Acid-Base Titrations

Introduction

Titration is a convenient quantitative method for accurately determining unknown concentrations of solutions. A necessary requirement for its use is that a standard solution (a solution of known concentration) reacts chemically with the solution whose concentration is being determined. The standard solution is added to a solution of unknown concentration until all of the unknown solution has reacted. From the known quantity and molarity (or normality) of the standard solution and the measured volume of unknown solution used, the unknown concentration can be calculated. For example, standard base solution (NaOH) is added from a burette to an accurately known volume of the acid solution (HCl).

$$HCl(aq) + NaOH(aq) \text{ ----------> } H_2O(l) + NaCl(aq) \qquad (1)$$

This reaction (neutralization) can be written as a NET IONIC equation as follows:

$$H^+(aq) + OH^-(aq) \text{ ----------> } H_2O(l) \qquad (2)$$

When sufficient NaOH has been added to react with all of the acid, the titration is complete -- the equivalence point has been reached. Most acid-base solutions are colorless and determining when one reactant has been totally consumed is difficult by simple observations. To allow us to visually determine this point, we use compounds called acid-base indicators to tell us when a reaction is complete. Dyes (usually weak organic acids) whose colors depend upon pH are often used to signal the completion of acid-base reactions. Indicators must be carefully chosen based on the pH of the equivalence point of the titration. In this experiment, a strong base (NaOH) is being added to a strong acid (HCl). An indicator that changes color when the pH becomes greater than 7 (more base is added than necessary) is used. Titrations involving a strong acid and a strong base commonly employ phenolphthalein as an indicator. Phenolphthalein changes from colorless to red at a pH of ≈ 8-10.

At the point when the indicator changes color (also called the endpoint) and from the net ionic equation given above, we can see that the moles of H^+ equal the moles of OH^-. From the molecular equation (Eq 1), we also see that the number of moles of HCl equals the number of moles of NaOH at the endpoint.

$$\text{moles } H^+ = \text{moles } OH^-$$

$$\text{moles acid} = \text{moles base}$$

$$M_aV_a = M_bV_b$$

Preparation of a standard NaOH solution is not a simple task. Solid NaOH is hygroscopic - it readily absorbs water from the atmosphere. Thus, solutions of exact NaOH concentrations cannot be prepared by weighing out the calculated amount of NaOH and dissolving it in a given quantity of water. To prepare standard NaOH solutions directly, anhydrous NaOH must be weighed in a water-free environment (under normal conditions, you can observe the mass of NaOH increase as you weigh it on an analytical balance). Another problem in the preparation of standard NaOH solutions is the need to remove dissolved CO_2 from the water used. NaOH reacts with CO_2 to produce Na_2CO_3.

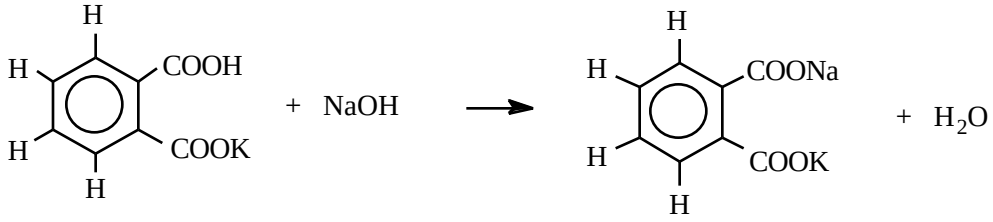
$$2NaOH(aq) + CO_2(aq) \text{ ---------> } Na_2CO_3(aq) + H_2O(l) \qquad (3)$$

NET IONIC REACTION:

$$2OH^-(aq) + CO_2(aq) \text{---------> } CO_3^{2-}(aq) + H_2O(l) \qquad (4)$$

When solutions of NaOH containing carbonates are added to acid, both the OH^- and the CO_3^{2-} react with the acid. Unless CO_2 is removed from the NaOH solution, acid-base titrations will be in error. To avoid a carbonate titration error, NaOH solutions are prepared from CO_2-free deionized water and protected from atmospheric CO_2.

A method less difficult than obtaining water-free NaOH and conditions and CO_2-free water is to prepare NaOH solutions and then determine the precise concentration by reacting the solution with a primary standard acid, potassium hydrogen phthalate (KHP). KHP is a non-hygroscopic (non water absorbing) compound obtained in high purity. It reacts with NaOH as indicated below:



The volume of NaOH solution required to react with a known weight of KHP is determined by titration. Since 1 mole of NaOH reacts with 1 mole of KHP, the concentration of NaOH can be calculated.

$$\frac{\text{mass of KHP}}{\text{MW of KHP}} = \text{moles of KHP}$$

$$\text{Moles of KHP} = \text{moles of NaOH (1:1 stoichiometry)}$$

$$\frac{\text{moles of NaOH}}{\text{volume of NaOH in L}} = \text{Molarity of NaOH (moles / L)}$$

The volume of NaOH in the above equation is the amount of NaOH required to reach the endpoint when titrating the KHP of known mass. Thus, in this experiment, we will not prepare a standard NaOH solution directly. We will prepare a NaOH solution and standardize it with KHP. Concentrations

Procedure

From the stockroom, each student should check out one 500 mL Florence flask with #2 stopper, two 125 mL Erlenmeyer flasks, one burette, and one burette clamp.

A. Preparation of NaOH.

1. Calculate the mass of NaOH required to make a 0.125 M NaOH solution.

2. Clean (but not dry) the 500 mL Florence flask.

3. Obtain the calculated amount of NaOH to the nearest 0.2 g using the automatic balance. The amount you obtain need not be exact since we will determine the actual concentration later.

4. Place the NaOH into the 500 mL Florence flask, add about 300 mL of deionized water, stopper, and shake until the sodium hydroxide has dissolved. Note that the solution process is exothermic - it produces heat. Once the NaOH dissolves, fill the bulb part of the Florence flask with deionized water and invert about 20 times to thoroughly mix the solution.

B. Standardization of the NaOH Solution With KHP.

1. Obtain three 0.450 - 0.550 gram samples of KHP using the automatic balance on sheets of weighing paper.

2. Transfer the KHP samples to clean (doesn't have to be dry) 125 mL Erlenmeyer flasks.

3. Reweigh the sheets of weighing paper and determine the mass of KHP transferred into each of the three flasks. Label the flasks to prevent mix-ups.

4. Add ≈ 25 mL of deionized water to each Erlenmeyer flask. Swirl the contents to dissolve the KHP completely. It may take up to 10 minutes to dissolve completely. Swirl frequently.

5. While you are waiting for the KHP to dissolve, clean your burette by rinsing at least twice with deionized water and twice with NaOH solution (10 mL of water or NaOH for each rinse is sufficient). Remember to also rinse the tip by running 1-2 mL of liquid through it. Over the sink, invert and rotate the burette allowing the liquid to completely rinse the sides of the burette while draining. Drain the tip with the burette inverted. Fill the burette with NaOH solution. Remove any bubbles on the sides by gently tapping the burette. Remove any air from the burette tip by running sufficient solution through it. Read the initial volume. It does not have to be zero.

6. Once the KHP in the flasks have dissolved, add 2-3 drops of phenolphthalein indicator to each Erlenmeyer flask.

7. Place a sheet of white paper under the Erlenmeyer flask. Record the initial burette reading and begin adding NaOH to the flask. While adding NaOH, swirl the solution. Initially, the pink color of the indicator may not appear and NaOH can be rapidly added. As more NaOH is added, the pink color lingers and the rate of addition should be decreased. A good rule is to add NaOH only as the pink disappears. Titrate the solution in the flask to a FAINT PINK endpoint. If drops of base fall on the inside wall of the flask, rinse it into the body of the solution with deionized water from your wash bottle. When the solution remains pink for at least 30 seconds, record the burette reading. Burettes should be read to ± 0.01 mL. Remember to read the bottom of the meniscus. A good endpoint is when one drop of base turns the solution from colorless to a faint pink.

8. Repeat Step 7 for the remaining two samples of KHP.

9. Determine the concentration of your NaOH solution. If any two molarities (normalities) deviate by more than ±2% repeat the titrations. You should have 3 titrations with no two calculated molarities (normalities) deviating more than ±2%.

C. Determination of The Concentration Of Unknown HCl.

1. Pipette 25.00 mL of the unknown HCl into a 125 mL Erlenmeyer flask using a volumetric pipette. Add 2-3 drops of phenolphthalein indicator.

2. Titrate the HCl with your standard base to a FAINT PINK endpoint. Record initial and final burette readings. Read at the bottom of the meniscus.

3. Titrate 2 more samples of HCl in a similar way. If any 2 titrations deviate by more than ±0.50 mL, perform more titrations. You should have 3 titrations with no two deviating more than ±0.50 mL.

Written Report
The written lab report should be neatly typed. Equations may be written into the lab report by hand. Your lab report should include the following sections:

1. **Title**
   Give the experiment a suitable title of your own making. As a subheading, indicate which class the report is written for (General Chemistry 1252 Lab), and the current date.

2. **Objective**
   State the objective for the experiment. The objective should be a short, one or two sentence statement concisely describing the objective of the lab. The objective is usually *chemical* in nature, not to "learn a technique".

3. **Theory**
   The theory section is the heart of the lab report. In this section, you discuss the chemical concepts or principles applicable to the experiment. In other words, what are the chemical principles that allow you to do this experiment and obtain the results you desire? Include all chemical reactions and mathematical equations applicable and clearly describe how you will use the equations to calculate your results. You should not include detailed procedures but only a brief overview of how you will obtain the data you need (the data which you will insert into your equations to calculate the final results). The theory section should smoothly take the reader from the objective to the results. The introduction included in the lab handout generally provides a good foundation for the theory section. The handout should be supplemented with information from your textbook. Do not copy the handout or textbook. *The lab report should be in your own words.*

***** The theory for this particular lab report should include a brief description of acid-base reactions, how the concentrations of acids can be determined via titrations with NaOH, how indicators can be used to determine the endpoint of a reaction, and why it is necessary to first determine the concentration of NaOH by titrating against KHP.

4. **Data**
   Tabularize your raw data, which are the actual measurements taken from your lab notebook. (Microsoft Word can very easily be used to make tables.) In this experiment, raw data includes each of the three masses of KHP, the three volumes of NaOH titrated against each mass of KHP, and the three volumes of NaOH titrated against the three samples of HCl.

5. **Calculations**
   Include all calculations leading to the results you obtained. Label all calculations by indicating what you are calculating. Show the equation used and then substitute your values for the apprpriate variables. *Use the correct number of significant figures and units in your calculations.*

   The following should specifically be included in the "Calculations" section for the titration exp.:
   a. For each of the 3 acceptable titrations of KHP, calculate the molarity of the NaOH. Show your calculations for each acceptable titration. Disregard any unacceptable titrations ($> \pm 2$ % error).
   b. Report the average molarity of the NaOH. It is this average value that is used for your calculations in Part b.
   c. Show calculations for each HCl titration. Report the molarity of the HCl for each titration. Report the average molarity of the unknown HCl.

6. **Results and Discussions**
   Tabularize your results and add any appropriate discussion, including the reproducibility and validity of your data (how good are your results, how do they compare with theory, etc.).

7. **Conclusion**
   For our purposes, the conclusion acts as an abstract, briefly summarizing the procedure, results, and conclusions. It should only be one short paragraph.

KHP Titrations

	Titration 1	Titration 2	Titration 3
Weight of KHP:	_____	_____	_____
Final Burette Reading:	_____	_____	_____
Initial Burette Reading:	_____	_____	_____
Volume of NaOH Used:	_____	_____	_____
Molarity of NaOH:	_____	_____	_____

HCl Titrations

	Titration 1	Titration 2	Titration 3
Volume of HCl:	_____	_____	_____
Final Burette Reading:	_____	_____	_____
Initial Burette Reading:	_____	_____	_____
Volume of NaOH Used:	_____	_____	_____
Molarity of HCl:	_____	_____	_____

Unknown Acid Titrations

	Titration 1	Titration 2	Titration 3
Volume of Unknown:	_____	_____	_____
Final Burette Reading:	_____	_____	_____
Initial Burette Reading:	_____	_____	_____
Volume of NaOH Used:	_____	_____	_____

Reagent Preparation for Standardization of NaOH

	Per Section
Sodium Hydroxide Pellets	100 g
Phenolphthalein	50 mL
0.1 M HCl	3.0 L
Potassium Hydrogen Phthalate	60 g
0.075 M H_2SO_4	3.0 L

Preparation of KHP: Dry the KHP at about 100^{o}C overnight to remove adsorbed water.

Preparation of Phenolphthalein: Dissolve 1 g of phenolphthalein in 50 mL ethanol and add 50 mL of water

Preparation of .1 M HCl: Dilute 200 mL of 12 M HCl (concentrated) to 20.0 L - this total volume is not critical

Preparation of 0.075 M H_2SO_4: Dilute 95 mL of 15.9 M H_2SO_4 (concentrated) to 20.0 L. Standardize this solution so we know the exact concentration before the students use it on Thursday.