Experiment 31: Oxidation Reduction Titration of Vitamin-C

(This experiment is from Santa Monica College.)

Purpose

- To standardize a KIO_3 solution using a redox titration.
- To analyze a commercial product for vitamin-C content via titration.

Background

The two reactions below occur in the same solution. We will use these reactions in this experiment.

(1) $KIO_3(aq) + 6\ H^+(aq) + 5\ I^-(aq) \rightarrow 3\ I_2(aq) + 3\ H_2O(l) + K^+(aq)$ *generation of I_2*

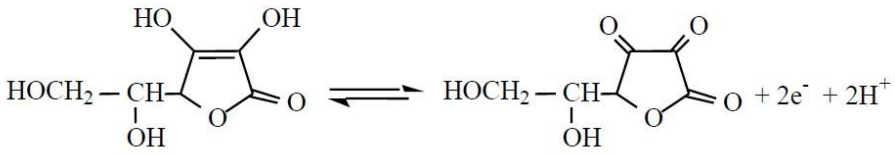
(2) $C_6H_8O_6(aq) + I_2(aq) \rightarrow C_6H_6O_6(aq) + 2\ I\text{-}(aq) + 2\ H^+(aq)$ *oxidation of vitamin-C*

Reaction one generates aqueous iodine, $I_2(aq)$. This is then used to oxidize vitamin-C (ascorbic acid, $C_6H_8O_6$) in reaction two. Both of these reactions require acidic conditions, so dilute hydrochloric acid, HCl *(aq)*, will be added to the reaction mixture. Reaction one also requires a source of dissolved iodide ions, $I^-(aq)$. This will be provided by adding solid potassium iodide, KI *(s)*, to the reaction mixture.

This is a redox titration. The two relevant half reactions for reaction (2) above are:

$I_2 + 2e^- \rightarrow 2\ I^-$

Reduction half reaction for Iodine at pH 5



Oxidation half reaction for vitamin-C ($C_6H_8O_6$) at pH 5

A few drops of starch solution will be added to help determine the titration endpoint. When the vitamin-C (ascorbic acid) is completely oxidized, the iodine, $I_2(aq)$, will begin to build up and will react with the iodide ions, I-*(aq)*, already present to form a highly colored blue I_3--starch complex, indicating the endpoint of the titration.

Chemicals

Approx. 0.01 M $KIO_{3(aq)}$	KI solid	1 M $HCl_{(aq)}$
1% $starch_{(aq)}$	Ascorbic acid (pure)	Vitamin C tablet

Equipment

1 Buret	1 Buret stand & clamp	125 Erlenmeyer flask
Weigh paper	spatula	Beakers (several)
Mortar and pestle (share)		

SAFETY: Avoid contact with iodine-based solutions, as they will stain your skin. Wear safety glasses and gloves at all times during lab class.

Proper Titration Techniques:

Using a Buret

Proper use of a buret is critical to performing accurate titrations. Your instructor will demonstrate the techniques described here.

1. **Rinsing:** Always rinse a buret (including the tip) before filling it with a new solution. You should rinse the buret first with deionized water, and then three times with approximately 5-mL aliquots of the solution you will be using in the buret. Be careful to avoid spilling the solution on hands or clothing.
2. **Filling:** Mount the buret on a buret stand. Fill the buret with the titrant to just above the 0.00 mL mark.
3. **Removing Air Bubbles:** There will be an air bubble trapped in the tip of a newly filled buret. To remove the air bubble, place a waste beaker under the tip of the buret and open the stopcock fully to allow solution to flow out of the buret. This will push the air bubble out of the buret. The volume of the titrant should now be within the calibration marks on the buret.
4. **Reading the Buret:** You should always read the volume in a buret at the bottom of the meniscus viewed at eye level (see Figure 1). A black or white card held up behind the buret helps with making this reading. Burets are accurate to ±0.02 mL and all readings should be recorded to two decimal places. Be sure to record both the starting and ending volumes when performing a titration. The difference is the volume delivered.

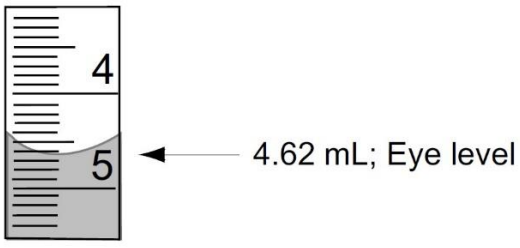


Figure 1. Reading a Buret

Part 1: Standardization of your KIO_3 solution

The KIO_3 solution has an approximate concentration of about ~0.01 M. You will need to determine the exact molarity to three significant figures. You will do this by titrating pure Vitamin C with your KIO_3 solution (KIO_3 is in the buret). Your final calculated results for each of three trials should differ by less than ± 0.001 M. Any trials outside this range should be repeated. You will need to calculate in advance how many grams of pure vitamin-C powder you will need to do this standardization (this is part of your prelaboratory exercise). Remember that your buret holds a maximum of 50.00 mL of KIO_3 solution and ideally you would like to use about 20 mL of KIO_3 solution for each titration (enough to get an accurate measurement).

1. Calculate the approximate mass of ascorbic acid you will need and have your instructor initial your calculations in your notebook. Remember, one mole of KIO_3 will yield 3 moles of I_2 (because of the KI added), and one mole of I_2 reacts with one mole of Vitamin C. Once you know the moles of Vitamin C, do a mole to gram conversion with the molar mass of Vitamin C (MW = 176.12 g/mole).
2. Weigh out approximately this amount of ascorbic acid onto tared weighing paper. Record the exact mass used in each trial to three decimal places in your notebook.
3. Transfer this mass into a 125-mL Erlenmeyer flask.
4. Add 20 mL of deionized water to the Erlenmeyer flask to dissolve the solid ascorbic acid.
5. Add 0.55 g of KI, 6 mL of 1 M HCl, and 4 drops of 1% starch solution to the Erlenmeyer flask. Swirl to thoroughly mix reagents.
6. Begin your titration. As the KIO_3 solution is added, you will see a yellow or blue color at the place were titrant hits the solution in the flask. While adding the KIO_3 swirl the flask to remove the color. As you get closer to the endpoint, the color will linger. Add the KIO_3 slowly as you approach the endpoint. Rinse the sides of the flask with DI water periodically during the titration. Stop adding the KIO_3 when the faint yellow or faint blue color does not disappear.
7. Calculate the molarity of this sample. Repeat the procedure until you have three trials where the molarities differ by less than ± 0.001 M.

Part 2: Analysis of the Vitamin-C Tablet (The Unknown)

1. Obtain one vitamin-C tablets.
2. Weigh the tablet and record its mass in your notebook.
3. Grind the tablet into a fine powder using a mortar and pestle.
4. Weigh out approximately 0.150 grams of the powdered tablet using tared weighing paper. Record the exact mass used in your notebook.
5. Transfer all of the sample from the weigh-paper into a 125 mL Erlenmeyer flask.
5. Dissolve the sample in 20 mL of deionized water and swirl well. Note that not all of the tablet will dissolve as commercial vitamin pills use insoluble binders to form the tablet.
6. Add 0.55 g of KI, 6 mL of 1 M HCl, and 4 drops of 1% starch solution to the flask before beginning your titration. Swirl to mix.
7. Begin your titration. As the KIO_3 solution is added, you will see a yellow or blue color at the place were titrant hits the solution in the flask. While adding the KIO_3 swirl the flask to remove the color. As you get closer to the endpoint, the color will linger. Add the KIO_3 slowly as you approach the endpoint. Rinse the sides of the flask with DI water periodically during the titration. Stop adding the KIO_3 when the faint yellow or faint blue color does not disappear.
8. Perform two more trials. If the first titration required less than 17 mL of KIO_3, increase the mass of unknown slightly in the remaining trials. Make sure you have three usable trials before ending your experiment. A usable trial is one in which you did not titrate past the endpoint.

Calculations

Part 1

Calculate the molarity of the KIO_3 for each trial.
Determine the average molarity of the KIO_3 solution ($(M_{T1} + M_{T2} + M_{T3}) / 3$)

Part 2

a) Use the average M_{KIO3} to calculate the moles of KIO_3 used in each trial.

b) Use the moles of KIO_3 used to calculate the moles of Vitamin C in each trial.

c) Calculate the grams of Vitamin C determined in each trial; convert to mg

d) Calculate the milligrams of Vitamin C per tablet.

$$\text{mg Vit. C in a tablet} = \frac{\text{mg Vit. C in powder used}}{\text{g of powder used}} \times \frac{\text{g tablet}}{\text{1 tablet}}$$

e) How does your mg/tablet Vitamin C compare to the amount listed on the bottle label? Calculate the % error to use in the conclusion.