

Determination of Citric Acid in Powdered Drink Mixes

Citric acid and its salts (sodium citrate and potassium citrate) are found in many foods, drinks, pharmaceuticals, shampoos, and cosmetics. The tartness of citrus fruits and beverages is due to citric acid.

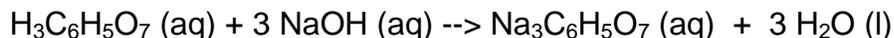
Today you are working in the quality control department at a manufacturing plant that produces a powdered drink mix. The quality control department monitors the percent citric acid in samples of the powdered drink mix to verify that the percentage is correct. If the powdered mix contains too much citric acid, the drink mix will be too tart; too little citric acid will produce a drink that lacks tartness. The tartness of the powdered mix is important to consumers buying this product.

Before beginning your experiment, make an estimate as to what percent of the drink mix is citric acid. Hint: Read the ingredients listed in the sample of the powdered drink mix. Ingredients present in the greatest amount are listed first. What percent of the drink mix is citric acid?

The quality control department will be using an acid-base titration to determine the percentage of citric acid in the sample.

Powdered drink mixes such as Kool-Aid contain citric acid ($\text{H}_3\text{C}_6\text{H}_5\text{O}_7$) and ascorbic acid (Vitamin C) in approximately a 100:1 (w/w) ratio. Both citric acid and ascorbic acid will react with the base NaOH. Since ascorbic acid is present in such an insignificant amount compared to citric acid, we will be concerned only with the total moles of acid present and will designate these moles as 'citric acid' moles. [Please note: It is possible to determine the actual amount of ascorbic acid present by titrating the sample with potassium iodate (using a starch indicator). See reference below.]

The powdered drink mix will be weighed and quantitatively dissolved in water. A volumetric portion of the solution (called an aliquot) will be titrated with NaOH using thymol blue as the indicator.



Citric acid

Sodium citrate

During a titration, a solution of known concentration, called a standard solution, undergoes a specific chemical reaction (of known stoichiometry) with a solution of unknown concentration. The moles of the "known" reactant can be determined from the molar concentration and volume of reactant used (Molarity x Volume = moles). The moles of the "known" reactant can be stoichiometrically related to the moles of the "unknown" reactant. A balanced equation is needed to determine the mole ratio of the reactants. The concentration of the "unknown" reactant and the amount present can be calculated.

An indicator is added to the reaction flask to determine the endpoint of the titration. Acid-base indicators are usually dyes that change colors at certain pH values. The indicator is one color in the acid form and another color in the basic form. Indicators are chosen such that they change colors near the equivalence point (where a stoichiometrically equivalent amount of acid and base is present). Phenolphthalein is often used in acid-base titrations (see the illustration in our textbook). However in this experiment, thymol blue will be the indicator, because of the color of powdered drink mixes. Thymol blue is a diprotic acid and has two color changes. At $\text{pH} < 2$ it is red. At pH values between 2 and 8.5, it is yellow. At $\text{pH} > 8.5$ it is blue.

At the start of the experiment excess acid is present in the flask, the solution is acidic and will appear red to yellow as base is added. At the end of the titration one drop of excess base will turn the solution to sky blue. The indicator changing to its basic color signals the endpoint of the titration.

To learn more about titrations, read the sections of our textbook dealing with molarity, acid-base reactions, and stoichiometry.

Reference: This experiment was adapted from Journal of Chemical Education Vol 81, No. 1, Oct. 2004 "Quantitative Determination of Citric and Ascorbic Acid in Powdered Drink Mixes", Sigmann and Wheeler.

Objectives:

1. Prepare a solution in a volumetric flask.
2. Use a volumetric pipet to transfer an aliquot of the diluted solution.
3. Use a buret to titrate a solution.
4. Determine the molarity of the prepared citric acid solution.
5. Calculate the grams and mass percentage of citric acid present in the sample.

Chemicals:

1 package Kool-Aid powdered drink mix
100 ml of 0.200 M NaOH (standardized)
Thymol blue (0.04% w/w aqueous)

Equipment:

50.0 ml Buret
10.0 ml Volumetric Pipet
250.0 ml Volumetric Flask and Stopper
250 ml Erlenmeyer Flask and Stopper
Pipet bulb
three 125 mL Erlenmeyer flasks
piece of white paper
buret clamp and stand

Download the Data Spreadsheet.

Diluting the Kool-Aid sample:

1. Obtain a package of Kool-Aid powdered drink mix.
2. Weigh the unopened package and record the mass to the nearest ± 0.0001 gram.
3. Carefully open the package and pour the mix into a 250.0 mL volumetric flask. Label the flask with your name and its contents.
4. Weigh the empty package (including all torn pieces of the wrapper) and record its mass.
5. Add approximately 200 mL of R.O. water, stopper, and mix well.
6. Add more R.O. water until the bottom of the meniscus rests on the 250.0 mL volumetric mark. (A disposable eye dropper is helpful). Accuracy with this measurement will affect your overall citric acid concentration!
7. Stopper the flask. Invert and mix well. The solution will appear cloudy due to the presence of insoluble calcium phosphate.

Obtaining the chemicals needed:

1. Obtain about **100 mL** of NaOH solution in a 250 mL Erlenmeyer flask and stopper the flask. Record the molarity of the NaOH solution and label the flask.
2. The vials of thymol blue indicator will be shared by all lab groups.

Rinsing the buret:

The buret must be cleaned prior to beginning the titration.

1. Rinse the buret with 3 - 50 mL portions of R.O. water. The water should flow freely with the stopcock open and there should be no leaks with the stopcock closed. Be sure to look for air bubbles in the tip of the buret.
2. Before filling the buret with the NaOH solution, rinse the buret with three 5.0 mL portions of the NaOH solution. Swirl and coat the sides of the buret with NaOH. If air bubbles persist, try to dislodge them with a light tapping or swirling.
3. Fill the buret to slightly above the top markings with NaOH. Open the stopcock slightly so that the meniscus stops at or just below the upper most mark. This measurement will be your initial volume for your trial titration. Fix the buret to the stand with a buret clamp.
4. **Practice reading the buret to ± 0.05 mL.**

Reminder about reading the buret:

When you are reading your buret, you are looking for the volume of NaOH used. You will be reading the buret unlike most cylinders and beakers. For example, if the buret reads at 20.00 mL, it does not mean that 20.00 mL remains in the buret.

Consider this the final reading of the titration. Subtract your initial volume from the final reading and you will have calculated the volume used.

Example: Initial reading = 5.00mL
Final reading = 20.00mL
Volume used = 15.00mL = (20.00ml - 5.00ml)

When reading a buret for a volume, you are reading it opposite from how you read a graduated cylinder. Most volumes are measured from the bottom to the top. Burets are instead read **from top to bottom**. If the meniscus is below the reading of 10 mL and above the reading of 11mL, your volume will be **less** than 11mL.

Doing a trial titration:

1. Fill the buret with the NaOH solution and record the volume where the meniscus is located, as the initial volume. (*It should be near 0.00 mL not 50.0 mL.*) **Read the buret to +/- 0.05 mL. Record this volume as the initial volume.**
2. Pipet 10.0 mL of the prepared Kool-Aid solution into a clean 125 mL Erlenmeyer flask. **Add 5 drops of thymol blue indicator to the flask.**
3. Place a sheet of white paper under the flask (to enhance the color change).
4. Begin the titration by adding 1.00 mL increments of NaOH. SWIRL the flask as NaOH is added to mix the contents. As you approach the end point, the yellow color will fade. Continue swirling and adding NaOH until 1.0 mL causes the solution to completely change to blue for 30 seconds or longer.
5. Record the volume of the NaOH in the buret, reading the volume at the meniscus. This is the **final volume**. Subtract the initial volume from the final volume to determine the volume of NaOH needed to approximate the end point.
6. All solutions used in this experiment can be rinsed into the sink with water.

Doing the actual accurate titrations:

1. Refill the buret with the NaOH solution and record the volume, where the meniscus is located, as the initial volume. (It should be near 0.00 mL not 50.0mL) **Read the buret to +/- 0.05 mL. Record this volume as the initial volume.**
2. Pipet 10.0 mL of the prepared Kool-Aid solution into a clean 125 mL Erlenmeyer flask. **Add 5 drops of thymol blue indicator to the flask.**
3. Using the volume of NaOH added in the trial titration subtract 2 mL from that volume. This is the amount of NaOH that can be added safely before a complete color change will occur.
4. Rinse any droplets that are clinging to the walls of the Erlenmeyer flask with a small stream of R.O. water from the squeeze bottle. SWIRL the flask to mix its contents.
5. While swirling, add small volumes of NaOH until one drop changes the color to blue. The color change should persist for 30 seconds or longer. Record this volume as the **final volume**. The volume of NaOH added can be calculated by subtracting the initial volume from the final volume.

6. Do four accurate titrations. The four trials should agree **within +/- 0.20 mL** variance in the volume of NaOH required to obtain the color change

Calculations:

1. Calculate the molarity of the Kool-Aid solution for each titration. Does each molarity have the correct number of significant figures?
2. Determine the moles of citric acid in the 250.0 mL solution.
3. Determine the molar mass of citric acid.
4. Determine the grams of citric acid in the sample.
5. Determine the mass percent of citric acid in the powdered sample.

Follow-up questions: On a separate paper, answer these questions and show all calculations.

1. Are there any colored drink mixes that would be difficult to use in this experiment? Explain why.
2. How many grams of citric acid are needed to prepare 175 ml of .0683 M solution of citric acid?
3. How many milliliters of .726 M NaOH would be needed to titrate 25.00 mL of .317 M citric acid solution?
4. Citric acid is produced by animals and plants during metabolism. The normal concentration of citric acid in human blood is about 2 mg per 100 mL. Calculate the molarity of citric acid in human blood?