

Experiment # 6

Determining the percent composition of a mixture by acid-base

Objective

Determine the percent composition of a mixture of sodium carbonate (Na_2CO_3), sodium bicarbonate (NaHCO_3) and a neutral component using an acid-base titration.

Introduction

Chemists Johannes Bronsted and Thomas Lowry proposed that acid-base reactions can be described as proton (H^+) transfer reactions. According to this Bronsted-Lowry proposal, an acid is defined as any compound that donates a proton and a base is defined as any compound that accepts a proton in a proton-transfer or acid-base reaction. It is often useful to think of acids in terms of a conjugate acid-base pair, where the acid is the proton donor and the compound produced when the proton is transferred is the conjugate base as shown in equation 1,



where HA represents the acid and A^- represents its conjugate base. Compound B in equation 1 represents the base and BH^+ represents its conjugate acid.

All acid-base reactions involving weak acids or weak bases are equilibrium reactions with K_a representing an acid ionization constant and K_b representing a base ionization constant. Proton transfer for an acid can be represented as in equation 2



K_a for this reaction is defined by equation 3

$$K_a = \frac{[\text{A}^-][\text{H}^+]}{[\text{HA}]} \quad \text{Equation 3}$$

where the square brackets indicate molar (M) concentrations of the species HA, H^+ , and A^- . A large value for K_a indicates a higher level of ionization at equilibrium. More ionization indicates more H^+ and thus represents a stronger acid.

The hydrogen ion concentration in pure (neutral) water is 1.00×10^{-7} . An acidic solution is defined as one with a hydrogen ion concentration, $[\text{H}^+]$, which is greater than that of pure water. A basic solution is one with a hydrogen ion concentration less than that of pure water. Because these concentrations are so small it is more convenient to work in units of pH, where pH is defined as the negative log of the hydrogen ion concentration (equation 4).

$$\text{pH} = -\log[\text{H}^+] \quad \text{Equation 4}$$

Using equation 4 it can be seen that the pH of pure water is 7.00. An acidic solution is one with a pH less than 7.00 and a basic solution has a pH greater than 7.00.

Some acids, such as carbonic acid, H_2CO_3 , are called polyprotic acids because they have the capability to donate more than one proton. In general, the second ionization constant, K_{a2} , for a polyprotic acid is significantly smaller than the first ionization constant, K_{a1} . In other words, the second proton is more difficult to remove than the first and proton transfer will therefore occur at a much higher pH.

Acid-base indicators are often used to indicate pH because they change color within a small pH range. In this lab we will use two pH indicators. The first indicator, phenolphthalein, changes from colorless to pink at about pH 8. That is, it is colorless in solutions with a pH of less than 8 and pink in solutions with a pH greater than pH 8.0. The second indicator, methyl orange is red below pH 5 and yellow above pH 5.

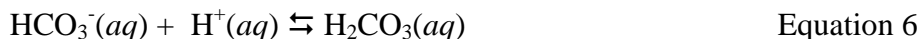
The purpose of this experiment is to analyze a mixture containing a neutral impurity and two salts of carbonic acid. This will be done by titrating the solution with acid using acid-base indicators to monitor the pH, and thus the progress of the reaction. The titration is performed in two steps, the first representing addition of one proton to carbonate ion, CO_3^{2-} , to produce bicarbonate, HCO_3^- ; and the second indicating addition of a second proton to bicarbonate to produce carbonic acid, H_2CO_3 .

In the first step, a standardized HCl solution is used to titrate the mixture in the presence of a pH indicator, phenolphthalein, which has an end point pH of about 8. Once the solution reaches this pH all of the sodium carbonate, but none of the bicarbonate, will have reacted with the acid. Therefore, the number of moles of sodium carbonate can be easily calculated knowing the stoichiometry of the reaction and the volume of acid used in the titration.



Note that sodium ions, being spectator ions, are not included in equation 5. The stoichiometry of equation 5 clearly shows that the number of moles of CO_3^{2-} present in the initial mixture is equal to the number of moles of acid added during the titration to the phenolphthalein endpoint.

The second step entails continuing the titration of the mixture resulting from step 1. Methyl orange, which has an endpoint at about pH 5, will be used as the pH indicator to monitor the protonation of bicarbonate. The bicarbonate in the mixture reacts with hydrochloric acid forming carbonic acid (equation 6) which then decomposes to form carbon dioxide and water (equation 7). Release of CO_2 gas into the atmosphere makes this reaction irreversible.



The number of moles of sodium bicarbonate can be easily determined from the stoichiometry of equation 6 and the volume of acid added. However, this value represents the total number of moles of bicarbonate, including both the bicarbonate that was formed in step 1 and the sodium bicarbonate originally present in the unknown mixture. Therefore, in order to calculate the amount of bicarbonate present in the original

mixture, one must subtract the amount of carbonate determined to be present in step 1 from the amount of bicarbonate present in step 2.

Procedure

Step 1

1. Rinse the buret with water then with about 5 mL of the HCl standard solution.
2. Fill the buret with standardized 0.1 M HCl solution. Record the exact molarity of the acid as noted on the bottle. Open the stopcock and fill the tip of the buret.
3. Obtain an unknown sample, record its identification code. Weigh out 0.20g (to +/- 0.01 g) of the unknown sample and transfer it to a 125 ml Erlenmeyer flask. Add about 15 ml of distilled water and swirl to dissolve the sample. At this point the solution must be colorless. If the sample does not dissolve completely, don't worry, it will when you start the titration.
4. Add two (2) drops of phenolphthalein indicator. The solution will turn pink.
5. Record the initial volume of acid in the buret. Titrate with the standard HCl until the solution *just* turns colorless. *It is extremely important not to over titrate!* If you do over titrate, make sure that a note about it is written in your observations. This solution will be used in step 2, do not discard it!
6. Record the final volume of the HCl solution in the buret.

Step 2

7. Add 2 drops of methyl orange indicator to the colorless solution resulting from the previous titration. The solution will turn yellow.
8. Record the initial volume of acid in the buret. Titrate with the standard HCl until the solution turns light salmon pink (or light peach pink). If the color is red you have over titrated.
9. Record the final volume of the HCl solution in the buret.
10. Dispose of the solution in the flask and clean the flask, rinsing very well with tap water, then distilled water.
11. Repeat Step 1 and Step 2 two more times each using a new 0.20 g sample of **the same** unknown each time.

Buret Cleanup

Drain the buret, put any remaining HCl solution into the waste container provided. Leave the buret secured to the ring stand. Close the stopcock and fill the buret with tap water. Drain the buret through the tip. Repeat the rinse once with tap water then once with distilled water. Return the buret to the instructor's cart.

Report

Show all calculations and remember to include discussion of results and error sources.

Calculations

For each step, the volume of HCl used must be recorded.

1. Calculate the number of moles of sodium carbonate in the unknown mixture. The molarity of HCl times the volume of HCl (in liters) equals the number of moles HCl ($M \times V = \text{mol HCl}$). According to the reaction stoichiometry (equation 5) at

- the phenolphthalein endpoint, the number moles of added hydrochloric acid is equal to the number of moles of sodium carbonate in the original mixture.
2. Using the reaction stoichiometry (equation 3) and the volume of HCl added calculate the total number of moles of sodium bicarbonate present in the reaction solution at the methyl orange endpoint.
 3. Calculate the number of moles of sodium bicarbonate in the original solution by subtracting the number of moles of sodium carbonate in step 1 from the total moles of bicarbonate titrated in step two. Remember that the sodium bicarbonate titrated in step 1 was converted into bicarbonate and was titrated in step 2 along with the sodium bicarbonate present in the original sample.
 4. Calculate the mass of sodium carbonate and sodium bicarbonate in the mixture by multiplying each by their appropriate molar mass.
 5. Calculate the weight percent [% (wt/wt)] of carbonate, bicarbonate, and the neutral component in your unknown. $\text{Weight \%} = (\text{calculated mass of compound in question}) / (\text{total mass of unknown sample}) \times 100\%$
 6. Calculate the mass and wt% of the neutral component based on the mass of the unknown mixture and the now known masses of Na_2CO_3 and NaHCO_3 .
 7. Calculate the average weight % for all determinations. You may also take a different approach and start by averaging the titrant volumes and work with averages throughout the calculations (particularly when the three sample masses are exactly the same). Remember not to round too early.

Discussion and Conclusion.

State your results, describe any problems encountered and how you solved them. Indicate how accurate you think your results are and why you think they are or are not accurate.

Chemistry 1225, Experiment 6, PRELAB

Name _____

1. Why does an aqueous solution of the unknown mixture turn pink upon addition of phenolphthalein in Step 1? Why does the solution become colorless at the end of titration in Step 1?

2. Calculate the number of moles of sodium carbonate present in a mixture of sodium bicarbonate, sodium carbonate and a neutral component if 6.0 ml of 0.100 M HCl were used to titrate 0.200 g of this mixture with phenolphthalein as the indicator.

3. Calculate the mass of sodium carbonate present in the mixture in question 2.

4. Calculate the number of moles of sodium bicarbonate originally present in the unknown mixture in question 2, knowing that 15.0 ml of 0.100 M HCl were used in step 2 of the titration, using methyl orange as the indicator.

