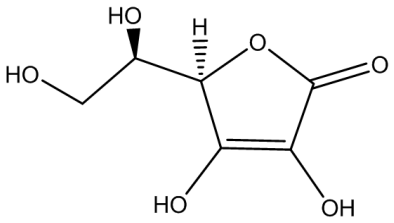
EXPERIMENT 5: Determination of an Analyte by Redox Back Titration

CAUTION: Please ensure you have proper gloves. Please ensure that you are not wearing open toe shoes. Do not wear open toe shoes with socks covering your feet. You must have proper shoes that fully cover your feet. You will not be permitted in laboratory without the proper safety attire. Failure to comply will result in mark of 0/10 and you will be removed from the laboratory.

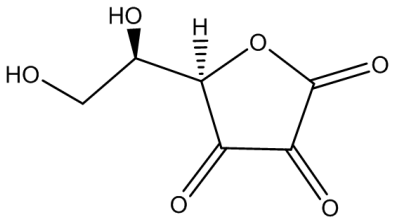
Ensure that when the fumehoods are used, you cover the bottles properly and leave the fume hood in a clean and neat state. Deduction in marks will result in not following proper protocols.

Aim:

To measure the vitamin content of a tablet by generating the oxidant in situ and back titrating with a reducing agent.



ascorbic acid



dehydroascorbic acid

Introduction:

The iodate (IO_3^-) ion has iodine centrally coordinated to three oxygen atoms with the entire molecule having a single negative charge. Reaction of this molecular ion with the simpler iodide (I^-) conveniently generates three equivalents of a powerful oxidizing agent, tri-iodide:

$$IO_3^- + 8I^- + 6H^+ \rightarrow 3I_3^- + 3H_2O$$

The high quantities of H^+ required to balance the equation is usually provided by a strong acid solution. In this experiment, ascorbic acid in the supplied tablets is quantified by reaction with a large excess of tri-iodide generated in the reaction vessel (in situ). The dehydroascorbic acid product does not undergo further oxidation. The remaining tri-iodide (I_3^-) is reacted with a reducing agent and the initial amounts of vitamin C determined from the difference between the initial and final quantities of tri-iodide (I_3^-). This process is generally referred to as *back-titration*.

For the thiosulfate reductant ($S_2O_3^{2-}$), we can write a balanced equation for its reaction with tri-iodide:

$$I_3^- + 2\,S_2O_3^{2-} \rightarrow 3I^- + S_4O_6^{2-}$$

where a one to two mole ratio is evident.

PROCEDURE

Part A: Preparation of Starch Indicator

This preparation can be done for every two benches (up to 12 workstations). Each workstation does not have to prepare the starch indicator. You can communicate with your colleagues to determine who will make the starch indicator.

Add approximately 0.5 g of starch to 250 mL of distilled water. This does not have to be done in a volumetric flask. It can be done in any vessel of your choosing. Weighing 0.5 g of starch indicator can be tricky, as you will see, so don't be too worried if you go over the 0.5 g.

Boil starch solution (0.5 g of starch made up to 250 mL of distilled water) solution for 20 minutes and cool to room temperature. *You can share hot plates but be cautious. Ensure you unplug the hotplate when you leave the laboratory.* When cooled, swirl/mix contents.

This is your starch indicator. Enjoy !

Part B: Preparation of 0.5 M sulfuric acid

Before you proceed, please ensure you are wearing proper gloves. Double up on gloves for this part of the procedure. Please ensure you are wearing goggles. Please ensure that you are not wearing open toe shoes. Do not wear open toe shoes with socks covering your feet. You must have shoes that fully cover your feet. You will not be permitted in laboratory without the proper safety attire. Failure to comply will result in mark of 0/10 and you will be removed from the laboratory.

You have to make up a volume of 300 mL of 0.5 M sulphuric acid. The concentration of 0.5 M does not have to be exact, it is an approximate concentration.

In the fume hood, you are provided with 98 % sulphuric acid (18.4 M). In order to obtain 300 mL of 0.5 M sulphuric acid, you can utilize the formula, (C = concentration; V = volume)

$$C_1 \times V_1 = C_2 \times V_2$$

Since you want approximately 0.5 M H_2SO_4 , fill a beaker or conical flask with 291 mL of distilled water. Then proceed to the fumehood. **CAUTIOUSLY** add 9 mL of concentrated (98 %) H_2SO_4 to the 291 mL of distilled water. Discard gloves and wash hands. Please ensure you follow proper safety procedures. Failure to comply will result in being thrown out of lab with a mark of 0/10. This 300 mL solution (9 mL H_2SO_4 + 291 mL distilled water), will be used as your 0.5 M sulphuric acid. Even if your volumes are not exact, it can be utilized as your 0.5 M sulphuric acid.

Part C: Preparation of Standard Potassium Iodate solution

The question is, based on a 250 mg, 500 mg or 1000 mg vitamin C tablet, and procedure below, what concentration should you use. Be prepared to determine the concentration you would like to use. You may have to come up with multiple answers based on the vitamin C strengths (250 mg, 500 mg, 1000 mg) and procedure.

Part D: Standardization of Sodium Thiosulphate

Collect approximately 200 mL of the standard potassium iodate (KIO_3) solution prepared (RMM=214). Rinse the 20/25 mL pipette provided with two 10 mL portions of the potassium iodate and transfer the precise volume to a clean 250 mL Erlenmeyer flask. Thoroughly dissolve approximately 4 g of $Na_2S_2O_3.5H_2O$ (RMM=248.2) in 250 mL of distilled water and use two 10 ml portions to rinse a rinse a burette. Fill the burette with the thiosulfate solution. Add roughly 2 g of KI and 20 mL of 0.5 M sulfuric acid to the iodate solution in the first flask and titrate this mixture immediately with the thiosulfate. When the colour fades to pale yellow, add 2 mL of the provided starch solution and continue the titration carefully until the blue colour disappears. Repeat the titration procedure to consistent values.

Part E: Quantification of Vitamin C in Tablet

Weigh vitamin C accurately tablet in uncrushed state.

Crush a vitamin C tablet. Divide the pulverized tablet into two samples of accurately determined known mass as transferred to separate Erlemneyer flasks. Add 60 mL of 0.5 M sulfuric acid to your first sample of the tablet. Swirl the flask, but do not be concerned if all of the powder does not dissolve. Add exactly 40/50 mL of the iodate solution to the flask (according to pipettes provided), 2g of potassium iodide and titrate immediately with thiosulfate and starch solution as performed in part A. When you have completed the titration of the first sample, repeat the titration with the second sample of pulverized tablet. If your titre volumes show too much variation (notwithstanding mass differences), repeat the determination with a second tablet.

Please ensure at this point that you thoroughly clean your workstation which will be inspected. Return any chemicals or reagents used from the centre bench to the centre bench. Failure to comply will result in deduction of marks.

Additional Exercises :

1. Calculate the percentage of vitamin C present in tablet.
2. Using this percentage or an alternative calculation, what is the mass of vitamin C present in a tablet?
3. Compare the experimental mass of vitamin C with the manufacturer's specifications.
4. Why is starch indicator prepared fresh?
5. What are the advantages of generating the oxidizing reagent in situ?
6. Why is the determination done immediately following the addition of potassium iodide?
7. Determine the isomer of ascorbic acid that is illustrated and write its IUPAC name.
8. Is tautomerism important in the function of ascorbic acid?
9. What evidence is there for the benefits of high vitamin C dosages as prophylaxis?