Experiment 4: Quantitative Analysis of Vitamin C

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I. Objective

The objective of this experiment is to determine the concentration of vitamin C in commercial vitamin C powder and fresh lemon juice using a redox titration method.

II. Technique

In this experiment, you will develop proficiency in essential laboratory techniques including accurately weighing chemicals, measuring liquid volumes, and handling common lab equipment such as a pipette, burette, volumetric flask, and a hot plate with magnetic stirrer.

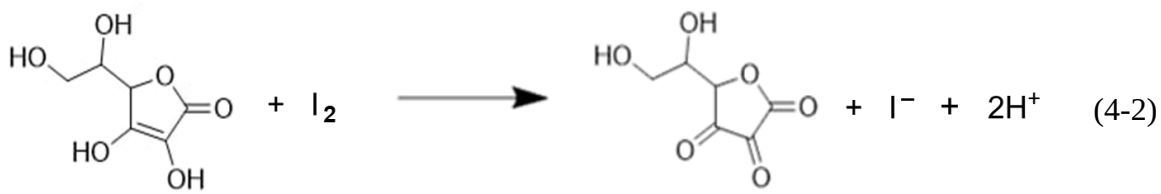
III. Introduction

Among all vitamins, vitamin C is one of the most commonly consumed as a dietary supplement, often taken to prevent deficiency and support immune function. Commercial vitamin C tablets are typically available in dosages of 100, 200, 500, or 1000 mg. However, it is natural for consumers to question whether the labeled amounts accurately reflect the actual content. In this experiment, we will evaluate the vitamin C content in various samples using redox titration.

Vitamin C, also known as ascorbic acid ($C_6H_8O_6$), acts as a reducing agent and can reduce species such as Fe(III) to Fe(II), or I_2 to I^-. In this procedure, we will utilize potassium iodate (KIO_3) as the titrant. When added to a sample solution containing sodium iodide (NaI) in an acidic medium, iodine (I_2) is generated via the following reaction (Equation 4-1):

$$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(l) \quad (4\text{-}1)$$

The iodine produced then reacts rapidly with ascorbic acid in the solution in a redox reaction (Equation 4-2)



(4-2)

Once all the ascorbic acid has been consumed, any excess I_{22} formed reacts with remaining iodide ions (I^-) to produce triiodide (I_3^-). The triiodide forms a blue-black complex in the presence of a starch indicator, signaling the end-point of the titration. By calculating the number of moles of iodate (IO_3^-) used, the vitamin C content in the sample can be quantitatively determined (Equation 4-3).

$$\frac{IO_3^-(\text{mol})}{1} = \frac{I_2(\text{mol})}{3} = \frac{C_6H_8O_6(\text{mol})}{3} \qquad (4\text{-}3)$$

IV. Equipment

Group Equipment/Materials (in Cabinet)		**Provided by Teaching Assistant**
10 mL Pipette (1)	100 mL Beaker (2)	Butterfly Clamp (1)
Safety Pipette Filler (1)	Stir Bar (1)	50 mL Burette (1)
100 mL Volumetric Flask (1)	Hot Plate Stirrer (1)	Fine Filter Screen
125 mL Erlenmeyer Flask (2)		

V. Chemicals

vitamin C powder	freshly squeezed lemon juice
1 M Sodium iodide, NaI	0.005 M Potassium iodate, KIO_3
1 M Hydrochloric acid, HCl	2% Starch solution

VI. Procedure

(A) Determination of Ascorbic Acid Content in Vitamin C Powder

1. **Sample Preparation**

   Weigh approximately 0.1 g of vitamin C powder and dissolve it in 50 mL of deionized (DI) water in a 100 mL beaker. Once fully dissolved, transfer the solution into a 100 mL volumetric flask. Rinse the beaker thoroughly and add the rinses to the flask. Dilute the solution to the calibration mark with DI water. Mix well. Transfer 25.0 mL of this solution into a 125 mL Erlenmeyer flask for titration.

2. **Reagent Addition**
   To the Erlenmeyer flask, add approximately 2.0 mL of 1 M sodium iodide (NaI), 2.0 mL of 1 M hydrochloric acid (HCl), and 1.0 mL of 2% starch indicator solution.
3. **Preparation of Titrant**
   Measure about 50 mL of 0.005 M potassium iodate (KIO_3) solution into a clean 100 mL beaker. Rinse a 25 mL burette with small portions (~5 mL) of the KIO_3 solution twice, then fill the burette with the same solution. Record the initial burette volume ($\mathbf{V_i}$) to the nearest 0.01 mL.
   - ***Note 1:*** Ensure no air bubbles remain in the burette tip. Adjust the liquid level just below the zero mark.
   - ***Note 2:*** Refer to laboratory technique videos for proper burette handling if needed.
4. **Titration**
   Titrate the prepared sample with 0.005 M KIO_3 until a permanent dark green color appears, indicating the end-point. Record the final burette volume ($\mathbf{V_f}$) to the nearest 0.01 mL.
5. **Duplicate Titration**
   Repeat the procedure with a second 0.1 g sample of vitamin C powder to perform a duplicate titration for accuracy.
6. **Calculation**
   Use the titration volume of 0.005 M KIO_3 to calculate the ascorbic acid (vitamin C) content in the sample.

(B) The ascorbic acid content of fresh lemon juice

1. **Sample Preparation**
   Each group should prepare 50 mL of freshly squeezed lemon juice, ensuring all pulp is removed by filtration.
2. **Transfer of Juice**
   Clean a 25 mL transfer pipette and rinse it twice with 5 mL portions of the lemon juice. Accurately transfer 20.0 mL of the filtered lemon juice into a 125 mL Erlenmeyer flask.
3. **Reagent Addition**
   Add approximately 2.0 mL of 1 M NaI, 2.0 mL of 1 M HCl, and 1.0 mL of 2% starch indicator to the flask.

4. **Titration**
   Titrate the lemon juice sample with 0.005 M KIO_3, recording the volume used to the nearest 0.01 mL at the dark green end-point.
5. **Duplicate Titration**
   Prepare a second 20.0 mL aliquot of pulp-free lemon juice and repeat the titration.
6. **Calculation**
   Calculate the ascorbic acid content of the fresh lemon juice sample using the titration data.
7. **Cleanup**
   Thoroughly rinse the burette and transfer pipette with water. Invert the burette, clamp it securely, and allow it to air dry.

References

1. Roberts, J. L., Jr. Chemistry in the Laboratory, 4th ed., 1997, W. H. Freeman: New York.

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Name: _____ **Student ID: _____**

Department: _____ **Group: _____** **Date: _____**

I. Experimental Data and Results

(A) Ascorbic Acid Content in Vitamin C Powder

Test	Mass of vitamin C powder (g)	Titration volume of 0.005 M KIO_3			Ascorbic acid content (g/g)
		V_i (mL)	V_f (mL)	$\Delta V = V_f - V_i$ (mL)	
1					
2					

- Average content of ascorbic acid in the vitamin C powder: _____ (g/g)
- Calculation: (*Show all steps of the titration calculation here, including mole-to-mole ratios, volume of titrant used, and final mass of ascorbic acid per gram of powder.*)

(B) Ascorbic Acid Content in Fresh Lemon Juice

- Reported ascorbic acid content (literature value):_____ mg/100 mL. (*Please cite a reliable data source, e.g., USDA or peer-reviewed literature.*)

- Observed ascorbic acid content (based on titration):

Test	Volume of lemon juice (mL)	Titration volume of 0.005 M KIO_3			Ascorbic acid content (mg/mL)
		V_i (mL)	V_f (mL)	$\Delta V = V_f - V_i$ (mL)	
1					
2					

- Average content of ascorbic acid in lemon juice:_____ (mg/100 mL)
- Calculation: (*Detail the titration-based calculation, showing conversion from volume of titrant to mass of ascorbic acid per 100 mL of juice.*)

II. Questions and Discussion

1. Based on its molecular structure, explain why vitamin C is water-soluble