Chemistry 3A

Introductory General Chemistry

Experiment 12b

Determination of Citric Acid in Soda

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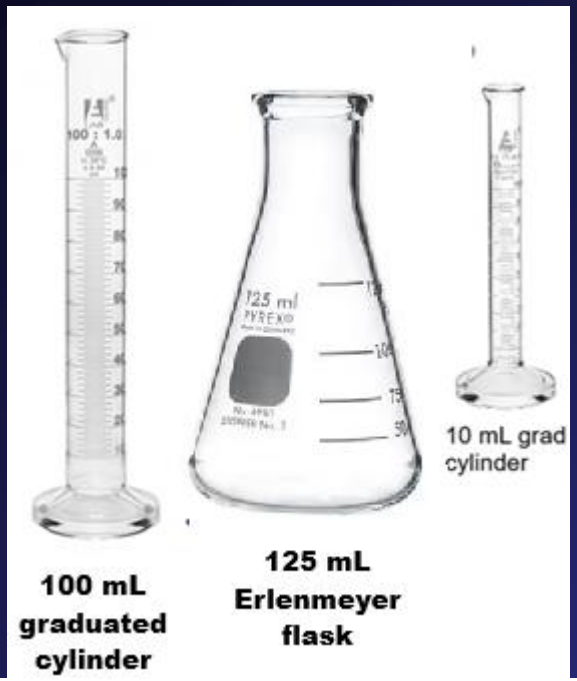
Introduction

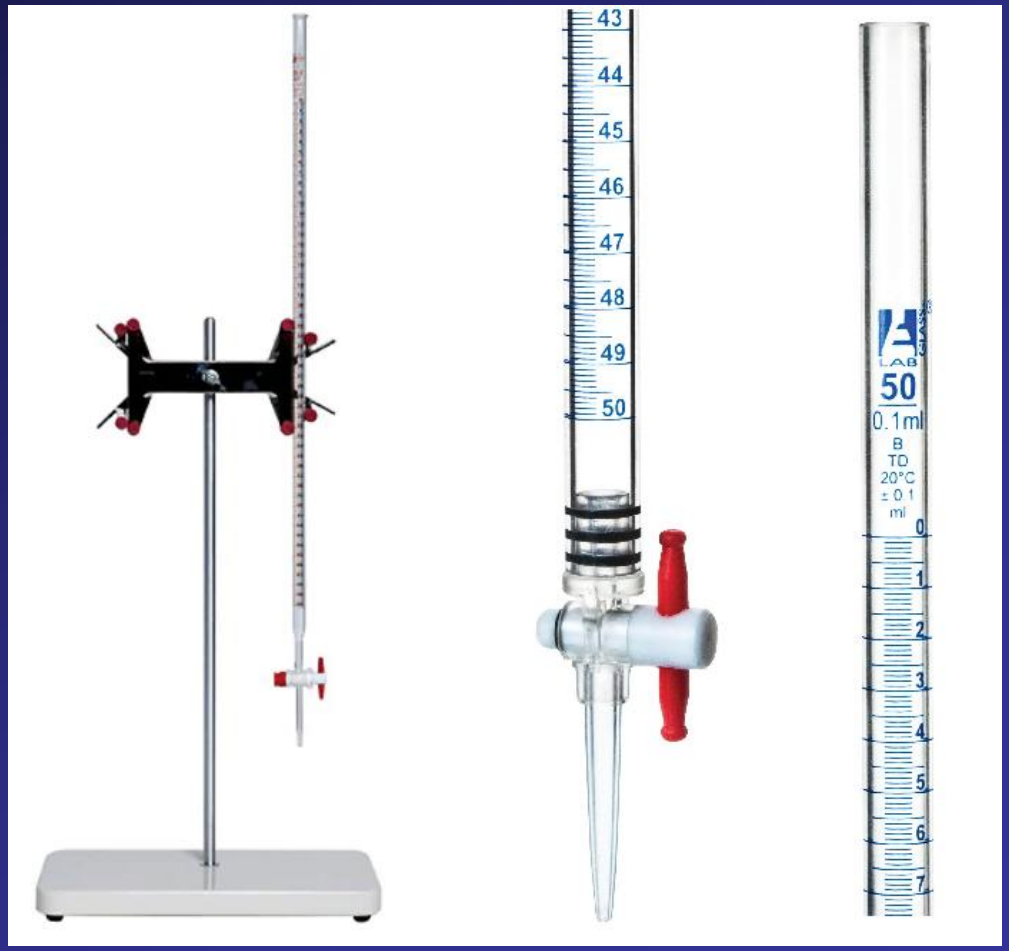
- Titrations are used to determine quantitatively amounts (as concentrations) of a substance in solution
- This experiment will use sodium hydroxide (NaOH) as a titrant to determine amount/concentration of citric acid in a lemon-lime soft drink

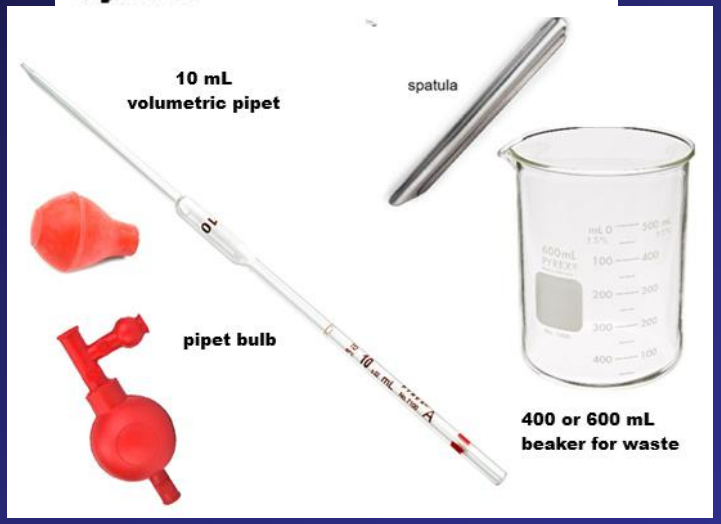
Procedure

- The titrant is placed in a buret and the analyte to be titrated will be placed in an Erlenmeyer flask
- Small volumes (aliquots)

Equipment You Will Use







Consumables You Will Use

Procedure

This experiment has two parts

- In part 1, you will do at least 3 trials of standardizing the concentration of a NaOH solution using oxalic acid dihydrate. This involves getting a mass of oxalic acid on the balance, dissolving it, and neutralizing it with a color pH indicator to its endpoint
- In part 2, you will use the NaOH solution to neutralize another acid, citric acid, determining the volume you used, to determine the concentration of the acid in a soft drink. This too will be done in 3 trials
- In each trial of all parts, it is necessary to achieve the proper color of the indicator to be accurate

Procedure

Preparing the buret

1. Rinse the buret with 5 mL DI water three times (turn buret on side and rotate it). Collect the rinse in a large waste beaker.
2. Repeat rinsing but with 5 mL of the titrant NaOH solution
3. Clamp the buret to the stand and with the waste beaker under it and stopcock open, add several mL to buret. As it flows, close stopcock. Ensure flow has filled buret tip

Procedure

Part I-Standardization

4. Fill buret with ~0.1 M NaOH titrant between the 0 and 3 mL mark
5. Record initial buret reading to correct precision: if the smallest division mark is 0.1 mL, then the precision should be ±0.01 mL
6. Oxalic acid
   a) Put glassine weigh paper on balance and tare (zero)
   b) Spoon out between 0.1000 and 0.1200 g oxalic acid dihydrate and record mass
   c) Transfer the acid completely without spilling into flask
7. Add about 10 mL DI-water in 10 mL graduated cylinder to the flask with acid, swirl to dissolve
8. Add 2-3 drops phenolphthalein to flask

Procedure

Part I-Standardization

9. Place piece of white paper under flask to observe any color change
10. Swirl flask and quickly add 10 mL NaOH from buret
11. Drop-by-drop titration
    a) The stopcock will be turned only to permit drop by drop addition of NaOH. Turn the stopcock on and off in controlled manner to dispense drops as necessary. As flask is swirled with each drop, look for pink color to appear. With each drop, the color may disappear more slowly. As the endpoint is reached, a very faint pink color will persist in solution.
    b) The best control is to quickly rotate stopcock by 180°, or to form a tiny drop in the tip, and touch tip to inside of flask wall

Procedure

Part I-Standardization

12. Record the final buret reading
13. Repeat the standardization steps for additional trials, and especially if over-titrations occurred (the pink is too intense/bright)

Procedure

Part II-Determination

14. Get about 40 mL of soft drink in small beaker. Note it will have lost its carbonation.
15. Use a 10 mL volumetric pipet to transfer precisely 10.00 mL to clean, well-rinsed Erlenmeyer flask.
16. Add 2-3 gtt phenolphthalein indicator to flask
17. Fill the buret to between 0-3 mL marks with the ~0.1 M NaOH solution.
18. Titrate the soda as for the standardization procedure
19. Record final volume
20. Repeat the number of trials.

Example Data Analysis

Part I-Standardization

1. Calculate the moles of NaOH reacting with the known mass of oxalic acid. Note that 2 mol NaOH reacts with 1 mol oxalic acid
2. Convert the NaOH titrant volume from mL to L
3. Calculate the concentration (molarity) of NaOH from the standardization

Part II-Determination

1. Calculate the liters NaOH (from mL) titrant used for the citric acid
2. Calculate the mole citric acid using mean NaOH concentration and volume NaOH. 3 mol NaOH reacts with 1 mol citric acid

Example Data Analysis

REFERENCE VALUES (FROM PRE-LAB QUIZ)

Oxalic acid dihydrate formula $HOOC-COOH * 2\ H_2O$

Oxalic acid dihydrate molar mass 126.068 g/mol

PART I- STANDARDIZATION

	Trial 1	Trial 2	Trial 3	Trial 4 (optional)
Mass of oxalic acid	0.1049 g	0.1194 g	0.1084 g	
Initial Buret Reading (NaOH)	2.54 mL	1.81 mL	2.34 mL	
Final Buret Reading (NaOH)	18.85 mL	21.15 mL	19.05 mL	

* Show all work for trial 1on the next page.

	Trial 1	Trial 2	Trial 3	Trial 4
Moles of sodium hydroxide	1.664×10^{-3}	1.893×10^{-3}	1.720×10^{-3}	
Net Volume NaOH used (L)	1.63×10^{-2}	1.93×10^{-2}	1.67×10^{-2}	
Molarity of sodium hydroxide	0.102	0.0979	0.103	
Average Molarity	0.101 mol/L = 0.101 M			

Example Data Analysis

Calculations Part I: Standardization:

Show your mathematical setup for ***Trial 1*** for each of the following, complete with units:

Moles of sodium hydroxide:

$$0.1049\ g\ OAA \times \frac{1\ mol\ OAA}{126.068\ g\ OAA} \times \frac{2\ mol\ NaOH}{1\ mol\ OAA}$$

$$= 0.001664\ mol\ NaOH = 1.664 \times 10^{-3}\ mol\ NaOH$$

Net Volume NaOH used, in liters:

$$(18.85\ mL - 2.54\ mL) \times \frac{1\ L}{1000\ mL} = 0.0163\ L$$

$$= 1.63 \times 10^{-2}\ L$$

Molarity of sodium hydroxide:

$$\frac{1.664 \times 10^{-3}\ mol\ NaOH}{1.63 \times 10^{-2}\ L} = \frac{0.102\ mol\ NaOH}{L}$$

$$= 0.102\ M\ NaOH$$

Example Data Analysis

PART II- DETERMINATION

NaOH Concentration 0.101 M (from standardization procedure)

	Trial 1	Trial 2	Trial 3	Trial 4 (optional)
Volume of "6 Down" Soda	10.00 mL	10.00 mL	10.00 mL	10.00 mL
Initial Buret Reading (NaOH)	2.75 mL	1.77 mL	2.10 mL	1.40 mL
Final Buret Reading (NaOH)	18.95 mL	17.40 mL	17.75 mL	17.25 mL

* Show all work for trial 1on the next page.

	Trial 1	Trial 2	Trial 3	Trial 4
Volume NaOH used (L)	0.01620	0.01563	0.01565	0.01585
Moles of Citric Acid in "6 Down" Soda	5.454×10^{-4}	5.262×10^{-4}	5.269×10^{-4}	5.336×10^{-4}
Molarity of Citric Acid in "6 Down" Soda	0.05454	0.05262	0.05269	0.05336
Average Molarity in "6 Down" Soda	trial 1 was overtitrated; trials2-4 mean= 0.05289			

Additional Calculations

"6 Down" is about 10 times more concentrated in citric acid than 7 Up®, so to calculate the realistic citric acid concentration in 7 Up®, just divide by 10.

Average Molarity in 7 Up® Soda	$0.005289\ M = 5.289 \times 10^{-3}\ M$
Grams Citric Acid in one can of 7 Up® (355 mL)	5.289×10^{-3} mol/L x 0.355 L x 192.124 g/mol = 0.361 g CA 361 mg citric acid

Example Data Analysis

Calculations for Part II: Determination

Show your mathematical setup for ***Trial 1*** for each of the following, complete with units:

Net Volume NaOH used, in liters:

$$(18.95 - 2.75)\ \text{mL} \times \frac{1\ \text{L}}{1000\ \text{mL}} = 0.0162\ \text{L}$$

Moles of citric acid: CA = citric acid

$$0.0162\ \text{L solution} \times \frac{0.101\ \text{mol NaOH}}{1\ \text{L solution}} \times \frac{1\ \text{mol CA}}{3\ \text{mol NaOH}}$$

$$= 5.454 \times 10^{-4}\ \text{mol CA}$$

Molarity of citric acid in 6-Down Soda:

$$\frac{5.454 \times 10^{-4}\ \text{mol CA}}{10.00\ \text{mL soft drink}} \times \frac{1000\ \text{mL soft drink}}{1\ \text{L soft drink}} = 0.05454\ \text{M CA}$$

Molarity of citric acid in 7-Up*:

** "6 Down" is about 10 times more concentrated in citric acid than 7 Up®, so to calculate the realistic citric acid concentration in 7 Up®, just divide by 10.*

$$\frac{0.05454\ \text{M CA ("6 Down")}}{10} = 5.454 \times 10^{-3}\ \text{M CA ("7-Up")}$$

Grams Citric Acid in one can of 7 Up® (355 mL):

$$\frac{5.454 \times 10^{-3}\ \text{mol CA}}{1\ \text{L drink}} \times 0.355\ \text{L drink} \times \frac{192.142\ \text{g CA}}{1\ \text{mol CA}}$$

$$= 0.372\ \text{g CA} = 372\ \text{mg citric acid}$$

Clean Up

- Pour waste solutions in the aqueous solution carboy located in the fume hood (usually placed near door)
- Clean up other equipment as done previously in the lab