Experiment 7
Titration of Juice

Background
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.

Principles

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 reaction between the analyte and titrant is known. 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]

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 titration in this lab.

[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.

Acid/Base Titration

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

You can determine the TOTAL amount of acid, both citric and ascorbic, present in a juice sample by titrating with a strong base such as sodium hydroxide, NaOH.
NaOH reacts with both acids as shown below:

Equation 1

$$C_3H_5O(COO\mathbf{H})_3\ (aq) + 3\ Na\mathbf{OH}\ (aq) \rightarrow Na_3C_3H_5O(COO)_3\ (aq) + 3\ \mathbf{H_2O}\ (l)$$

Citric acid
*Note: It requires three moles of hydroxide to titrate every mole of citric acid.

Equation 2

$$\mathbf{H}C_6H_7O_6\ (aq) + Na\mathbf{OH}\ (aq) \rightarrow NaC_6H_7O_6\ (aq) + \mathbf{H_2O}\ (l)$$

Ascorbic acid
*Note: It requires one mole of hydroxide to titrate every mole of ascorbic acid.

Oxidation/Reduction (Redox) Titration

I_2 reacts with ascorbic acid **only**. I_2 can be formed from KIO_3 under acidic conditions.

Equation 3—Generation of iodine

$$\underset{(\text{from } KIO_3)}{IO_3^-\ (aq)} + \underset{(\text{from KI})}{5I^-\ (aq)} + \underset{(\text{from HCl})}{6H^+\ (aq)} \rightarrow 3I_2\ (aq) + 3H_2O\ (l)$$

Ascorbic acid is readily oxidized to dehydroascorbic acid by iodine (see Equation 4 below).

Equation 4—Oxidation of ascorbic acid

$$C_6H_8O_6\ (aq) + I_2\ (aq) \rightarrow C_6H_6O_6\ (aq) + 2I^-\ (aq) + 2H^+\ (aq)$$

Once all the ascorbic acid is consumed by I_2, we will see a blue color. If you're curious how we know the reaction is done (and has reached the equivalence point), then read on! Once the vitamin C runs out, excess iodine forms a starch- I_3^- complex signaling that the neutralization is complete, as shown in the equations below:

Excess iodine forms triiodide

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

Endpoint is reached

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

Because the redox titration only involves a reaction of the ascorbic acid, 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.

Experiment 7 Turn-in Sheets

Titration of Juice

Procedure

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

1. Use a volumetric or graduated pipet to measure out approximately 10 ml of juice. [2] Record the precise volume of juice using an appropriate number of significant figures (a volumetric pipet has an uncertainty of ±0.01 mL). Transfer the juice to a clean 125 ml Erlenmeyer flask. Add about 25 mL of distilled water. (Does adding water change the number of moles of acid in your sample?) Add 2-4 drops of phenolphthalein indicator to the flask.

2. Obtain about 50 mL of 0.0500 M NaOH. Prepare and fill your buret with NaOH solution.

3. Record your starting buret reading (have your lab partner verify your measurement—use the proper number of significant figures!).

4. Titrate the juice sample. The endpoint may be easier to see if you have a white sheet of paper under the flask. Stop after the drop of hydroxide that results in a faint pink color that persists for at least 30 seconds.

5. Record your ending buret readings so that you can calculate the volume of NaOH added.

Repeat the titration twice more with two new samples of juice by repeating steps 1 through 5. Take only enough NaOH for two more trials.
Make sure to rinse the flask between trials.

Disposal of reagents: Everything should go into the appropriate waste container in the fume hood.

Safety Precautions

HCl is corrosive and NaOH is caustic. 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.

Use this space to record your observations and/or data

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

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.

Use this space to record your observations and/or data

2. Use a volumetric or graduated pipet to measure out 5 ml of juice. Record the precise volume of juice using an appropriate number of significant figures . (A volumetric pipet has an uncertainty of ±0.01 mL)

3. Transfer the juice to a clean 125 ml Erlenmeyer flask. Add the following (approximate amounts given; actual amounts used do not need to be recorded): 50 mL distilled water, one pipet full of 1.0 M hydrochloric acid, a spatula-tip full of KI, and 5 drops of 3% starch solution to the flask and swirl to mix the contents.

Safety: HCl is corrosive and KI is a possible skin and lung irritant.

4. Record your starting buret reading (use significiant 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.

Repeat the titration twice more with two new samples of juice by repeating steps 2 through 4. Take only enough KIO_3 for two more trials.
Make sure to rinse the flask between trials.

Disposal of Reagents: Everything should go into the appropriate container in the fume hood.

[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.

Data

Construct a data table that organizes all of your data for each trial and for each titration (Part 1 and 2). Make sure to include the precise volume of juice used for each trial, the starting and ending buret readings, the volume of titrant added, and an average value of this volume for your trials (or report the best value).

Don't forget to use units and significant figures.

Calculations

Show your work and use the proper number of significant figures.

1) Based on the average or best volume of KIO_3 used and equations **3** and **4** on the first two pages of this lab, determine the number of moles of ascorbic acid in 5.00 mL of juice according to your redox titration.

2) A) Calculate the number of moles of ascorbic acid in a 10.0 mL aliquot of juice.

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

   B) Calculate the number of moles of H^+ from ascorbic acid in a 10.0 mL aliquot of juice. Hint: Use equation 1 or 2.

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

4) Based on your answer to #3 above, how many total moles of H^+ (from citric and ascorbic acids combined) were neutralized? (Hint: What is the mole ratio of H^+ to OH^- in an acid-base reaction?)

5) Use your answers to #2 and #4 to determine how many moles of H^+ were neutralized from citric acid alone.

6) How many moles of citric acid are in each 10.0 ml aliquot? (Hint: How many moles of H^+ are in one mole of citric acid? See equation 1).

Follow-up Questions:

1) Was there more citric acid or ascorbic acid in your juice sample? Assuming juice to have a density of 1.00 g/mL, calculate the mass percent of citric acid and the mass percent of ascorbic acid in your juice.

$$\text{mass }\% = \frac{\text{mass of acid}}{\text{mass of juice}} \times 100\%$$

2) Could the titration described in this experiment be performed with orange juice or purple grape juice? Explain.

3) a. Based on your results in this experiment, calculate the average number of milligrams of vitamin C per cup of juice. From question 6 on page 72, you should already know your concentration in units of moles per 10 ml. Convert this to mg per cup.

Conversions:
1 cup = 8.0 fluid ounces
1 gal = 128 fluid oz.
1 gal = 3.785 L

b. The minimum daily recommended dose (daily value, DV) of vitamin C is 60. mg. Calculate the % DV of vitamin C in a cup of juice based on your results. (Your answer might be greater than 500%).

%DV = (mg of vitamin C obtained / 60. mg) x 100%

4) If other acids in the juice had reacted with the I_2 how would this have affected the accuracy of the reported %DV above? Would the %DV reported be higher or lower than the actual % DV?

Pre-Lab Assignment (2-pages)

Refer to Chapter 6 in your textbook on solutions, concentration (molarity), and titrations.

1) Why is a redox titration necessary in addition to the acid-base titration in this experiment?

2) If a buret is graduated every 0.1 ml, how many decimal places should you report for a measurement?

3) 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 27.3 ml of 0.1003 M NaOH to reach the endpoint of the titration. How many moles of acid (H^+) were in her juice aliquot?
(Hint: You cannot use Equations 1 or 2 since the juice is a mixture of both acids--both a source of H^+--and you don't know the ratio yet so you can only deal with the total H^+.)
(2nd Hint: What does sodium hydroxide react with? In what mole ratio?)

4) The student measured out a second 15.0 ml aliquot of juice, then added HCl, KI, and starch. It took 10.0 ml of 0.00100M KIO_3 to reach the endpoint of the titration.

   a. How many moles of KIO_3 (or IO_3^-) were needed to reach the equivalence point?

   b. Based on part a, how many moles of I_2 were formed from KIO_3?
   (Hint: Use an equation given on the first two pages of this lab)

   c. Using your answer in part b, how many moles of ascorbic acid were present in the juice sample?

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5) In question 3 you calculated the total moles of H^+ (from both citric and ascorbic acids) in 15.0 ml of juice while in question 4 you found out the moles of just ascorbic acid were present in the sample.

   a. Calculate the moles of H^+ from citric acid alone in the juice sample.

   b. The number of H^+ ions that dissociate from an acid is not equal to the number of acid molecules from which they dissociate if the acid is polyprotic (contains more than one acidic hydrogen).
   Calculate the moles of citric acid in the juice sample (Hint: How many hydrogen ions can dissociate from one molecule of citric acid? Use an equation given on the first page of this lab.)

6) What would you see (or not see) if you did not add phenolphthalein to the flask in question 3?