Determination Weight Proportion of Ascorbic Acid (Vitamin C) in a Commercial Vitamin Supplement by Iodometric Titration

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**Abstract**: A standard solution of KIO3 (0.010 M) was prepared as a primary standard for use in an iodometric titration after converting a 25.00 mL aliquot of the iodate ion solution to I3- with excess KI. The first titration was against a solution of sodium thiosulfate of unknown concentration. After this first titration, the concentration of this solution was found to be 0.0643 M. Preparing the same solution of I3- again a second time, this time a known quantity of vitamin C tablet was made to react with the solution before the sodium thiosulfate titration, so that the vitamin C would reduce some of the I3-. By determining the leftover amount of I3- by reacting the rest with the sodium thiosulfate solution, the weight percentage of ascorbic acid in the vitamin C tablet was found to be 39.79% w/w.

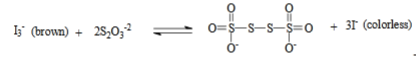
**Introduction**

Volumetric titrations are a common method of quantitative analysis, allowing for the determination of the concentration of an unknown solution using a solution of known concentration and a stoichiometric chemical equation. This technique is carried out by slowly adding one solution from a buret (called the titrant) to a known volume of the other solution in a conical flask, until the reaction is complete. The titrant can be either the solution of known concentration (called the standard solution) or the solution of unknown concentration. Finally, the completion of the reaction is usually signified by either a color change or a precipitate forming or disappearing. Common titrations are based on acid-base, redox, complex formation, or precipitation reactions in equilibrium.­1

In this experiment, iodometric titration will be used to determine the amount of vitamin C (ascorbic acid) present in the commercial tablets. This is a redox reaction between KIO3 and sodium thiosulfate, however, these two chemicals do not react directly. Sodium thiosulfate is reacted with I3-, which is generated after reaction KIO3 with excess KI, as seen in Equation 1. I3-, which has a slight brown color, then reacts with sodium thiosulfate to produce colorless I-, as seen in Equation 2.



Equation 1: Redox Reaction of Iodate and Iodide in Acid

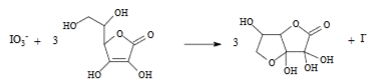


Equation 2: Redox Reaction of Triiodide and Thiosulfate

To determine the amount of vitamin C, the tablet will be added to the solution before the sodium thiosulfate to react some, but not all, of the I3-, as ascorbic acid acts as a reducing agent. This behavior seen in Equation 3, while the full reaction with IO3- is detailed in equation 4.



Equation 3: Oxidation of Ascorbic Acid



Equation 4: Combined reaction of Iodate ion and Ascorbic Acid

Thus, using this stoichiometric ratio, along with the known weight of the tablet itself, the weight percent of ascorbic acid can be determined.

**Experimental**

KIO3 (CAS#: 7758-05-6) and KI (CAS#: 7681-11-0) were purchased from Aldrich Chemical Company (St. Louis, MO). Sodium thiosulfate, starch, and H2SO4 solutions were provided by the TAs, while vitamin C tablets were from Solgor Vitamin and Herb Co. (Leonia, NJ).

To begin, KIO3 (0.538 g) was accurately weighed and dissolved to make a 250.00 mL solution of 0.010 M KIO3. A buret (50 mL) was filled with the given sodium thiosulfate solution, and an aliquot (25.00 mL) of the KIO3 solution was pipetted into an Erlenmeyer flask. The KIO3 solution was mixed with excess solid KI (0.5 g) and dilute H2SO4 (10 mL, 0.5 M). The mixture was then titrated with the NaS2O3 solution provided. When the solution began to lose color, a couple drops of starch were added to form a deep blue color. Titration continued until the blue color completely disappeared, and the titrated volume was measured and recorded. This trial was repeated two more times, for a total of three trials.

Then, a vitamin C tablet was crushed with mortar and pestle, and approximately 0.150 g was accurately weighed out and recorded. The KIO3 solution was prepared the same way as before, with the same amounts of excess solid KI and dilute H2SO4. The crushed vitamin C tablet was added to the flask, and then the solution in the flask was titrated with the same NaS2O3 solution in the same manner as the first part of this experiment, with starch being added when the solution turned a pale yellow. The titrated volume was recorded at the end of the titration. This was repeated two more times, for a total of three trials.

**Results and Discussion**

The concentration of the KIO3 solution was calculated to be 0.010 M. By combining equations 1 and 2, the result is an equation that shows the stoichiometric ratio between iodate and thiosulfate in a ratio of 1 to 6 moles.



Equation 5: Combined Equation of Potassium Iodate and Thiosulfate in Acid

From the three trials, the average volume of sodium thiosulfate solution needed to titrate the I3- generated from the 25.00 mL of 0.010 M KIO3 solution was 23.44 mL. Since there were 2.51 x 10-4 mols of KIO3 consumed in this reaction, 6 x 2.51 x 10-4 mols of sodium thiosulfate must have been consumed, or 1.51 x 10-3 mols. Thus, the concentration of the sodium thiosulfate was calculated to be 0.0643 M.

The next phase of this experiment involves reacting some of the I3- in solution with ascorbic acid and titrating the rest with the sodium thiosulfate. From the three trials, the average weight of the ascorbic acid powder used was 0.155 g, and the average amount of sodium thiosulfate titrant used was 12.57 mL. This signifies that the average amount of Na2S2O3 consumed was 8.08 x 10-4 moles, and from the previously mentioned stoichiometric ratio, the average amount of KIO3 that reacted with the sodium thiosulfate was 8.08 x 10-4 / 6 mol, or 1.35 x 10-4 mol. Subtracting this amount from the original 2.51 x 10-4 mol KIO3 means that ascorbic acid reacted with 1.17 x 10-4 moles of the iodate ion. According to equation 4, iodate and ascorbic acid react in a 1 to 3 mole ratio, which means that there was 3 x 1.17 x 10-4 moles of ascorbic acid, or 3.50 x 10-4 moles of ascorbic acid. Thus, with the molar mass of ascorbic acid being 176.12 g, the average weight of the ascorbic acid was calculated to be 0.062 g, which accounts for 39.79% of the commercial vitamin C tablet.

This result shows that while ascorbic acid makes up almost 40% of the weight of the tablet, the remaining 60% is lost to inactive ingredients in the tablet. While this method of determining the weight percentage only involved a simple titration, the lack of understanding of what might be in the inactive ingredients might make these results invalid, as any reducing agent would react with the I3- and produce the same result as the ascorbic acid. If there were other reducing agents in the tablet, it would factor into extra calculated mass of the vitamin C tablet, causing error in our results.

**References**

1. Harris, D. C. *Quantitative Chemical Analysis*, 8th ed.; W.H. Freeman and Co: New York, 2010. Chapter 15.