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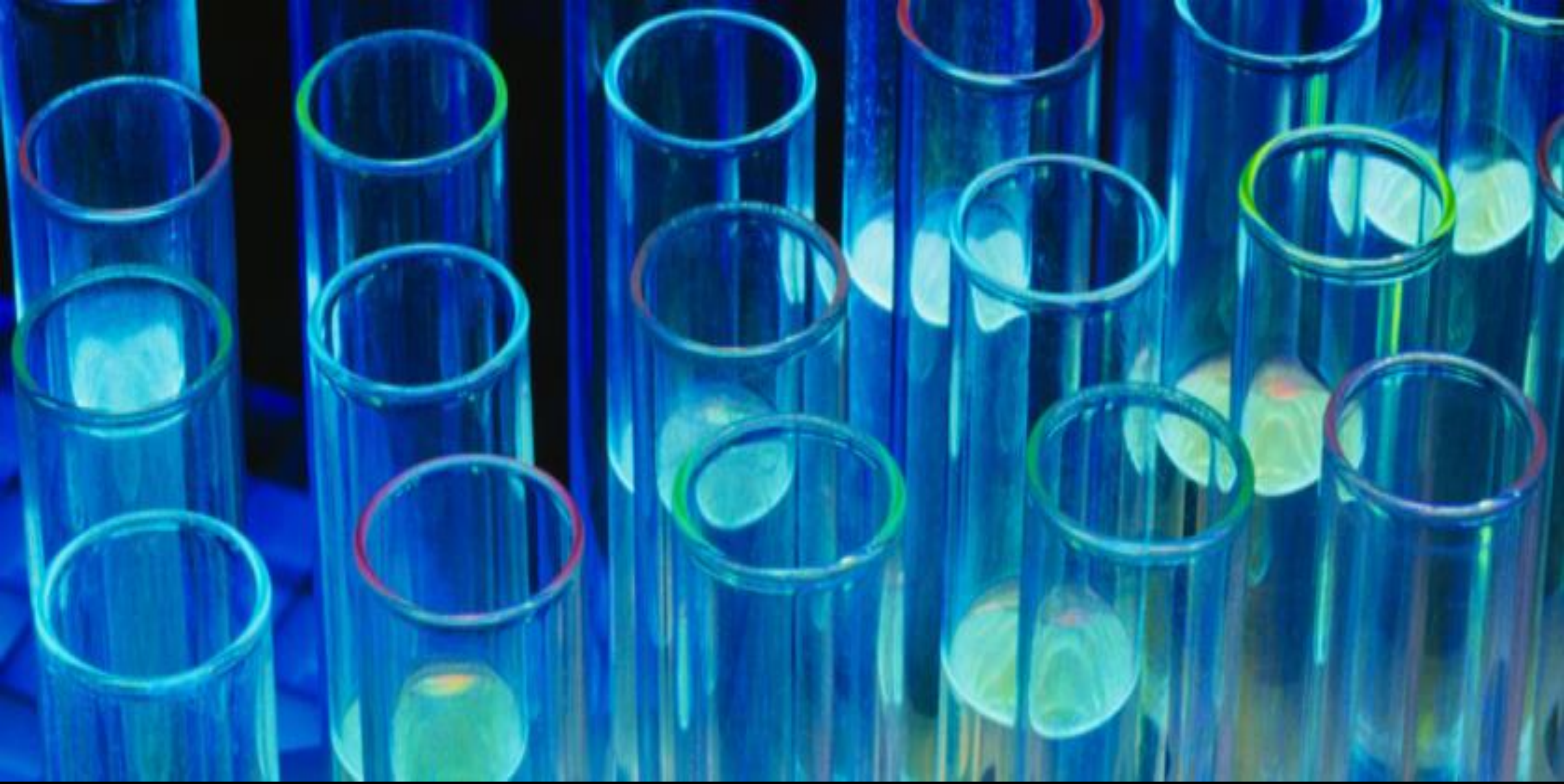
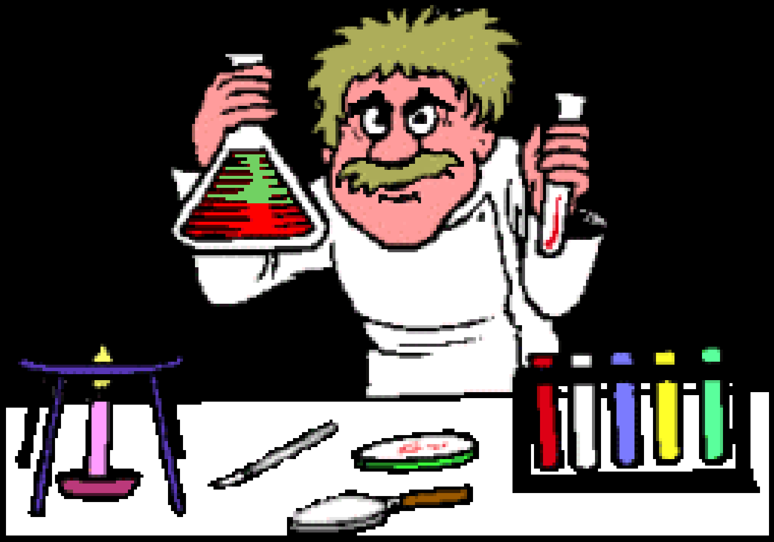


113-2
Expt. 4 - Quantitative Analysis of Vitamin C

114.05.23 (time required: 1.5 hours)

1. Objective

- **To determine the vitamin C content in high-dose vitamin C powder and lemon juice using redox titration methods.**



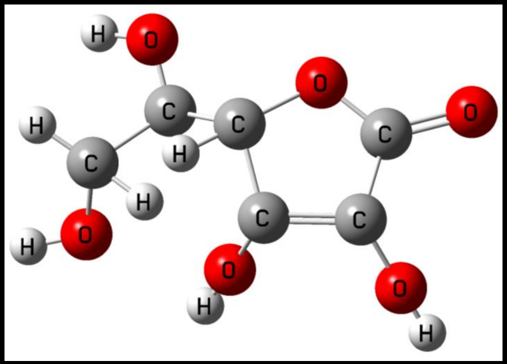
2. Principle

- **Commercial vitamin C supplements are available in a range of dosages, such as 100 mg, 200 mg, or 500 mg. But do these products actually contain the amount of vitamin C stated on the label? This experiment aims to quantify the vitamin C content using a redox titration technique, allowing for a precise comparison between the labeled and actual values.**

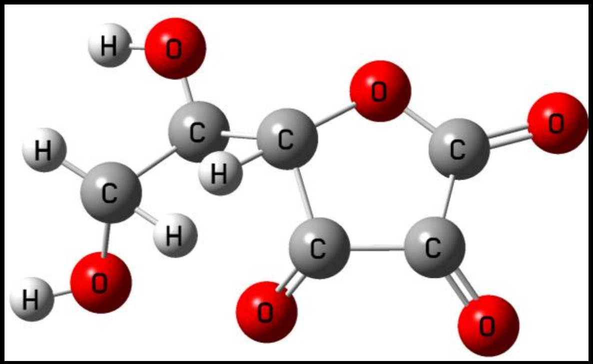
- Vitamin C, also known as ascorbic acid, is a strong reducing agent capable of reducing Fe(III) to Fe(II) or I_2 to I^-. In this experiment, a potassium iodate (KIO_3) solution is used as the titrant in an acidic medium, where it first reacts with sodium iodide (NaI) to generate molecular iodine (I_2):

$$IO_3^- + 5I^- + 6H^+ \longrightarrow 3I_2 + 3H_2O \qquad \text{(eq.4-1)}$$

- **Iodine molecules (I_2) readily undergo a redox reaction with ascorbic acid in the solution, as illustrated in Equation 4-2:**



$+ \; I_2 \longrightarrow$



$+ \; 2I^- + 2H^+$

(eq.4-2)

- Once all the ascorbic acid in the solution has reacted, any excess iodine (I_2) combines with iodide ions (I^-) to form triiodide (I_3^-). This species interacts with the pre-added starch indicator, producing a blue-black complex that signifies the titration endpoint. Using the known molar amount of iodate (IO_3^-) and its stoichiometric relationship with I_2 and ascorbic acid, the ascorbic acid content in the sample can then be accurately calculated.

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

3. Equipment and Chemicals

Equipment

In cabinet	Provided by TA
Graduated pipette x 1	Burette (50 mL) x 1
Safety pipette filler x 1	Burette clamp
Stir bar x 1	Fine filter screen
Volumetric flask (100 mL) x 1	
Erlenmeyer flask (125 mL) x 2	
Beaker (100 mL) x 2	
Hot plate stirrer x 1	

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*

* : Corrosive　* : Toxic　* : Irritative

* : Oxidizing　* : Flammable

參考資料：https://pubchem.ncbi.nlm.nih.gov/

4. Experimental Procedures

A. Determination of Ascorbic Acid Content in Vitamin C Powder

1. Accurately weigh 0.1 g of vitamin C powder using an analytical balance.

2. Transfer the powder to a 100 mL beaker, add 50 mL of deionized (DI) water, and stir thoroughly until the powder is completely dissolved. Then, transfer the solution to a 100 mL volumetric flask. Rinse the beaker thoroughly with small portions of DI water and add the rinses to the flask. Finally, dilute the solution to the calibration mark with DI water and mix well.

3. Pipette 25.0 mL of the prepared vitamin C solution into a 125 mL Erlenmeyer flask.
4. Add the following reagents to the flask:
   – 2.0 mL of 1 M sodium iodide (NaI)
   – 2.0 mL of 1 M hydrochloric acid (HCl)
   – 1.0 mL of 2% starch indicator solution
5. Rinse a burette thoroughly with deionized water, then rinse it three times with approximately 5 mL of 0.005 M potassium iodate (KIO_3) solution. Fill the burette with the same KIO_3 solution, remove any air bubbles, and record the initial volume (V_i) to the nearest 0.01 mL.

6. **Titrate the vitamin C solution with 0.005 M KIO_3 until a persistent dark green color appears, indicating the endpoint of the titration. Record the final volume (V_f) to the nearest 0.01 mL.**

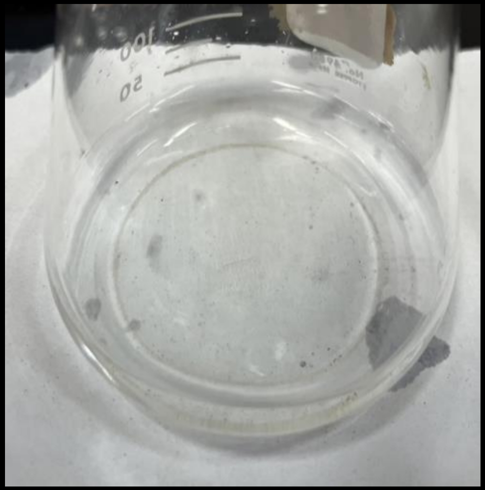


Figure 1. Solution appearance before titration (colorless)

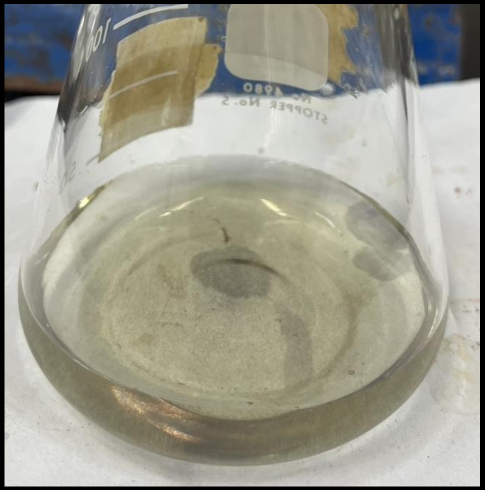


Figure 2. Solution appearance after titration (greenish)

7. **Repeat *Steps 1 through 6* to perform a duplicate titration for improved accuracy.**
8. **Using the titration volumes of 0.005 M KIO_3, calculate the average ascorbic acid content in the vitamin C powder sample.**

B. Determination of Ascorbic Acid Content in Lemon Juice

1. Prepare 50 mL of freshly squeezed lemon juice, ensuring that all pulp and fibers are removed through filtration.

2. Rinse a 25 mL pipette thoroughly with deionized water, then rinse it twice with approximately 5 mL of the lemon juice. Accurately pipette 20.0 mL of the filtered juice into a 125 mL Erlenmeyer flask.

3. Add the following reagents to the Erlenmeyer flask:
   - 2.0 mL of 1 M sodium iodide (NaI)
   - 2.0 mL of 1 M hydrochloric acid (HCl)
   - 1.0 mL of 2% starch indicator solution
4. Titrate the solution with 0.005 M potassium iodate (KIO_3) and record the volume used to the nearest 0.01 mL.

5. **Accurately measure another 20.0 mL of lemon juice and repeat the titration to obtain a duplicate measurement.**
6. **Using the titration volumes of 0.005 M $KIO_3(aq)$, calculate the ascorbic acid content in the lemon juice, expressed in mg per 100 mL.**

Figure 3. Lemon juice sample before titration

Figure 4. Lemon juice sample after titration (endpoint reached)

5. Precautions

1. Dispose of all waste liquids in the designated waste container for proper recycling.
2. Shake the 2% starch indicator thoroughly before use if any precipitation is observed.
3. Always wear a lab coat, goggles, and gloves during the lab session.
4. Handle all chemicals with care, as they may be toxic or hazardous. Follow all laboratory safety regulations and the instructions of the TAs at all times.
5. Removal of any chemicals or laboratory equipment from the lab is strictly prohibited.

6. Experimental Data

(A) Ascorbic Acid Content in Vitamin C Powder

1. Average content of ascorbic acid in vitamin C powder: ______ (g/g)

2. Determination of ascorbic acid content in vitamin C powder:

Test	Weight of Vitamin C Powder (g)	0.005 M KIO_3 Titration Volume			Ascorbic Acid Content (g)
		V_i (mL)	V_f (mL)	ΔV (mL) = $V_f - V_i$	
1					
2					

(B) Determination of Ascorbic Acid Content in Lemon Juice (approximately 50 mL per lemon)

1. Reported ascorbic acid content (*literature value*) **: _______mg / 100 mL**

2. Determination of ascorbic acid content:

Test	Volume of Lemon Juice (mL)	0.005 M KIO_3 Titration Volume			Ascorbic Acid Content (mg/100 mL)
		V_i (mL)	V_f (mL)	ΔV (mL) $= V_f - V_i$	
1					
2					

Average ascorbic acid content in lemon juice: _______ mg /100 mL

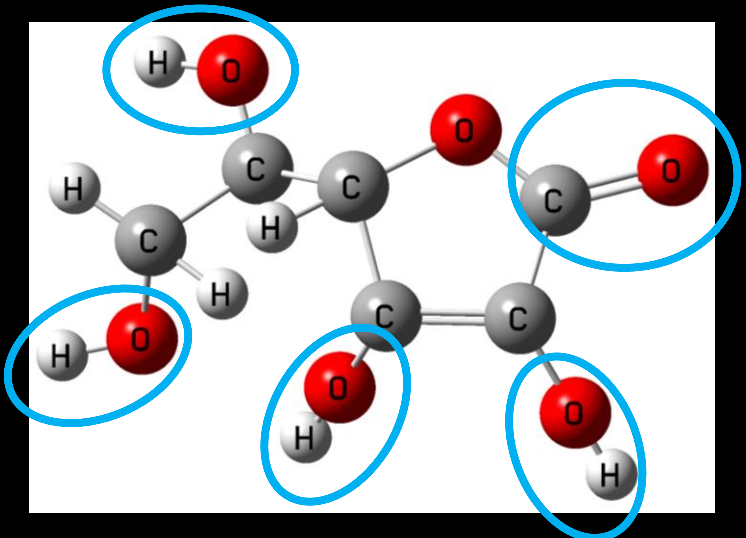
7. Experimental Error

	Experimental Value	Theoretical Value	% Error
Vitamin C Powder (g)			
Lemon Juice (mg / mL)			

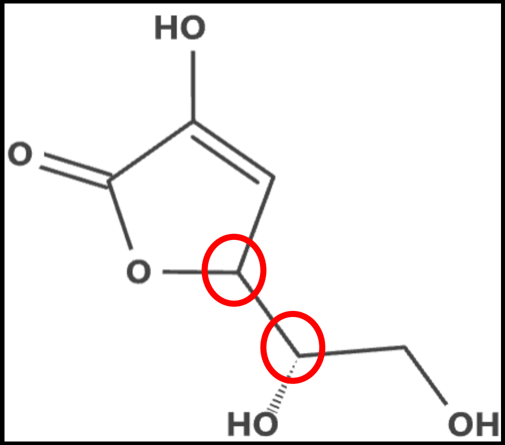
8. Questions

1. **Vitamin C is a water-soluble vitamin. Explain this property based on its chemical structure.**

Ans:
The more -OH groups and -COOH groups a molecule has, the more it can form hydrogen bonds with water and dissolve in it. Vitamin C has four -OH groups and one C=O group in its molecule, which gives it excellent water solubility.



2. When the four hydrogen atoms bonded to the carbon atom in methane (CH_4) are replaced with four different substituents, the resulting molecule and its mirror image become non-superimposable. Such a pair of molecules are called ***enantiomers***. A carbon atom bonded to four different groups is referred to as a chiral carbon, commonly indicated as **C***. Identify the chiral carbon(s) in the structure of ascorbic acid.



参考來源 : https://mulicia.pixnet.net/blog/post/27223478

3. **Why is the titration carried out in an acidic medium in this experiment?**

Ans:

Potassium iodate solution needs to react with sodium iodide in an acidic environment to produce iodine molecules, which then can undergo a redox reaction with ascorbic acid.

4. Research and report the typical vitamin C content in lemons. Compare this value with your experimental results, and cite a reliable reference source for the reported value.

Ans:
Lemon: 34 (mg/100g).
The experimental value is 27.7 mg/100g, with some vitamin C present in the lemon's fibers, pulp, and peel.

參考來源:https://www.dietician.com.tw/knowledge/item/1131.html

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