 100 ml volumetric flask and dilute to volume.

3. Using a bulb, pipet exactly 20 ml of 0.015 M arsenious acid into each of three flasks. Add 25 ml of water to each. Then add about 4 g of sodium bicarbonate to each, and check the pH is still 7-8 with pH paper. If the pH is not in the range add more (+1 g) sodium bicarbonate.
4. To each flask, add about 5 ml of starch solution and titrate with 0.015 M iodine to the first appearance of the blue starch-triiodide color. Approach the end point cautiously, since the color of the indicator is so intense that you may obtain a very dark color with a very small excess of titrant.

Part II: Determination of ascorbic acid in vitamin C tablet

1. Weigh vitamin C tablet on an analytical balance and transfer it to a 100 ml beaker. Add about 25 ml distilled water and dissolve the tablet. Transfer quantitatively to a 100 ml volumetric flask and dilute to fill to the mark, (the vitamin C tablet contains not only ascorbic acid but many other additives that may be water insoluble so not all the tablet will dissolve in water).
2. Pipet exactly 10 ml of the vitamin C solution and transfer to a 250 ml flask and add 5 ml starch indicator. Cover the opening of the flask with a piece of cardboard with a small hole for the buret tip. Titrate rapidly to reduce air oxidation of the ascorbic acid, but proceed dropwise near the end point, a deep blue starch-triiodide color.
3. Repeat the titration for 2 additional samples. Do not pipet a sample into a flask until you are ready to titrate it.

Pre laboratory assignment: determination of ascorbic acid in vitamin C tablets

1. A vitamin C tablet, claimed to contain 500 mg on the label, is dissolved and titrated with 28.54 ml of 0.1000 M iodine. Calculate the mg of vitamin C (Formula weight = 176.12 g/mol) in the tablet.

2. In the following reaction determine the reducing and oxidizing agents:

   a) $I_2 + H_3AsO_3 + H_2O \rightarrow 2I^- + H_3AsO_4 + 2H^+$

   b) $2Zn(s) + O_2(g) \rightarrow 2ZnO(s)$

The determination of ascorbic acid in vitamin C tablet

Data and calculations:

Name: _____ID:_____

Partner's name: _____Date: _____

General observations

Did the tablet dissolve completely? **……..……**

Color of the solution before adding starch indicator. **…….…….**

Color of the solution after adding starch indicator. **……..**

Data and results

Part I: Preparation and standardization of Iodine

Mass of arsenic (III) oxide **…….……..g**

Moles of arsenic oxide **…….……..mol**

Moles of arsenious acid **…….……..mol**

$$As_2O_{3(s)} + 2OH^- + H_2O \rightarrow 2H_2AsO_3^- + 2H^+ \rightarrow 2H_3AsO_3$$

Concentration of arsenious acid **…….……..M**

	Trial #1	Trial #2	Trial #3
Initial volume of iodine (ml)	……	……	……
Final volume of iodine (ml)	……	……	……
Net volume of iodine (ml)	……	……	……

Iodine average volume ……. mL

Concentration of Iodine ……. M

$$I_2 + H_3AsO_3 + H_2O \rightarrow 2I^- + H_3AsO_4 + 2H^+$$

Sample calculations:

Part II: Determination of ascorbic acid in vitamin C tablet

Mass of vitamin C tablet **……..……..g**

	Trial #1	Trial #2	Trial #3
Initial volume of iodine (ml)	……	……	……
Final volume of iodine (ml)	……	……	……
Net volume of iodine (ml)	……	……	……
Iodine average volume (ml)		……	

Concentration of ascorbic acid …… M

$$I_2 + C_4H_6O_4(OH)C{=}COH \rightarrow 2I^- + 2H^+ + C_4H_6O_4C(=O){-}C{=}O$$

Mass of ascorbic acid in 100 ml solution …… g

Percent ascorbic acid in vitamin C tablet …… g

Sample calculation: