

Vitamin C Analysis (Redox Titration)

Stephanie R. Geggier
New York University, Department of Chemistry

PURPOSE: The goal of this experiment is to determine the ascorbic acid content in juice and a vitamin C tablet using redox titration with 2,6-dichloroindophenol.

Required Reading & Videos

- Ref. (1), section 4.4 (Oxidation-Reduction (Redox) Reactions), solve sample problem 4.18
- Very important:** Read sections G and H of *Basic Laboratory Skills and Equipment*, review section F
- Video: Making a Solution Using a Volumetric Flask
- Video: Using an Automatic Pipet

Introduction

Ascorbic acid (AA), commonly known as vitamin C, is an organic compound of carbon, hydrogen and oxygen that occurs naturally in fruits and vegetables. It is an essential nutrient in the diet but is readily oxidized or destroyed when exposed to heat and oxygen during food processing, packaging and storage. Vitamin C deficiency causes scurvy - a widespread sailor's disease until the early 19th century, with serious symptoms including bleeding of the mucous membranes, anemia and eventually death. In 1747, James Lind, a naval surgeon, conducted an experiment with sick sailors. He discovered that citrus fruits cured scurvy. In 1928, vitamin C was isolated for the first time, and five years later its chemical structure was determined. Vitamin C was named ascorbic acid to indicate its antiscorbutic (anti-scurvy) properties. Soon after its discovery, an industrial process was developed to produce ascorbic acid from glucose. Today it is the most produced vitamin in the world exceeding 100,000 tons per year (2).

Titrimetric Analysis of Vitamin C

AA is not only used as a dietary supplement but also as a food preservative. It acts as an antioxidant — a substance that inhibits oxidation and free radical damage to cells. When AA is involved in redox reactions, it plays the role of a reducing agent by donating two electrons:



In this reaction, AA is oxidized to dehydroascorbic acid. The content of AA in samples can be determined by titrating the samples with 2,6-dichloroindophenol (DCP). DCP is also an organic compound and acts as an oxidizing reagent.



In this reaction, DCP is reduced to leuco dye. Combining these two chemical equations, we obtain the equation for the reaction of AA with DCP:



DCP is a blue dye, but it is pink under acidic conditions. In a titration with blue DCP solution, the dye initially turns pink when it enters the acidic solution, but then the color is immediately removed because it is reduced to the colorless leuco dye. Once the titration is complete, a fraction of a drop of excess DCP solution imparts a pink color to the solution. The error introduced by the excess DCP is negligible. DCP is very reactive and decomposes over time. Therefore, DCP solutions must be freshly prepared and standardized just prior to use. **Standardization** is the titration of a titrant with a weighed quantity of a primary standard. With the known amount of the primary standard used, the concentration of the titrant can be calculated. In this experiment, you will standardize the DCP solution with reagent-grade AA. The titration is performed in the presence of metaphosphoric acid and acetic acid (MPA-HOAc solution) to stabilize AA. Keep in mind that this experiment is quantitative. Work accurately and transfer solids and liquids quantitatively. All students must demonstrate their ability to use an automatic pipet prior to the experiment.

Apparatus

125-mL Erlenmeyer flask, buret, automatic pipet & tip, 50-mL volumetric flask, 100-mL volumetric flask, funnel, mortar and pestle, and graduated cylinders.

Chemicals

- MPA-HOAc solution (contains 3% metaphosphoric acid and 3.4% acetic acid): Toxic and corrosive. Causes severe skin burns and eye damage.
- Ascorbic acid: not hazardous
- 2,6-Dichloroindophenol solution (the molarity of DCP will be determined in Part I): May cause eye, skin, and respiratory tract irritation. Spell on skin. Immediately flush skin with excess water for 15 minutes while removing contaminated clothing. This solution also contains 0.004 M NaHCO₃.

Experimental Procedure

Part I. Standardizing the DCP Solution

- Using a 50-mL volumetric flask, prepare an aqueous solution that is approximately 0.005 M in AA (see video and required reading for instructions). Include the procedure and calculations in your notebook **prior to lab**. Record the mass of AA accurately and recalculate the molarity. Pour the solution into a beaker.
- The automatic pipet (AP) at your station should **only** be used to transfer AA solution and Vitamin C tablet solution (Part III). Rinse the tip between different solutions. The AP is a delicate and expensive instrument. Handle it carefully and follow the instructions! Transfer 2.50 mL of your AA stock solution to a 125-mL Erlenmeyer flask using the AP at your station.

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