Quantitative Analysis of Vitamin C

Last revised: 2023/10/15

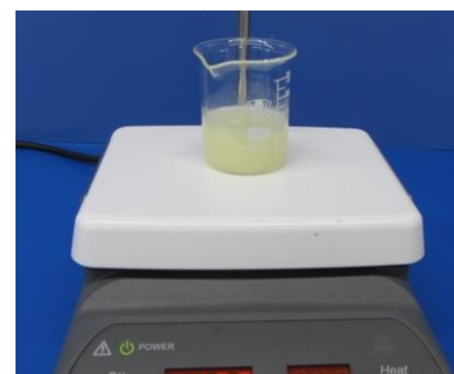
Prelab Preparation

◻ Dissolution of Vitamin C Tablet

1. Accurately weigh one vitamin C tablet
2. Wrap each tablet in weighing paper, then use a hammer to gently crush the tablet on concrete surface (NOT on the lab bench)
3. Transfer the crushed vitamin C tablet to a 125 mL Erlenmeyer flask for low dosage, or a 100 mL beaker for high dosage.
4. Add 50 mL DI water and a stir bar to solution, then stir to dissolve

◻ Wash one 100 mL beaker and place it in oven to dry (for taking 0.025 M KIO_3)

✓ Do NOT use the heating function of the hot plate



High dosage in a beaker

Preparation

Collect the following items

- One 25 mL transfer pipet and one pipet filler
- Two 125 mL Erlenmeyer flasks
- One funnel (for titrant)
- The TA will distribute one magnetic stir bar to each group

From your personal equipment

- One clean and dry 100 mL beaker (for taking KIO_3 solution)

Self-prepared

- Two vitamin C tablets (dosage ≤ 250 mg) or one high-dosage (> 250 mg)
- Commercial fruit juice or citrus fruits

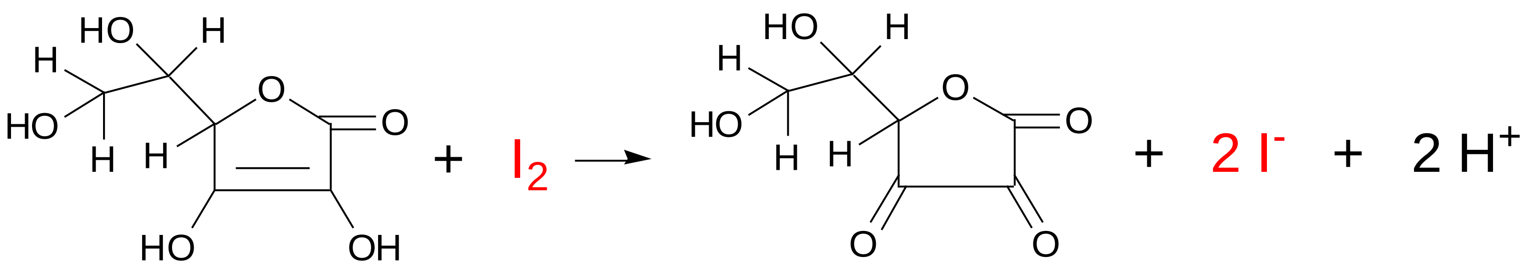
Objective and Principles

- **Objective:**

  Determine the actual content of vitamin C in commercially available tablets and juices by reduction-oxidation titration

- **Lab techniques:**
  - **Performing titration using a buret**
  - **Transfer pipet**
  - Hot plate/magnetic stirrer
  - Lab dispenser
  - Volumetric flask

Redox Titration



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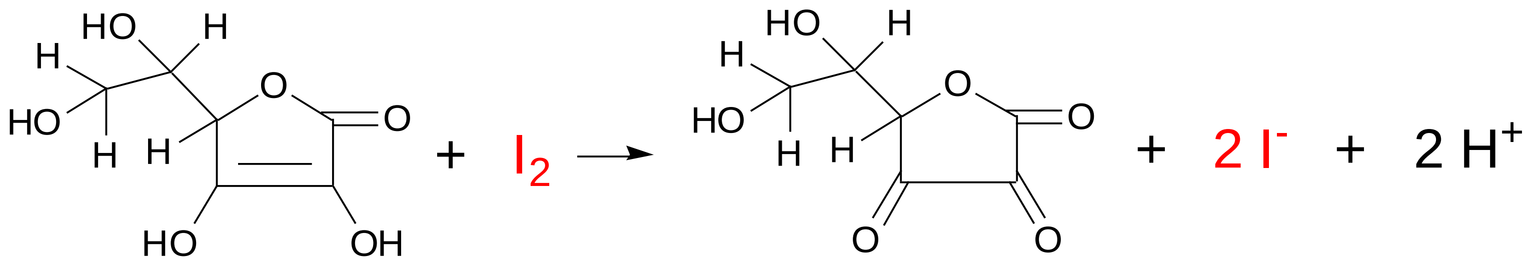
L-(+)-Ascorbic acid

- **Vitamin C** (L-(+)-Ascorbic acid, $C_6H_8O_6$) is a reducing agent, i.e. oxidized in the reaction
- $\mathbf{I_2}$ (the oxidizing agent) is generated by the reaction between $\mathbf{KIO_3}$ and **NaI** in an acidic medium:

$$IO_3^- + 5\ I^- + 6\ H^+ \rightarrow \mathbf{3\ I_2} + 3\ H_2O$$

- A known concentration of KIO_3 (titrant) is used to titrate vitamin C solutions (concentration to be determined)
- Once the equivalence point (# mole vitamin C = # mole I_2) is passed, the additional $\mathbf{I_2}$ would react with starch indicator to produce a **blue-black complex** that indicates the titration end point

Redox Titration



L-(+)-Ascorbic acid

$$IO_3^- + 5\,I^- + 6\,H^+ \rightarrow \mathbf{3}\,I_2 + 3\,H_2O$$

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

$$C_6H_8O_6\,(\text{mol}) = I_2\,(\text{mol}) = IO_3^-\,(\text{mol}) \times \mathbf{3}$$

Volume of KIO_3 solution used → # mole IO_3^- → # mole vitamin C

Experiment Tasks

Vitamin C Tablet

Low-dosage (≤ 250 mg)

2 tablets

OR

High-dosage (> 250 mg)

1 tablet (2 titrations)

- ✓ Avoid using foaming ingots or jelly drops (capsules are okay)

Juice

50 mL per portion

* 2 portions

OR

Squeezed Juice from one citrus fruit

*** 2 portions**

- ✓ Avoid carbonated drinks or juices of dark colors

Task 1-1: Low-Dosage Vitamin C Tablet

1. Accurately weigh two low-dosage vitamin C tablets (one of the tablets is dissolving)
2. Wrap each tablet in weighing paper, then use a hammer to gently crush the tablet on concrete surface (NOT on the lab bench)
3. Dissolve each portion of crushed tablet with 50 mL DI water in 125 mL Erlenmeyer flasks, then stir to dissolve

✓ Do NOT use the heating function of the hot plate

Task 1-1: Low-Dosage Vitamin C Tablet

4. For each vitamin C tablet sample solution, add 2 mL 1 M NaI, 2 mL 1 M HCl, and 1 mL 2% starch solution (volumes are preset on lab dispensers)
5. Titrate the solution with 0.025 M KIO_3 (read and record V_i and V_f to 0.01 mL) until the titration end point
6. Repeat the procedure on the other sample solution
7. Use the results to calculate the ascorbic acid contents in each vitamin C tablet

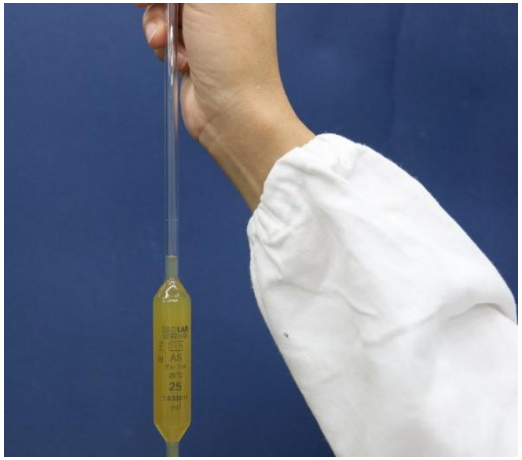
Task 1-2: High-Dosage Vitamin C Tablet

1. Accurately weigh **<u>one</u>** high-dosage vitamin C tablet
2. Wrap the tablet in weighing paper, then use a hammer to gently crush the tablet <u>on concrete surface</u> (NOT on the lab bench)
3. Dissolve the crushed tablet with 50 mL DI water in 100 mL beaker, then stir to dissolve
4. Transfer the solution to a 100 mL volumetric flask; rinse the beaker 2-3 times with DI water and combine the solution to the volumetric flask
5. Dilute the solution to 100 mL; transfer the diluted solution to another clean beaker

✓ Do NOT use the heating function of the hot plate

Task 1-2: High-Dosage Vitamin C Tablet

6. Use a 25 mL transfer pipet to take 25.0 mL of diluted sample solution to a 125 mL Erlenmeyer flask, add 2 mL 1 M NaI, 2 mL 1 M HCl, and 1 mL 2% starch (volumes are preset on lab dispensers)
7. Titrate the solution with 0.025 M KIO_3 (read and record V_i and V_f to 0.01 mL) until the titration end point
8. Take another portion of 25.0 mL diluted sample solution and repeat the titration procedure
9. Use the results to calculate the ascorbic acid contents in the original vitamin C tablet



Task 2: Vitamin C Content in Juice

1. Use a 25 mL transfer pipet to take **50.0 mL** of package juice (do the transfer twice) into a 125 mL Erlenmeyer flask
2. Add 2 mL 1 M NaI, 2 mL 1 M HCl, and 1 mL 2% starch (volumes are preset on lab dispensers)
3. Titrate the solution with 0.025 M KIO_3 (read and record V_i and V_f to 0.01 mL) until the titration end point
4. Take another portion of 50.0 mL juice sample and repeat the titration procedure
5. Use the results to calculate the ascorbic acid contents in the package juice

Result Calculation

$$C_6H_8O_6\ (mol) = I_2\ (mol) = IO_3^-\ (mol) \times 3$$

Task 1 – Vitamin C tablet (____ mg/tablet)

- Low-dosage: $(\underbrace{\Delta V\ mL\ \times\ 0.025\ M}_{mmol\ IO_3^-}) \times \frac{3\ C_6H_8O_6}{1\ IO_3^-} \times\ 176.12\ \frac{mg}{mmol}$
- High-dosage: $(\underline{\Delta V\ mL\ \times\ 0.025\ M}) \times \frac{3\ C_6H_8O_6}{1\ IO_3^-} \times\ 176.12\ \frac{mg}{mmol} \times \boxed{\frac{100.0\ mL}{25.0\ mL}}$

Task 2 – Juice (____ mg/100 mL)

- $(\Delta V\ mL\ \times\ 0.025\ M) \times \frac{3\ C_6H_8O_6}{1\ IO_3^-} \times\ 176.12\ \frac{mg}{mmol} \times \boxed{\frac{100.0\ mL}{50.0\ mL}}$

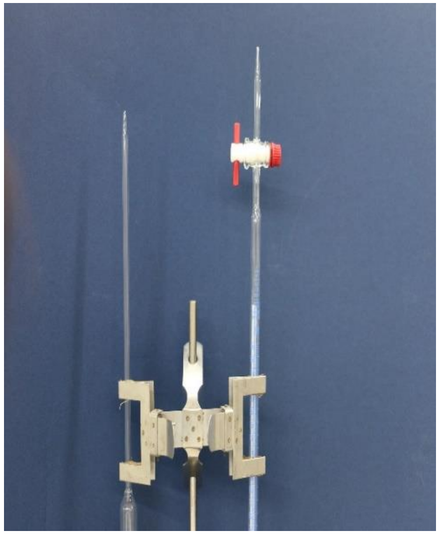
Additional Notes

- Use a **clean and dry** 100 mL beaker to take **~30 mL** 0.025 M standard KIO_3 solution (any remaining water in the beaker would affect the actual concentration of KIO_3)
- Refer to slides for detailed instruction for using a **transfer pipet, volumetric flask, buret**, and lab dispenser
- Wash the **transfer pipet**, **volumetric flask**, and **buret** thoroughly after use (do NOT put them into oven)
- **All the solution waste containing iodine should be collected and recycled**
- Do not eat or drink during the lab session

✓ Place the used pipet in an ultrasonic cleaner to prewash for 1 min., then rinse clean with tape water

Clean-Up and Check-Out

- Rinse the juice bottles with tape water and recycle to the designated bin
- The cleaned buret and transfer pipet should be clamped upside-down on the buret clamp
- Return the magnetic stir bar to TA
- Clean up the lab bench and check personal equipment inventory (<u>have an associate TA sign the check list</u>)

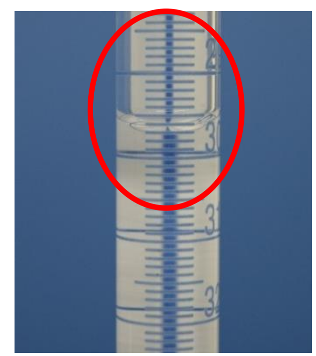


- This is a **Brief Report** experiment:
  - ✓ Complete calculation using correct significant figures
  - ✓ **Hand in prelab/lab note/report together to the TA**

- Groups on duty shall stay and help clean up the lab

Titration

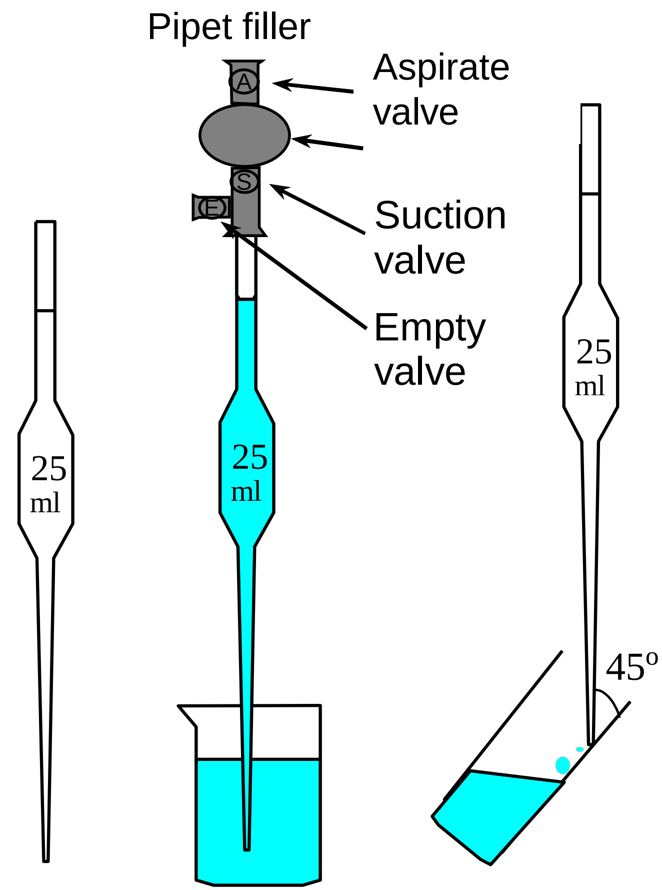
- Clean the buret with DI water, then rinse twice with ~5 mL of titrant (use a funnel to add titrant)
- Open the stopcock to repel the air at the buret tip
- Adjust the height of buret so that its tip is lower than the lid of receiving flask
- Read and record the initial volume (V_i) on the buret to 0.01 mL
- With the stopcock on the right side, use your left hand to control the stopcock while the right hand swirls the receiving flask in a circular motion
- At the titration end point, read and record the final volume (V_f) on the buret to 0.01 mL
- After the experiment, wash the buret and let it dry upside-down on the buret clamp



T14 Video (YouTube link)

Transfer (Volumetric) Pipet

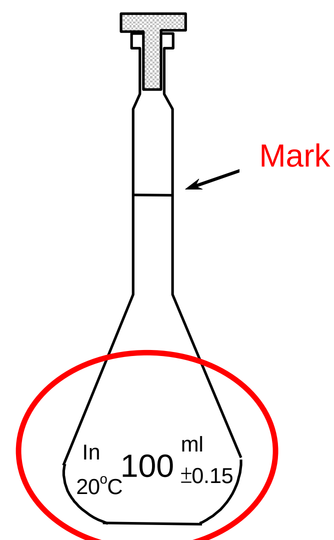
- Clean the pipet and rinse it twice with small amount of the liquid to be transferred
- Press valve **A** of the pipet filler and simultaneously squeeze the bulb to expel air from it, then insert the top of pipet gently into the pipet filler
- Bring the pipet tip below the liquid surface, press valve **S** to draw liquid until it rises above the inscribed line
- Remove the pipet filler and quicky use an index finger to close the top of pipet
- Use finger to adjust the liquid level to the inscribed line. Wipe off any excess liquid near the pipet tip
- Use the other hand to hold the new container. Maintain the pipet in a vertical position and let its tip touch the inner wall of the container. Release the index finger so that liquid is transferred
- Do not force out any liquid remaining at the tip
- Wash the pipet thoroughly after use



T12 Video (YouTube link)

Volumetric Flask

- Clean the volumetric flask thoroughly, then rinse it with a small amount of solvent
- Using a funnel, transfer the solution to be diluted into the volumetric flask
- Fill solvent into the flask until about half full, swirl the flask to let the solution mix
- Add more solvent so that the liquid level approaches (but does not exceed) the inscribed mark
- Use a dropper pipet to add solvent slowly, so that the liquid level matches the inscribed mark
- Install the stopper cap (hold with a finger), invert the flask several times to ensure thorough mixing
- Pour the solution into a beaker for later use (do not store solution in the flask)
- Wash the volumetric flask immediately after use and let it air dry (do not put flask on a hot plate or in an oven)



T13 Video (YouTube link)

Lab Dispenser

(1) Check the pre-set volume on the dispenser. Do not change the setting unless instructed to do so

(2) Place the receiving flask under the tip of dispenser

(3) To remove the air bubbles in the dispenser, lightly pull the piston pump up and down for several times

(4) Gently pull the piston pump up until it reaches the end of travel range, then slowly push the piston down to obtain the solution