Purpose

To determine the concentration of citric acid in drink mix using redox and acid-base titrations

Learning Objectives

To utilize acid-base and redox titrations to quantitate the amount of citric acid and Vitamin C (ascorbic acid) in a powdered drink mix packet.

To understand how stoichiometry allows for quantitation of an unknown quantity via titration.

To learn to perform accurate and precise titrations.

Equipment

- 50-mL buret
- 250-mL Erlenmeyer flasks
- 500-mL volumetric flask
- 10-mL and 25-mL volumetric pipets
- Graduated cylinders
- Magnetic stir bar
- Stir plate
- Funnel
- Beakers

Chemicals

- Powdered drink mix packets
- 0.1 M NaOH
- Thymol blue indicator solution
- 5.00×10^{-4} M KIO_3
- 0.5% starch solution
- KI
- 0.5 M HCl

Introduction

Powdered drink mixes such as Kool-aid® consist primarily of citric acid which gives the drink its characteristic tart flavor that balances the sweetness from the added sugar. In addition to citric acid, the mix contains food dyes, preservatives such as calcium phosphate, artificial and natural flavors, and Vitamin C (Figure TI.1). Vitamin C is also known as ascorbic acid. The ratio of citric acid to ascorbic acid is approximately 100:1. In this experiment, **titration** techniques will be used to quantitate the amounts of citric acid and ascorbic acid in a packet of powdered drink mix.

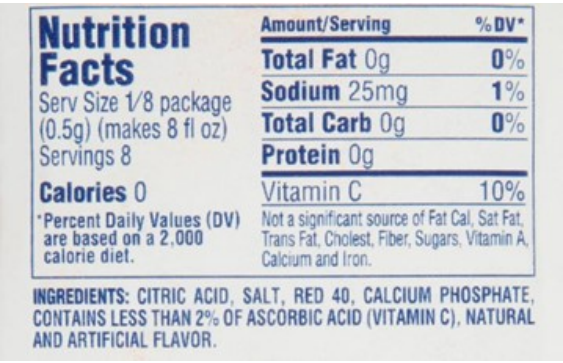

Figure TI.1: The nutritional information and list of ingredients from a strawberry-flavored Kool-aid brand powdered drink mix.

The structures of citric acid and ascorbic acid are shown in Figure TI.2. As might be predicted from their names both molecules act as acids and can react with a strong base. The acidic protons in each molecule are indicated by red boxes.

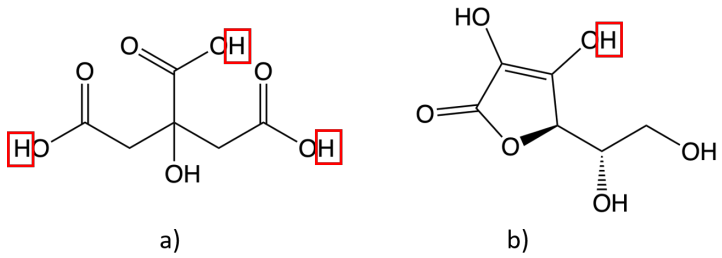

Figure TI.2: Structures of citric acid and ascorbic acid. Acidic protons are indicated with red boxes.

Titration is an analytical technique used to determine the concentration of an unknown substance in solution. In a titration, a reaction is done in which the molar concentration of one reagent solution is known, while the molarity of a second reagent solution is unknown. The known reagent solution is called the **titrant** while the unknown reagent solution is called the **analyte**.

A titration is complete when it has reached its **equivalence point**, which is when the mole-to-mole ratio of the reagents that were mixed together is equal to the mole-to-mole ratio of these reagents in the balanced chemical equation. If the molar concentration and the volume of titrant added are precisely known, the moles of titrant added can be found and the moles of analyte determined from the mole-to-mole ratio.

Many different types of reactions where the stoichiometry is known are suitable for analysis by titration. including acid-base reactions, redox reactions, and complexation reactions. In this experiment, both acid-base and redox titrations will be performed.

In order to perform a titration, a signal to indicate when the equivalence point is reached is required. This is often done by use of an indicator. An indicator is a substance that changes color as soon as there is a minute amount of excess titrant after all the analyte has been reacted. The color change is called the end point of the titration. Ideally, the end point of the titration occurs when 1 drop or less of titrant is added.

First, the total moles of acid in a sample will be determined by titration with sodium hydroxide (NaOH), a strong base. Both citric acid and ascorbic acid will react with the base according to the balanced equations, Reaction TI.1 and Reaction TI.2. Note that citric acid has three acidic protons so each citric acid molecule will react with three NaOH.

$$H_3C_6H_5O_7\,(aq) + 3\,NaOH\,(aq) \longrightarrow 3\,H_2O\,(l) + Na_3C_6H_5O_7\,(aq) \qquad \text{(Reaction TI.1)}$$

$$HC_6H_7O_6\,(aq) + NaOH\,(aq) \longrightarrow H_2O\,(l) + NaC_6H_7O_6\,(aq) \qquad \text{(Reaction TI.2)}$$

The total moles of NaOH that will be required to reach the endpoint of the reaction will be equal to 3 times the moles of citric acid plus the moles of ascorbic acid in the sample. The indicator used for this titration will be thymol blue which changes from yellow to blue at a pH between 8.0 and 9.6. This color change will signal the end point of the titration.

Next, the total moles of ascorbic acid in a sample will be determine by a redox titration. Ascorbic acid or Vitamin C can undergo a 2 electron oxidation to produce dehydro-L-ascorbic acid (Figure TI.3).

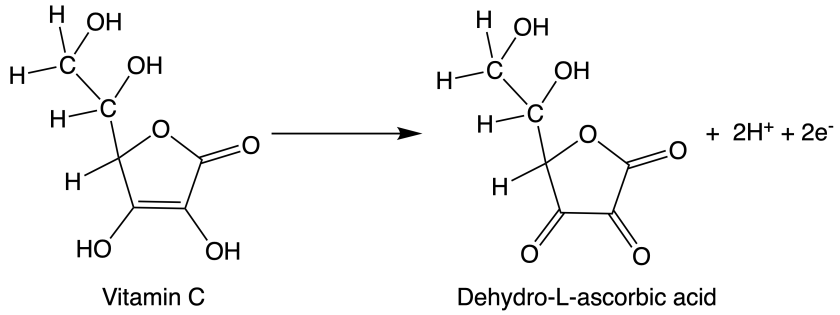

Figure TI.3: Oxidation of vitamin C to dehydro-L-ascorbic acid

This oxidation can be accomplished by reaction with aqueous iodine (I_2) according to the Reaction TI.3.

$$HC_6H_7O_6\,(aq) + I_2\,(aq) \longrightarrow C_6H_6O_6\,(aq) + 2\,H^+\,(aq) + 2\,I^-\,(aq) \qquad \text{(Reaction TI.3)}$$

Because iodine solutions are difficult to make precisely and are not shelf-stable, iodine will be generated in situ during this experiment. Excess solid potassium iodide (KI) and acid will be added to the sample and it will be titrated with a potassium iodate (KIO_3) solution. In the presence of acid, KI and KIO_3 react to produce I_2 (Reaction TI.4).

$$IO_3^-\,(aq) + 5\,I^-\,(aq) + 6\,H^+\,(aq) \longrightarrow 3\,I_2\,(aq) + 3\,H_2O\,(l) \qquad \text{(Reaction TI.4)}$$

The indicator for the redox titration will be a starch solution. Excess I_2 will react with I^- in solution to form I_3^- (triiodate ion, Reaction TI.5) which forms a complex with starch that is dark blue (Reaction TI.6).

$$I_2\,(aq) + I^-\,(aq) \longrightarrow I_3^-\,(aq) \qquad \text{(Reaction TI.5)}$$

$$I_3^-\,(aq) + \text{starch} \longrightarrow I_3^-\text{–starch complex (dark blue)} \qquad \text{(Reaction TI.6)}$$

Procedure

Safety Precautions

- Methyl blue can stain skin. Gloves should be worn.
- Sodium hydroxide is a strong base and hydrochloric acid is a strong acid. If skin or eye contact occurs, flush with running water for at least 15 minutes.
- Potassium iodide is a possible skin and lung irritant. Avoid contact with skin. Do not breathe in.
- All solutions are non-hazardous and can be washed down the drain with running water.
- Wear appropriate lab attire including safety goggles.
- Absolutely no substances found in a chemistry lab should be ingested.

Each lab group (2–3 students) will work independently. You may not share data with other lab groups.

Part I. Preparation

Preparation of Powdered Drink Mix Sample

1. **Data Table:** Create a data table on the notebook pages at the end of this procedure that includes the following: Flavor of Drink Mix and Mass of Powdered Drink Mix (g). Fill in this data table as you complete this experiment.

2. Weigh the sample (contents of package) of powdered drink mix. Record the weight.

3. Fill a 500-ml volumetric flask halfway with distilled water. Add the powdered drink mix. Swirl and/or invert flask (with stopper) for at least two minutes until the drink mix is fully dissolved. Check that no solid remains after the solution is resting for one minute. Dilute to the mark with distilled water and carefully invert to fully mix.

Preparation of a Buret

> You will perform **both** titrations. NaOH solution will be used if you are performing the acid-base titration. KIO_3 solution will be used if you are performing the redox titration.

1. Use soap or detergent and a buret brush (located at the front sinks) to wash your buret thoroughly.

2. Rinse your buret with tap water and then distilled water.

3. If droplets are left on the inner walls after draining the buret, repeat steps 1 and 2.

4. Add 2–3 mL of the NaOH or KIO_3 solution to rinse the buret. Tilt and rotate the buret so that all of the inner walls are rinsed. Drain the buret, discard this rinse solution, and repeat this step two more times.

5. Clamp the buret in an upright position in a buret clamp that is attached to a ring stand. Place an empty beaker underneath the tip of the buret as a waste beaker.

6. Using a funnel, fill the buret above the zero mark with the solution of NaOH or KIO_3 while the stopcock is closed. Flush out air bubbles by opening the stopcock fully. These bubbles are often difficult to see. Drain the buret into the waste beaker until the meniscus lies between the zero and 1-mL marks. You may allow the meniscus to pass the 1-mL mark if all the bubbles are not gone. Add more of the NaOH or KIO_3 solution if necessary.

> Always remove the funnel from the buret before using the buret to do a titration.

> **Lab Technique of the Day: Filling a Buret and Measuring the Volume of Solution in a Buret.** Each lab partner must show your teaching team member the technique used for adding additional solution to the buret and measuring the volume the buret contains. This does not need to be done when the buret is initially prepared, but at any point that the buret is being filled.

Part II. Determine the Moles of Total Acid (Citric and Ascorbic): Acid-Base Titration

1. **Data Table:** Create this data table on the notebook pages at the end of this procedure. Fill in this data table as you complete this experiment.

Table TI.1: Part II. Acid-Base Titration Data

	Trial titration	Exact trial 1	Exact trial 2	Exact trial 3
Initial reading of buret (mL)	_____	_____	_____	_____
Final reading of buret (mL)	_____	_____	_____	_____

2. Obtain approximately 150 mL of the 0.1 M standardized sodium hydroxide (NaOH) solution in a clean, dry, labeled beaker. Be sure to record the exact molarity.

3. First perform a trial titration to determine the approximate endpoint of the titration and observe the color change that indicates the endpoint of the titration.
   a. Read the position of the bottom of the meniscus on your buret filled with NaOH. Make sure that your eyes are on the same level as the meniscus or a serious error will occur. Record your reading. This is the initial buret reading.
   b. Measure a 10.00 mL aliquot of the powdered drink mix solution made in part I and quantitatively transfer it to your Erlenmeyer flask. Add approximately 40 ml of distilled water and 10 drops of thymol blue indicator to the flask. Add magnetic stir bar.
   c. Place the flask on a hot plate/stirrer under the buret with the tip of the buret inside the mouth of the flask. Turn on the stirrer and make sure the heat remains OFF.
   d. Add the NaOH solution in increments of about 1 mL while swirling or stirring with a stirring plate. When using a stirring plate, make sure the stirring rate is fast enough to mix well, but slow enough to prevent spattering. Observe the color of the solution after each addition.
   e. The trial titration is finished when the addition of about 1 mL causes the color of the solution to change and the color change persists for at least 30 seconds with stirring.

      > The endpoint color varies according to the initial color of the drink solution being tested. Save the first titrated sample in a beaker to use as a color reference for the next titrations.

   f. Record the final buret reading. Subtract the initial reading from this reading to find the volume required for the approximate end point.

4. Perform three exact titrations.

   a. Repeat steps a–c from step 2 with a new sample.
   b. Subtract ~5 mL from the volume that you calculated in step f of the procedure used for the trial titration. Rapidly add the resulting volume of NaOH to the flask from the buret.
   c. Rinse the inner walls of the flask with distilled water from a plastic bottle.
   d. Continue the titration on a drop-by-drop basis with stirring after each drop. The end point is the first permanent color change. If you are unsure about the end point, record the buret reading before you add another drop. Remember that finding the true end point requires patience and some skill.
   e. Record the final buret reading and calculate the volume of the solution of NaOH that was used in the titration.
   f. Repeat the procedure twice more with two additional samples. You may need to refill the buret with NaOH before repeating the titration.
   g. If the volumes at the end points of the three exact titrations differ by more than 0.15 mL (about 3 drops), repeat the titration with additional samples of until three volumes have this precision.

5. Clean up. Rinse all solutions down the drain with a lot of water.

Part III. Determine the Moles of Ascorbic Acid: Redox Titration

1. **Data Table:** Create this data table on the notebook pages at the end of this procedure. Fill in this data table as you complete this experiment.

Table TI.2: Part III. Redox Titration Data

	Trial titration	Exact trial 1	Exact trial 2	Exact trial 3
Initial reading of buret (mL)	_____	_____	_____	_____
Final reading of buret (mL)	_____	_____	_____	_____

2. Record the exact molarity of the approximately 5.00×10^{-4} M KIO_3 (potassium iodate) solution. *Record the exact concentration in molarity.*

3. First perform a trial titration to determine the approximate endpoint of the titration and observe the color change that indicates the endpoint of the titration.

   a. Read the position of the bottom of the meniscus on your buret filled with KIO_3. Make sure that your eyes are on the same level as the meniscus or a serious error will occur. Record your reading. This is the initial buret reading.
   b. Measure a 25.00 mL aliquot of the powdered drink mix solution made in part I and quantitatively transfer it to your Erlenmeyer flask. Add approximately 20 ml of distilled water, 0.5 g of solid KI (potassium iodide), 3 ml of 0.5 M HCl and 3 ml of 0.5% starch solution to the flask. Add magnetic stir bar.
   c. Place the flask on a hot plate/stirrer under the buret with the tip of the buret inside the mouth of the flask. Turn on the stirrer and make sure the heat remains OFF.
   d. Add the KIO_3 solution in increments of about 1 mL while swirling or stirring with a stirring plate. When using a stirring plate, make sure the stirring rate is fast enough to mix well, but slow enough to prevent spattering. Observe the color of the solution after each addition.
   e. The trial titration is finished when the addition of about 1 mL causes the color of the solution to change and the color change persists for at least 1 minute with stirring.

      > The endpoint color varies according to the initial color of the drink solution being tested. Save the first titrated sample in a beaker to use as a color reference for the next titrations.

   f. Record the final buret reading. Subtract the initial reading from this reading to find the volume required for the approximate end point.

4. Perform three exact titrations.

    a. Repeat steps a–c from step 2 with a new sample.
    b. Subtract ~5 mL from the volume that you calculated in step f of the procedure used for the trial titration. Rapidly add the resulting volume of KIO_3 to the flask from the buret.
    c. Rinse the inner walls of the flask with distilled water from a plastic bottle.
    d. Continue the titration on a drop-by-drop basis with stirring after each drop. The end point is the first permanent color change. If you are unsure about the end point, record the buret reading before you add another drop. Remember that finding the true end point requires patience and some skill.
    e. Record the final buret reading and calculate the volume of the solution of KIO_3 that was used in the titration.
    f. Repeat the procedure twice more with two additional samples. You may need to refill the buret with KIO_3 before repeating the titration.
    g. If the volumes at the end points of the three exact titrations differ by more than 0.15 mL (about 3 drops), repeat the titration with additional samples of until three volumes have this precision.

5. Clean up. Rinse all solutions down the drain with a lot of water.

Cleanup

- All solutions can go down the drain with excess water.
- Clean and return all glassware to the appropriate location.
- Wipe down your workspace.
- Enter your data into the Data Entry portion of Labflow.
- Check out with your AI.