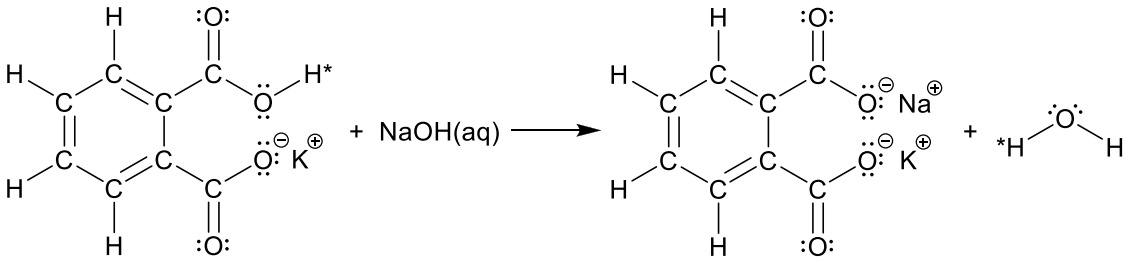
Acid-Base Titrations – Background

Part 1 – Standardization of ~0.1 M NaOH(aq):

In Part 1 of today's experiment, you will prepare an approximately 0.1 M solution of NaOH(aq) by diluting a 6 M solution of NaOH(aq). You will then accurately determine the exact concentration of your ~0.1 M NaOH(aq) solution by a process known as standardization. To "standardize" means to accurately determine the concentration of a solution, so that solution may be used for another measurement. You can think of it as calibrating a solution. Crystalline potassium hydrogen phthalate (abbreviated KHP) will be used as the primary standard acid. By titrating a NaOH solution against a measured mass of KHP, you can accurately determine the concentration of the NaOH solution. Then, it is possible to titrate solutions of acids having unknown concentrations with the NaOH solution (whose concentration has now been determined) to find the respective unknown acid molarities.

Potassium hydrogen phthalate (KHP) is a weak, monoprotic (one acidic H, denoted by H*) organic acid that reacts with aqueous sodium hydroxide according to the reaction:

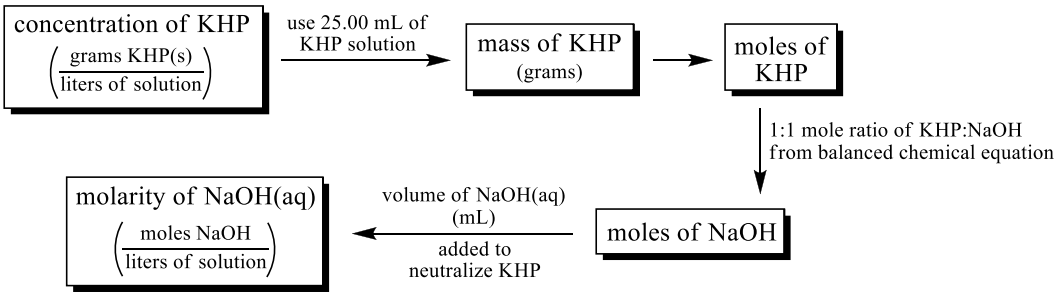


potassium hydrogen phthalate(aq)
KHP
($C_8H_5KO_4$)

In order to detect the equivalence point (the endpoint when the reactants are exactly neutralized), an indicator dye, such as phenolphthalein, is added to the reaction mixture. The endpoints of your titrations will be signaled by the phenolphthalein color change. The indicator, in this case, is sensitive to the relative amount of hydroxide ion in solution which increases quickly once the KHP reactant is used up.

Part 1 – Calculations:

To calculate the exact concentration of your ~0.1 M NaOH(aq) solution, the following logic stream can be used. The stockroom will provide you with a stock solution of KHP(aq), whose concentration is known and labeled on the bottle. Be sure to record this concentration, with proper units, in the data section of your notebook!



Part 2 – Determination of the Unknown Concentration of Sulfuric Acid:

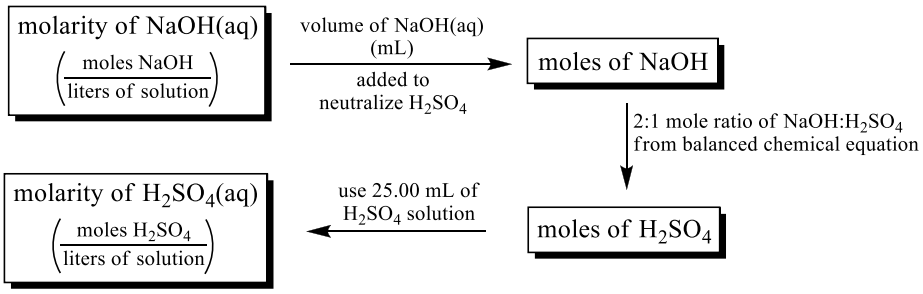
By now, you'll know the exact concentration of your ~0.1 M NaOH(aq). In Part 2 of today's experiment, you'll titrate your ~0.1 M NaOH(aq) solution against a solution of sulfuric acid (H_2SO_4) whose concentration is unknown.

$$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$$

Once again, phenolphthalein will be used to indicate the equivalence point of the titration; the point where enough NaOH(aq) has been added to completely consume the H_2SO_4(aq) and any further addition of NaOH(aq) quickly raises the pH of the solution.

Part 2 – Calculations:

Although the balanced chemical equation is different from Part 1, the logic to determine the unknown H_2SO_4(aq) concentration is nearly the same, albeit in reverse.



Sample Buret Readings Using a Meniscus:

In order to obtain data with good precision, you must develop good techniques with a buret, a specially designed piece of volumetric glassware. Your instructor will cover the proper use of a buret with you. You must read the buret to the proper level of precision (significant figures) as with all volumetric glassware. Below is an example of a section of a buret. Note that each of the three different examples are properly recorded to two decimal places with the last digit estimated with an "educated approximation" (commonly called the "doubtful digit").

initial volume = V_i = 20.24 mL

volume = 20.82 mL

final volume = V_f = 21.40 mL

Understand that the graduations of a buret are backward from a graduated cylinder, and burets are meant to measure the volume *dispensed*. Record the initial volume, record the final volume, and subtract the two values ($V_f - V_i$) to calculate the total volume dispensed.

Acid-Base Titrations – Procedure

Purpose:

In the first part of this experiment you will prepare and standardize a solution of sodium hydroxide to use in this week's experiments and also to use in next week's experiment (so don't throw it away).

Special Supplies – Things to borrow and return on the same day:

- 1 liter polyethylene bottle
- 500 mL polyethylene bottle (return after next week's experiment)
- 25 mL volumetric pipet
- pipet pump or pipet bulb
- buret and brush

The volumetric pipet and buret are fragile and expensive.
Treat them with care and respect.

Procedure:

Clean glassware and proper lab techniques are essential for success in this experiment. Your instructor will provide guidance and suggestions.

Part 1a. ***Preparation of the ~0.1 M NaOH Solution***

Using the 6 M NaOH solution and deionized water available in the lab, prepare one liter of approximately 0.1 M NaOH solution. You will need to calculate the necessary quantities ahead of time and be prepared to show your instructor how you will prepare this solution. Your instructor will go over how to safely handle, pour, and measure the 6 M NaOH(aq). Prepare your approximately 0.1 M NaOH solution in the 1 liter polyethylene bottle which you have pre-rinsed several times with deionized water. Although there are usually no volumetric graduations on the plastic bottle, adding deionized water to the top (before the bottle tapers) will result in ~1 liter of solution. Given that this solution will be standardized in Part 1b, an accurate volume of solution is not critical. Cap the bottle and be certain that this solution is thoroughly mixed.

Rinse the 500 mL polyethylene bottle with deionized water and discard the rinse water. Use approximately 20 mL portions of your ~0.1 M sodium hydroxide solution to rinse the 500 mL polyethylene bottle three times, discarding each NaOH(aq) rinse in the proper waste container. Then, fill this bottle with your ~0.1 M NaOH solution. Put the 500 mL bottle away in your drawer to use in the next experiment.

For today's experiment, use the remaining ~0.1 M NaOH solution from the 1 liter polyethylene bottle. (When you finish today, rinse out the 1 liter bottle and return it so the next class can use it.) At this point you only know that the concentration of the sodium hydroxide solution is approximately 0.1 M. Next, you will determine the exact concentration of the solution by standardization.

Part 1b. ***Standardization of the ~0.1 M NaOH Solution***

Pour 80-85 mL of the KHP(aq) stock solution into a clean, dry, labeled beaker from your drawer. You will pipet the KHP(aq) from this beaker and NOT from the stock bottle. A total of

75 mL will be used for the titrations and you need a little extra to rinse your pipet, so 80-85 mL should suffice. Do not waste the KHP solution and DO NOT pour any excess back into the stock bottle. Excess KHP(aq) should be discarded in the proper waste container.

Use a 25.00 mL volumetric pipet to precisely deliver a 25.00 mL aliquot of the standard KHP solution into a 200 or 250 mL Erlenmeyer flask. (You may need to practice with the pipet and water until you can carry out this operation well.) Then add about 25 mL of deionized water, measured with a 100 mL graduated cylinder, washing down the sides of the flask in the process. The exact volume of H_2O added is not important because the addition of H_2O will not change the moles of KHP present in the flask. Next, add 2 drops of phenolphthalein indicator solution. Don't forget this indicator or the titration will not work and you will spend much longer in the lab than you had anticipated!

Rinse a buret twice with approximately 10 mL of the ~0.1 M NaOH solution and drain the solution through the buret tip into a waste beaker. Then, using a funnel, fill the buret with the ~0.1 M NaOH solution. Make sure there are no air bubbles in the tip of the buret or just above the stopcock. If bubbles are present, open the stopcock, let the ~0.1 M NaOH(aq) run through to push out any bubbles, and then refill the buret. Run base solution (NaOH) out of the buret until the meniscus level is just below the 0.00 mL graduated mark. Pay special attention to where the buret is marked 0.00 mL and where it is marked 50.00 mL as it is typically marked opposite of a graduate cylinder (0.00 mL is at the top). Remove the funnel to prevent clinging solution from dripping down and changing your readings later. Read the bottom of the meniscus to determine the initial volume of the base. If you have trouble viewing the bottom of the meniscus, you may obtain a buret viewing card from the stockroom. Record the initial volume to the nearest 0.01 mL in your notebook. Be sure to keep all of the significant figures when you record the value. You are now ready to titrate!

The most efficient way to do this experiment is to add base from the buret to the KHP solution fairly slowly the first time, swirling the flask and contents, as you add the base. As you approach the endpoint, the area in the KHP solution where the drops of NaOH(aq) fall will turn pink; then the pink color will disappear as the solution becomes mixed. From this point on, add the ~0.1 M NaOH(aq) dropwise, with constant swirling. Occasionally wash down the sides of the flask with a little deionized water from your wash bottle. The endpoint is reached when one drop (or less) of the ~0.1 M NaOH solution causes the solution to become permanently pale pink throughout. Don't worry too much about getting exactly to the one drop, this time. This is your "quick and dirty" trial. Record the final volume, again recording the reading to the nearest 0.01 mL. The difference between the initial and final volumes is the volume of NaOH solution needed to reach the endpoint of the reaction.

Repeat the titration two more times using a clean Erlenmeyer flask each time. After the first titration, the rest should go more quickly since you now have some idea of how much base is required for each aliquot of KHP solution. The base may be added quickly until you are within 2 or 3 mL of the end point. Then, change to slow dropwise addition. Be sure you read the buret by estimating each reading to the nearest 0.01 mL. It is a good idea to record the volumes when you are close to the endpoint so that if you add a little too much you have a value of the volume just before that happened. Be sure to record all of your data, in your table, in your notebook, as you collect it. This data will be used to calculate the exact concentration of your standard sodium hydroxide solution.

Part 2. ***Determine the Concentration of Unknown Sulfuric Acid***

Obtain a sulfuric acid solution of unknown concentration. Pour 80-85 mL of the $H_2SO_4(aq)$ stock solution into a clean, dry, labeled beaker from your drawer. You will pipet the $H_2SO_4(aq)$ from this beaker and NOT from the stock bottle.

You must record the unknown ID number/letter in your notebook!
If you do not record it, your instructor will not know the true value,
so you cannot receive any credit for accuracy. Don't let that happen!

A total of 75 mL will be used for the titrations and you need a little extra to rinse your pipet, so 80-85 mL should suffice. Do not waste the H_2SO_4 solution and DO NOT pour any excess back into the stock bottle. Excess $H_2SO_4(aq)$ should be discarded in the proper waste container.

Pipet a 25.00 mL aliquot of your unknown sulfuric acid solution into a clean 250 mL Erlenmeyer flask. Titrate this $H_2SO_4(aq)$ solution with your standardized NaOH solution, using the same buret. Use phenolphthalein as the indicator, as described previously. Repeat this titration two more times. Record your data as you collect it, with the correct number of significant figures!

Waste Disposal:

- Dispose of excess 6 M NaOH(aq) into the waste container. DO NOT pour excess back into the stock bottle!
- Dispose of all 0.1 M NaOH(aq) rinses (1 liter polyethylene bottle and buret) into the waste container.
- Dispose of excess KHP(aq) and $H_2SO_4(aq)$ into the waste container. DO NOT pour excess back into the stock bottle!
- Dispose of all titrated solutions (KHP + NaOH and H_2SO_4 + NaOH) into the waste container.

Spills and Glassware:

- Use wet paper towels to wipe up small spills.
- Large spills of acid (KHP or H_2SO_4) or base (NaOH) should be immediately reported to your instructor.
- Rinse the pipet and buret and other glassware with deionized water before returning them.

Calculations:

Follow the flow charts presented in the Background, making sure to properly convert units as needed, and account for reaction stoichiometry. Show all units! Watch significant figures!

Conclusion:

Report the average molarity of your NaOH(aq) solution. Is it close to your goal of 0.1 M? Report the average molarity of your $H_2SO_4(aq)$ solution, with its ID number/letter. Comment of the precision of your KHP and H_2SO_4 titrations as shown by their standard deviations. What are possible sources of error that may have, or could affect your precision? In your opinion, do you trust your results/calculations and was the lab successful?