Lab #1: Acid-Base Titration Revisit

Objectives:

1. To accurately prepare solution from a solid solute.
2. To understand the importance of standardizing a solution using a primary solution.
3. To accurately determine the concentration of an acid and a base using titration.

Introduction:

Even though titration between acids and bases is a common technique to determine unknown concentrations, a good analytical chemist must ensure that his or her titrant solutions are standardized. *A standardized solution means that the solution's concentration is known accurately*. A titrant solution stored in a container or a volumetric flask might react with components of the air above it making its true concentration different than what was labeled. This is especially true for NaOH and KOH solutions. Even making NaOH and KOH solutions directly from solids can be problematic. Solid alkali hydroxides have the property of absorbing water from the air (hygroscopic) and hence, it makes obtaining their accurate masses directly quite difficult. In addition, alkali hydroxides solutions can react with CO_2 in the air to produce Na_2CO_3. So unless the solvent is deionized water and the CO_2 is removed from the atmosphere during the making and storing of the base, the subsequent titration will be in error. Therefore, a proper titration can only be performed when the titrant is standardized on the same day by a primary solution.

A primary solution is a solution that can standardize a titrant solution properly. In order for a solution to be considered a primary solution, it must fulfill the following requirements.

- It should be 100.00% pure, although 0.01 to 0.02% impurities are tolerable.
- It should be stable to drying temperatures, and it should be stable indefinitely at room temperature. The primary standard is always dried before weighing.
- It should have a large molar mass (usually over 100 g/mol). A large molar mass will reduce experimental error since a relatively large amount of it will have to be massed in order to get enough moles to perform the titration and, the relative error in massing a greater amount of material is smaller than for a small amount. (Common primary solutions are KHP – Potassium Hydrogen Phthalate and Oxalic Acid Dihydrate.)

In order to perform a titration certain criteria must be met between the unknown solution and the primary standard. The requirements of a titration are:

- The reaction between unknown and standard should be stoichiometric. This means there should be a well-defined and known reaction between the two.
- The reaction should be rapid.
- There should be no side reactions, and the reaction should be specific. If there are interfering substances, they must be removed.
- There should be a marked change in some property of the solution when the reaction is complete. This may be a change in the color of the solution or in some electrical or other physical property of the solution. A color change is usually brought about by addition of an indicator whose color is dependent on the properties of the particular solution.
- The point at which stoichiometric amounts of each reagent have reacted is called the *equivalence point*. The point at which the reaction is observed to be complete is called the *end point*. The end point should coincide with the equivalence point.
- The reaction should be quantitative which means the reaction should proceed to completion.

In this particular lab activity, we will be using a solution of NaOH as a titrant after it has been standardized by a primary solution of $KHSO_4$. Afterwards, we will use the standardized NaOH solution to determine an unknown concentration of H_2SO_4.

Hypothesis / Pre-lab Exercise:

1. Calculate the mass of $NaOH_{(s)}$ to make 500 mL of a 0.100 M solution.
2. Write a net ionic equation for the reaction between $NaOH_{(aq)}$ and $KHSO_{4\,(aq)}$.
3. Calculate the volume of 0.100 M of $NaOH_{(aq)}$ needed to neutralize a solution made with 0.90 g of $KHSO_{4\,(s)}$.
4. Write a molecular equation for the titration of $H_2SO_{4\,(aq)}$ and $NaOH_{(aq)}$.

Materials:

Electronic Balance	Funnel	Volumetric Flask (500 mL)	Deionized Water
Beaker (250 mL)	Stirring Rod	Pipet Bulb	$KHSO_{4\,(s)}$
Masking Tape	Pipet (10 mL)	4 Erlenmeyer Flasks	$NaOH_{(s)}$
Scoopula	Ring Stand	Buret	$H_2SO_{4\,(aq)}$
Wash Bottle	Drying Oven	Buret Clamp	Phenolphthalein Indicator
Graduated Cylinder		Buret Funnel	Bromothymol Blue Indicator

Procedure:

A. Making 0.100 M of $NaOH_{(aq)}$

1. With the 250 mL beaker on the electronic balance, calibrate it to zero.
2. Using the scoopula and a beaker, carefully measured out the approximate mass of NaOH needed for the solution (see Pre-lab exercise 1). Record the actual measurement used.
3. Pour about 200 mL of deionized water into the small beaker. Using a stirring rod, dissolve as much of the NaOH as possible.
4. Pour the solution into the 500 mL volumetric flask using a funnel and a stirring rod.
5. Wash the small beaker, funnel and stirring rod with distilled water in a wash bottle. All washed fluid should be transfer to the volumetric flask during the actual washing. Be careful not to pass the mark on the volumetric flask.
6. Top up the volumetric flask with deionized water up to the mark. Cap the flask and shake. Afterwards, label your name and NaOH on a masking tape and place it on the volumetric flask.

B. Standardized $NaOH_{(aq)}$ using $KHSO_4$ as a Primary Solution

1. Coat the buret with the 0.100 M of $NaOH_{(aq)}$ from the volumetric flask (as prepared in Part A) at least twice, and discard the wash fluid in the sink.
2. Set up the titration apparatus with the ring stand, buret clamp, buret and buret funnel.
3. Fill the buret with the 0.100 M of $NaOH_{(aq)}$ using the buret funnel. Be sure not to pass the 0 mL mark.
4. Place an Erlenmeyer flask on the electronic balance and calibrate it to zero. Mass approximately 0.40 g to 0.50 g of pre-dried $KHSO_4$ (instructor has already dried it out ahead of time) into the flask. Record the result.
5. Add about 20 mL of deionized water to the Erlenmeyer flask with the solid $KHSO_4$. Afterwards, add 3 to 4 drops of phenolphthalein indicator.
6. Record the starting volume of the $NaOH_{(aq)}$. Begin titration to standardize $NaOH_{(aq)}$. Swirl the Erlenmeyer flask when adding the $NaOH_{(aq)}$. The endpoint will be a light pink color. Record the final volume of the NaOH added. Calculate the net volume of base added. (If the solution becomes dark pink, you have added too much $NaOH_{(aq)}$. Record the volume and the colour anyway.)
7. Repeat steps 3 to 6 until three consistent results are achieved.
8. Discard the solutions of the Erlenmeyer flasks down the sink. Rinse well with tap water first, and then rinse with deionized water. Dry the outside of the flasks.

C. Titrating $H_2SO_{4\,(aq)}$ using NaOH as a Standardized Solution

1. Coat the 10 mL pipet with the $H_2SO_{4\,(aq)}$ at least twice and discard the wash fluid down the sink.
2. Pipet 10 mL of $H_2SO_{4\,(aq)}$ to each of the four Erlenmeyer flasks.
3. To each Erlenmeyer flask, add 3 to 4 drops of bromothymol blue indictor.

4. Fill the buret with the 0.100 M of $NaOH_{(aq)}$ using the buret funnel. Be sure not to pass the 0 mL mark.
5. Record the starting volume of the $NaOH_{(aq)}$. Begin titration to determine the concentration of $H_2SO_{4\,(aq)}$. Swirl the Erlenmeyer flask when adding the $NaOH_{(aq)}$. The endpoint will be a green color. Record the final volume of the $NaOH_{(aq)}$ added. Calculate the net volume of base added. (If the solution becomes blue, you have added too much $NaOH_{(aq)}$. Record the volume and the colour anyway.)
6. Repeat steps 4 and 5 until three consistent results are achieved.
7. Discard the solutions of the Erlenmeyer flasks as well as any other solutions down the sink. Rinse all glassware with tap water and set aside to dry.

Observations:

Part A: Making the NaOH Solution:

Actual Mass of KOH used to make 500 mL $NaOH_{(aq)}$	

Part B: Titration to Standardize NaOH:

$KHSO_{4\,(s)}$ titrated by 0.100 mol/L of $NaOH_{(aq)}$				
	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Mass of $KHSO_{4\,(s)}$ used				
Initial Volume of NaOH				
Final Volume of NaOH				
Volume of NaOH added				
Phenolphthalein Colour				

Part C: Titration of H_2SO_4 by NaOH:

10.0 mL of $H_2SO_{4\,(aq)}$ titrated by Standardized $NaOH_{(aq)}$				
	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Initial Volume				
Final Volume				
Volume of NaOH added				
Bromothymol Blue Colour				

Analysis:

Part A: Making the Standardized NaOH Solution:

1. Based on the mass of NaOH used, determine the initial concentration of the NaOH solution.

Part B: Standardizing NaOH Solution using $KHSO_4$:

1. For each of the acceptable trial, determine the standardized concentration of $NaOH_{(aq)}$.
2. Average the results above.

Part C: Acid and Base Titration:

1. Using the standardized NaOH concentration determined in Part B, calculate the experimental concentration of $H_2SO_{4\,(aq)}$ for each acceptable trial.
2. Average the above results.

Evaluation:

Part A: Making the Standardized NaOH Solution:

1. Why is it not necessary to measure out the exact mass of $NaOH_{(s)}$ in the experiment compared to the pre-lab calculation?
2. What is the purpose of dissolving the $NaOH_{(s)}$ in significantly less volume of water in the beaker when we need the final solution volume of 500 mL?
3. Besides using deionized water to make a standardized solution of NaOH, what might be a good way to eliminate the CO_2 in the water?

Part B: Standardized NaOH Solution using $KHSO_4$:

1. How did the initial [NaOH] compare to the standardized [NaOH]? Why was there a difference?
2. What would happen to the calculated [H_2SO_4] if you use the initial [NaOH] instead of the standardized [NaOH]? (Specifically, would the calculated [H_2SO_4] be higher or lower using the initial [NaOH]? Please show your work.)

Part C: Acid and Base Titration:

1. Predict and explain what would happen to the calculated [$H_2SO_{4\,(aq)}$] (would it increase or decrease) when there is/are
   a. distilled water left in the Erlenmeyer flask when $H_2SO_{4\,(aq)}$ is transferred.
   b. distilled water left in the pipet when $H_2SO_{4\,(aq)}$ is transferred to the Erlenmeyer flask.
   c. air bubbles in the pipet when $H_2SO_{4\,(aq)}$ is transferred to the Erlenmeyer flask.
   d. distilled water left in the buret when $NaOH_{(aq)}$ is added.
   e. air bubbles in the buret when $NaOH_{(aq)}$ is added and are dislodged.
2. The approximate theoretical concentration of $H_2SO_{4\,(aq)}$ is 0.0880 M. Compare your calculated [$H_2SO_{4\,(aq)}$] with this theoretical concentration by determining the % error. What are the possible sources of error?
3. Why did we use phenolphthalein in the standardization of NaOH by $KHSO_4$, but we use bromothymol blue for the titration of H_2SO_4 by NaOH? (Hint: Think of the equivalence points of both titrations and the endpoints of the indicators.)

Conclusion:

1. Accounting for the % errors, what would you do to improve the procedures of this lab?
2. Summarize what you have learned from this lab.