Juice Titration

Background

Acids in Juice

Juice contains both citric and ascorbic acids. Citric acid is used as a natural preservative and provides a sour taste. Ascorbic acid is a water-soluble vitamin (vitamin C) that must be consumed regularly to ensure proper body function. Lack of vitamin C may result in scurvy, a disease with symptoms that include diarrhea, bleeding gums, and hemorrhage. Sailors on long sea voyages used to be very susceptible to this disease (have you ever seen old films in which a captain refers to his crew as a "scurvy lot"?). Scurvy was eliminated from British ships with the introduction of "limes" (which we call lemons today) into the sailors' daily rations. This led to the nickname "limey". Citrus fruits, tomatoes, and other fresh vegetables are also good sources of vitamin C. The minimum daily requirement (MDR) of vitamin C is 60 mg/day.
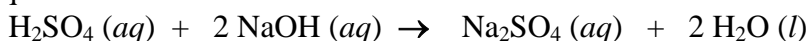
What is a titration?

A titration is a procedure for determining the concentration of a solution by allowing a carefully measured volume of the substance being analyzed (the analyte) to react with another solution (the titrant), whose concentration is known. The stoichiometry of the chemical reaction between the analyte and titrant is known.

titrant
(in buret)

The point in the titration where enough of the titrant has been added to react exactly with the analyte is called the equivalence point. The equivalence point is often marked by an indicator, a substance that changes color at (or very near) the equivalence point.[1]
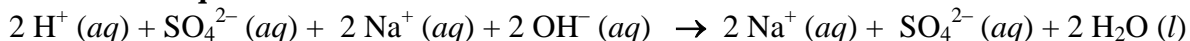
analyte

There are many types of titrations. The most common are acid/base and oxidation reduction titrations. You will be using both of these types of titrations in this experiment.
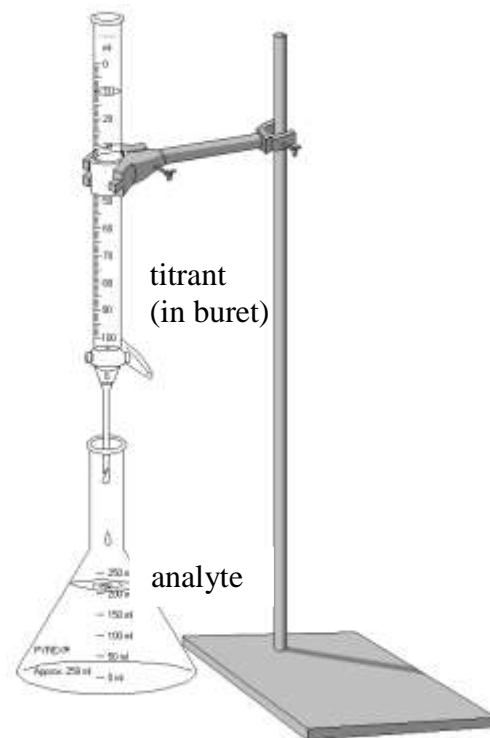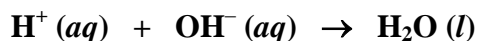
Acid/Base Titration

Here's a neutralization between sulfuric acid and sodium hydroxide. The equation for this reaction is:

$$H_2SO_4 \ (aq) \ + \ 2\,NaOH \ (aq) \ \rightarrow \ Na_2SO_4 \ (aq) \ + \ 2\,H_2O \ (l)$$

The **total ionic equation** for this reaction is:

$$2\,H^+ \ (aq) + SO_4^{2-} \ (aq) + \ 2\,Na^+ \ (aq) + 2\,OH^- \ (aq) \ \rightarrow 2\,Na^+ \ (aq) + \ SO_4^{2-} \ (aq) + 2\,H_2O \ (l)$$

The **net ionic equation** for this reaction is:

$$H^+ \ (aq) \ + \ OH^- \ (aq) \ \rightarrow \ H_2O \ (l)$$

[1] *The point at which the indicator changes color is called the "**endpoint**". We will normally assume that the endpoint is equal to the equivalence point.*

Instead of sulfuric acid, this lab involves two different acids: citric acid and ascorbic acid (both are acids, thus each reacts with NaOH). You can **determine the TOTAL amount of acid (total moles of H$^+$ = moles of H$^+$ from citric acid + moles of H$^+$ from ascorbic acid)** present in a juice sample by titration with NaOH, a strong base.

Equation 1

citric acid
C$_6$H$_8$O$_7$
MW=192.12 g/mol

sodium hydroxide

sodium citrate
(a salt)

water
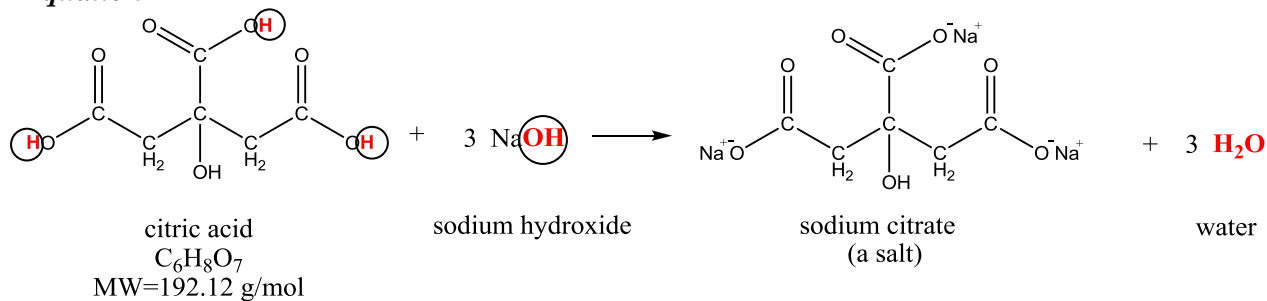
*Note: There are three acidic protons (the circled H atoms in the structure) per molecule of citric acid. And, one mole of hydroxide ion is needed to neutralize one mole of protons. Thus, **three** moles of hydroxide are required to titrate each mole of citric acid.

Equation 2

ascorbic acid
(vitamin C)
MW= 176.12 g/mol

sodium hydroxide

sodium ascorbate
(a salt)

water
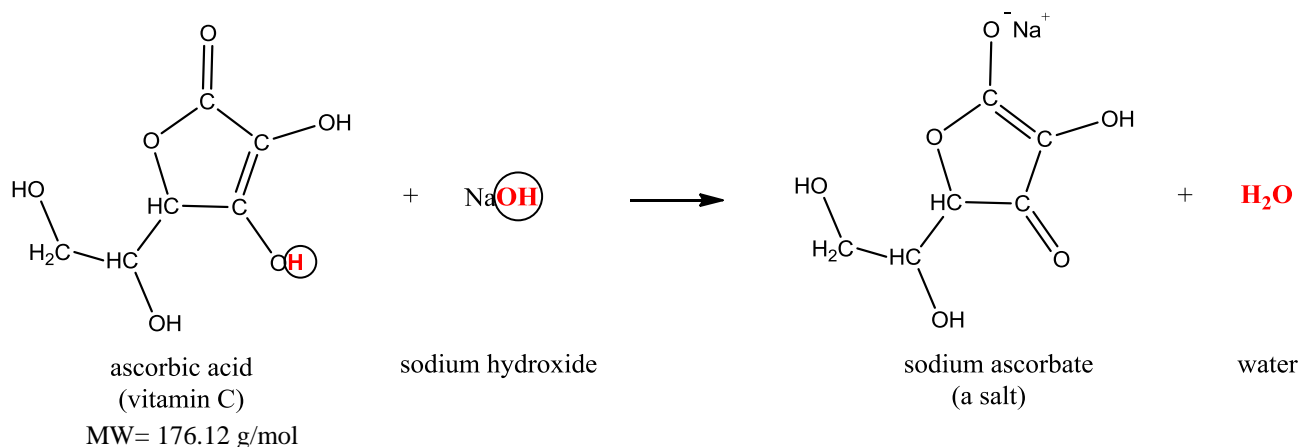
*Note: There is one acidic proton (the circled H atom in the structure) per molecule of ascorbic acid. And, one mole of hydroxide ion is needed to neutralize one mole of protons. Thus, **one** mole of hydroxide is required to titrate each mole of ascorbic acid.

Overall for the titration of the total acid in juice:
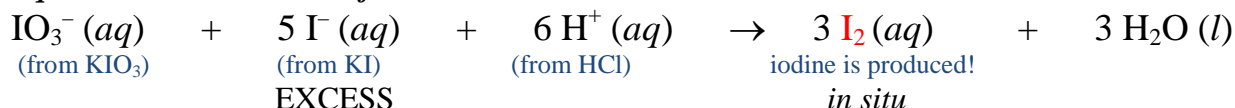
3 H$^+$ (citric acid) + 1 H$^+$ (ascorbic acid) + 4 OH$^-$ $\longrightarrow$ 4 H$_2$O + sodium salts of both acids
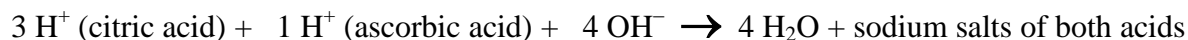
An acid-base titration of juice with sodium hydroxide will allow us to calculate the total moles of H$^+$ ions.

Oxidation/Reduction (Redox) Titration

The acid-base reactions above show that sodium hydroxide reacts with both acids. That means only the total amount of acid in solution can be determined using an acid-base titration. To determine the amount of each of the acids separately, we need something that will only react with one of the acids. Iodine (I_2) reacts with ascorbic acid **only** and not citric acid. The reaction between I_2 and ascorbic acid is a redox reaction.

Instead of loading a buret directly with I_2, we will need to generate I_2 *in situ*. I_2 can be formed from potassium iodate (KIO_3) under acidic conditions, according to the following chemical equation:

Equation 3—Generation of iodine

$$IO_3^-\ (aq) \quad + \quad 5\ I^-\ (aq) \quad + \quad 6\ H^+\ (aq) \quad \rightarrow \quad 3\ I_2\ (aq) \quad + \quad 3\ H_2O\ (l)$$

(from KIO_3)   (from KI)   (from HCl)   iodine is produced!

           EXCESS   *in situ*

*Note: It requires **one** mole of iodate ion (IO_3^-) to produce **three** moles of I_2.

Once I_2 is produced in the flask, the I_2 oxidizes ascorbic acid to dehydroascorbic acid according to the following chemical equation:
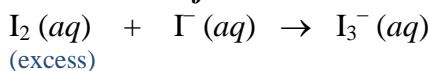
Equation 4—Oxidation of ascorbic acid

ascorbic acid (vitamin C)
$C_6H_8O_6$
MW=176.12 g/mol

iodine

dehydroascorbic acid

iodide ions   hydrogen ions
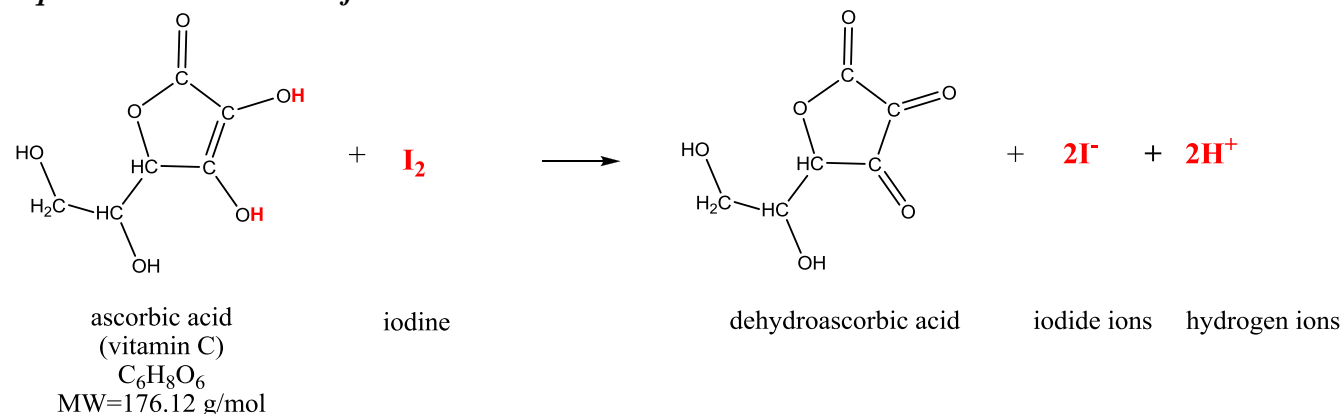
*Note: It requires **one** mole of iodine to oxidize **one** mole of ascorbic acid.

Once all of the ascorbic acid is consumed by I_2, we will see a blue color. How do we know when the reaction is done (and has reached the equivalence point)? Once the ascorbic acid runs out, excess iodine forms a starch- I_3^- complex signaling that the neutralization is complete, as shown in the chemical equations below:

Excess iodine forms triiodide

$$I_2\ (aq) \quad + \quad I^-\ (aq) \quad \rightarrow \quad I_3^-\ (aq)$$

(excess)

Endpoint is reached

$I_3^-\ (aq)$ + starch → starch-I_3^- complex (blue)

Because the redox titration involves a reaction of the ascorbic acid only, the amount of ascorbic acid can be determined from juice. Taking these results and the total amount of acid determined in the acid-base titration, the amount of citric acid can also be determined.

$$\text{moles of } H^+ \text{ (citric acid)} = \text{total moles of } H^+ - \text{moles of } H^+ \text{ (ascorbic acid)}$$

Laboratory Technique for Burets

Burets are used to deliver a recorded amount of liquid or solution to another container. A buret is marked in milliliters like a graduated cylinder, but buret markings show **0 mL** at the top, and the numbers increase as you go down the buret. The stopcock controls the liquid flow. It is *open* when *parallel* to the length of the buret and *closed* when *perpendicular* to the length of the buret.

- **Washing and rinsing the buret:** To clean a buret, wash its interior with soap and tap water using a beaker (never place it directly under the faucet*). Next, rinse the buret with 5-10 mL portions of DI water. With the buret over the sink and the stopcock open, pour the water into the buret and let it drain out the tip. *Most breakage occurs during washing, and burets do *NOT* fit under the faucet.
- **Conditioning the buret:** After the buret is well-drained, close the stopcock and add about 5 mL of the *titrant* (the solution to be used into the buret). Tilt the buret sideways and roll the barrel to completely rinse the inner walls of the buret. Drain the solution through the buret tip to insure the tip is also conditioned. Repeat this step at least twice to be sure all interior surfaces are rinsed with titrant.
- **Filling the buret:** Close the stopcock. Use a clean funnel to fill the buret with titrant just above the "0" mark. Place a container under the buret tip, and open the stopcock briefly to fill the buret tip with solution, leaving no air bubbles, and to get the level of meniscus to fall within the markings of the buret. If the tip does not fill with solution when the stopcock is in the open position, there may be an air bubble in the stopcock. Consult your instructor.
  Note: ***The initial level of titrant need not be exactly at 0.00 mL*** as the initial level of liquid will be recorded and subtracted from the final volume to determine the volume delivered.
- **Reading the buret:** Always remove the funnel used to fill the buret before taking any measurements. Record the volume of titrant by noting the bottom of the meniscus. On the buret shown below, numbers marked for every 1 mL, and the ten lines between each number represent every 0.1 mL. Thus, the level of titrant in the buret can be estimated to one more decimal place than the markings or to the nearest 0.01 mL.

  Thus, in the figure to the right, the meniscus is about halfway between 25.0 and 25.1 mL, so the level of titrant can be recorded as ***25.04 mL, 25.05 mL, or 25.06 mL*** depending on whether the bottom of the meniscus appears to be just above, just at, or just below halfway, respectively.
- **Cleaning the buret:** Afterwards, empty the buret, disposing of the titrant according to the waste disposal instructions for each experiments. Wash the buret with soap and tap water, then rinse with several portions of tap water, allowing some tap water to run through the tip. Do a final rinse with small portions of DI water, allowing the DI water to run through the tip, then return the buret to the stockroom.

Safety Precautions

HCl is corrosive, NaOH is caustic, and KI is a possible skin and lung irritant. Handle both with care. In case of contact with skin, rinse the area with large amounts of water and notify your instructor. Wear goggles at all times in the chemistry laboratory. You may wear gloves for extra protection.

Chemical Waste
Everything should go into the appropriate waste container in the fume hood.

Procedure

Buret Preparation
1. Obtain about 50 mL of 0.0500 M NaOH.   (Make sure your goggles are on!)

2. Prepare a buret for titration by rinsing it with two small portions of distilled water, followed by two 5-mL portions of the sodium hydroxide solution. Fill the buret with the sodium hydroxide solution and follow the usual procedures for eliminating air bubbles and setting the initial level.
(NOTE: You should not need more than 50 mL of NaOH for the buret preparation and all three trials of the acid-base titration. If you took too much NaOH, do not return it to the original container. See if anyone else needs it; if not, put it in the chemical waste container.)

Before you begin, make sure you understand how to read the buret properly. It is read from top to bottom, rather than from bottom to top. With a buret, you always read and record two values: the starting volume and the ending volume. The actual volume delivered is determined by calculating the difference.

Acid-Base Titration— How much total acid is in the juice?

3. Use a volumetric or graduated pipet to measure out 10.00 mL of juice.[2] Transfer the juice to a clean Erlenmeyer flask (the 250-mL size should be large enough). Add about 25 mL of distilled water. (*Does adding water change the number of moles of acid in your sample?*)

4. Add 2-4 drops of phenolphthalein indicator to the flask.

5. Record your starting buret reading. (*Have your lab partner verify your measurement—use the proper number of significant figures! A buret has an uncertainty of ± 0.01 mL.*)

6. Using the buret, add NaOH dropwise to the juice sample with constant swirling of the solution (analyte flask). The endpoint may be easier to see if you have a white sheet of paper under the flask. Stop when a last drop (or partial drop) of hydroxide solution results in a faint pink color that persists for at least 30 seconds.

7. Record your ending buret reading. Calculate the volume of NaOH delivered (added to flask).

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[2] Pineapple, white grapefruit, or apple juice can be used.   Good results can also be obtained by using "Invisible" Kool-aid.

8. Pour the contents of the flask into the chemical waste container. Rinse the flask with tap water, then a couple of times with distilled water. Use the volume of NaOH delivered for the first trial to estimate if you have enough NaOH in your buret for two more trials; add more NaOH to the buret if necessary. Repeat the titration twice more with two new samples of juice by repeating steps 3 through 7. The volumes delivered for each of the trials in a titration should be within **± 0.20 mL**.

Think about it: What was the point of the acid-base titration you just did?

Redox Titration—How much ascorbic acid (Vitamin C) is in the juice?[3]

1. Obtain about 100 mL of 0.00100 M KIO_3 solution. Prepare and fill a buret with this solution.

2. Use a graduated pipet or buret to measure out 5.00 mL of juice. (Record the precise volume.)

3. Transfer the juice to a clean 125 mL Erlenmeyer flask. Add the following reagents to the flask:
(Approximate amounts are given; the actual amounts used do not need to be recorded.)

-   50 mL distilled water,
-   one full Pasteur pipet (disposable glass pipet) of 1.0 M hydrochloric acid,
-   a spatula-tip full of KI,
-   and 10 drops of 3% starch   (The starch solution must be fresh.)

Swirl to mix the contents.

4. Record your starting buret reading (use significant figures!) Titrate the juice until a permanent (lasts at least 30 seconds), **faint** blue color is noticed. Record your ending buret readings so that you can calculate the volume of KIO_3 added.
NOTE: If the endpoint seems strange, tell your instructor. Maybe the starch solution is not fresh.

5. Repeat the titration twice more with two new samples of juice by repeating steps 2 through 4. The volumes delivered for each of the trials in a titration should be within **± 0.20 mL**. (Prior to each trial, check to see if you have enough KIO_3 in your buret. Make sure to rinse the flask between trials!)

Think about it: What was the point of the redox titration you just did?

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[3]Additional ascorbic acid was added to the juice by our lab staff. Originally it was not present in quantities that were significant compared to the citric acid.

Report Sheets

Juice Titration

Name_____ Date_____

Lab partner_____ Section_____

Data

Note: The volumes delivered for each of the trials in a titration should be within **± 0.20 mL**.

Table 1: Acid-Base Titration

	Trial 1	Trial 2	Trial 3	Average
Volume of juice (mL)	10.00 mL	10.00 mL	10.00 mL	
Initial volume (mL) NaOH				
Final volume (mL) NaOH				
Volume of **0.0500 M** NaOH delivered (mL)				

Table 2: Redox Titration

	Trial 1	Trial 2	Trial 3	Average
Volume of juice (mL)	5.00 mL	5.00 mL	5.00 mL	
Initial volume (mL) KIO_3				
Final volume (mL) KIO_3				
Volume of **0.00100 M** KIO_3 delivered (mL)				

Report Sheets
Calculations

Using the data you collected, perform the following calculations. Show your work and use the proper number of significant figures in your final answer.

<u>REDOX TITRATION</u>

1. Calculate the number of moles of KIO_3 used to titrate 5.00 mL of juice using the average volume of KIO_3 used in your redox titrations.

2. Based on the moles of KIO_3 calculated above, calculate the moles of I_2 generated in your redox titration. (See equation 3.)

3. Determine the number of **moles of ascorbic acid** present in 5.00 mL of juice based on the moles of I_2 used. (See equation 4.)

4. Based on the moles of ascorbic acid in 5.00 mL of juice, how many moles of ascorbic acid is present in 10.00 mL of juice? (Yes, this question is easy!)

(Think about it: Why do we want to know this info for a 10.0 mL aliquot?)

5. Using your answer above, calculate the number of moles of H^+ from ascorbic acid in a 10.0 mL aliquot of juice.

HINT: Use equation 2.

Moles of H^+ due to ascorbic acid alone:

Report Sheets

ACID-BASE TITRATION

6. Based on the average volume of NaOH used, determine the total number of moles of NaOH used in the acid-base titration of 10.0 mL of juice

7. (a) What is the net ionic equation for an acid base reaction?

(b) Based on your answer above, how many total moles of H^+ (from citric and ascorbic acids combined) were neutralized?

Total moles of H^+ due to both acids combined:

8. Use your answers to #5 and #7 to determine how many moles of H^+ were neutralized from citric acid alone.

Moles of H^+ due to citric acid alone:

9. The number of moles of H^+ ions is not necessarily the same as the number of acid molecules (from which they dissociate). Acids which can dissociate more than one H^+ ion are called polyprotic.

Use your answer above and equation 1 to determine the moles of citric acid (a polyprotic acid) in each 10.0 mL aliquot.

HINT: How many H^+ ions dissociate from one mole of citric acid?

Moles of citric acid:

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Pre-Lab Assignment:
Juice titration

Name_____

Section_____

Read the background section of this lab experiment to answer the following questions. Refer to the chapter on molarity and titrations (aqueous solutions) in your textbook.

1. Which type of titration is used to quantify the amount of ascorbic acid in juice?
(a) acid-base titration only   or   (b) redox titration only   or   (c) both titrations

2. Which type of titration allows you to quantify the amount of citric acid in juice?
(a) acid-base titration only   or   (b) redox titration only   or   (c) both titrations

3. If a buret is graduated every 0.1 mL, to what decimal place should you report for a measurement?

_____ mL

4. A student prepares and fills a buret with NaOH.
What is wrong with the following starting point for the titration?
(see picture at right)

5. What would you see (or not see) during a titration if you did not add phenolphthalein (indicator) to the flask?

6. A student measured out a 15.0 mL aliquot (portion) of juice into an Erlenmeyer flask. She added 3 drops of phenolphthalein indicator to the solution. It took 27.30 mL of 0.1003 M NaOH to reach the endpoint of the titration.

(a) Determine the moles of NaOH required for the titration. Use sig figs!

(b) What is the net ionic equation for an acid-base neutralization reaction?

(c) Determine the total moles of H^+ in the juice aliquot. Use sig figs!