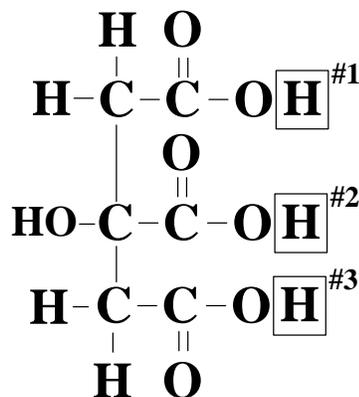


THE DETERMINATION OF CITRIC ACID IN FRUIT JUICES

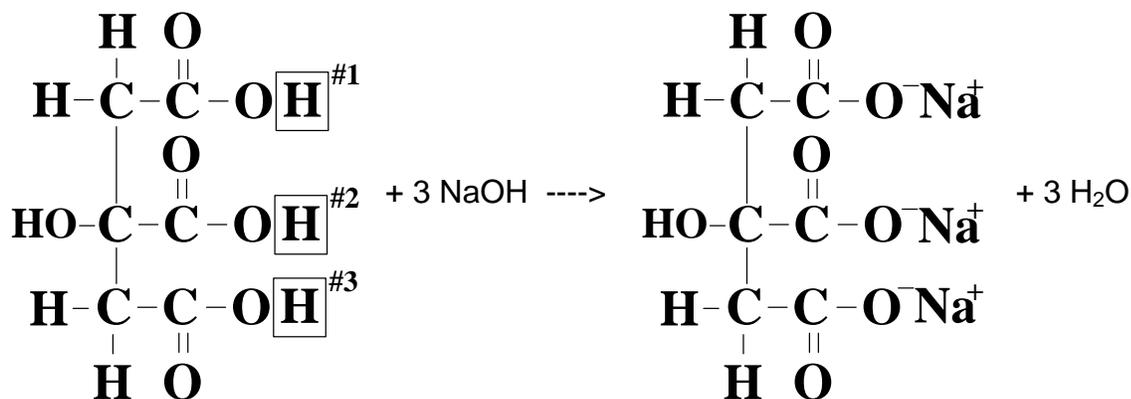


INTRODUCTION

Citric acid is a naturally occurring acid which, as implied in the name, is found in all citrus fruits. We will be investigating four such fruit juices today: orange, grapefruit, lemon, and lime. Citric acid contains three carboxylic acid functional groups and has a molecular formula of $\text{H}_3\text{C}_6\text{H}_5\text{O}_7$. The carboxylic acid functional group is pictured to the left.



Structurally, citric acid is written as pictured. Each proton of each carboxylic acid group has been numbered. Citric acid, when dissolved in water, becomes a triprotic acid capable of donating 3 protons (see the numbered hydrogen atoms in the structure). If a strong base is added to the acid, it will also react via a neutralization reaction to form a salt and water. Each of the numbered hydrogens (H^+) will react with one hydroxide ion (OH^-) from the NaOH to form water (H_2O). A sodium ion (Na^+) remaining from the now dissociated sodium hydroxide will take the place of each hydrogen. The reaction is pictured at the top of the next page.



It is important in a number of everyday items, such as orange juice, that the citric acid be quantitatively measured. Industries manufacturing juice products must know the amount of juice, both for regulatory purposes (FDA) and for their own manufacturing specifications. They can quantitatively determine the amount of juice by measuring the amount of citric acid. The procedure commonly used to do this takes advantage of the known reactivity of citric acid with sodium hydroxide and is known as a **titration**.

A titration is a means of quantitative analysis in which the substance to be measured (in a liquid solution) is reacted stoichiometrically with another reagent (called a titrant) until it has completely reacted. The end of the reaction is usually signaled with the appearance of a color from another non-interfering substance called an indicator. In the case of the citric acid titration, a known amount of orange juice is measured into an Erlenmeyer flask with an indicator solution containing phenolphthalein (the indicator). Sodium hydroxide, at a known concentration, is then carefully added into the sample until all of the acid has reacted. When all of the acid has completely been neutralized, the addition of 1 additional drop of the sodium hydroxide solution, the titrant, causes the solution to become basic. The basic solution will be marked by the appearance of a pinkish color in the solution of orange juice.

The device used to add the titrant (NaOH) to the juice sample is called a buret. It allows us to measure the exact amount of solution added during the titration. Knowledge of this, the concentration of NaOH solution in moles/liter, and the known stoichiometry of the reaction allows us to calculate the citric acid concentration in the juice sample.

TECHNIQUES

Using a Buret

1. Rinse the buret barrel, stopcock and tip several times with 5-10 mL of distilled water. Close the stopcock.
2. Rinse the buret with several 3-5 mL portions of titrant. Tilt and roll the buret so that the titrant comes in contact with the entire inner surface of

the barrel. Drain each rinse through the buret tip and discard down the drain. Do not reuse this solution.

3. Place the buret in the buret clamp which is fastened to a ringstand. Close the stopcock and fill the buret with fresh titrant to just above the zero mark. Open the stopcock briefly to remove any air bubbles in the tip and be sure the meniscus is below the zero mark. Wait 30 seconds before reading and recording the volume (± 0.01 mL).
4. See the "Meniscus" section on page 12 for how to read a meniscus.
5. The buret should not be refilled between repeat titrations if there is sufficient titrant volume remaining.
6. To clean the buret, open the stopcock and rinse well with tap water and soap. Rinse twice with distilled water and place the buret with the tip up into the buret clamp to drain. Be sure the stopcock is open.

SAFETY AND DISPOSAL

- The titrant used in this lab is 0.5 M NaOH. At this concentration, the basic solution can cause skin irritation and damage the finish on the table tops. If spilled, please neutralize with acetic or boric acid and wash your skin with lots of water.
- All solutions may be disposed of down the drain with water.
- Do not put pH paper down the sink.

EXPERIMENTAL PROCEDURE



will appear to indicate helpful hints, additional information, or interesting facts.

I. Preparation for titration

- A. Using the procedure in the Techniques section, set up the buret using 0.5 M NaOH as the titrant. Make sure to record the molarity of the NaOH. Record the initial volume. (Data Sheet Item #1).
- B. Commercial juice products will be used.
- C. One pair should perform the titrations on the orange and grapefruit juices while the other pair should perform the titrations on the lemon and lime juices. Information should be shared amongst both

pairs so that everyone has the data concerning all four citric fruit juices.

- D. Measure 10.0 mL of the juice sample in a 10 mL graduated cylinder. Record the exact volume of juice used if not 10.0 mL (for example, 9.8 mL) and pour the juice into a clean Erlenmeyer flask.
- E. Add approximately 30 mL of water and 3 drops of phenolphthalein solution. Record the initial pH value of the juice using pH paper.

II. Titration - Fruit Juices

- A. Titrate the unknown solution with the sodium hydroxide with constant swirling until 1 drop of NaOH causes the permanent appearance of a pink color in the solution.



The closer you get to the end point, the longer the pink color will persist. The term “permanent” refers to the point at which the pink color remains even with swirling and mixing.

- B. Rinse the inside of the flask by squirting with water and swirl to make sure pink color remains.
- C. If pink color doesn't remain, continue adding NaOH dropwise until the pink color remain.
- D. Record the final volume reading of the buret and the final pH value of the juice. (Item #2).
- E. Repeat this procedure two more times for a total of three trials for each juice sample.

III. Calculations

For each trial, calculate the following:

1. The volume of NaOH used
2. The moles of NaOH used
3. The moles of Citric Acid in sample
4. The grams of Citric Acid in sample (MM of citric acid = 192 grams/mole)
5. The % (mass/volume) Citric Acid in sample.

For each juice, calculate the average % Citric Acid in sample. Share and discuss your data with the two other students at your lab bench.