Experiment 20-Acid-Base Titration: Standardization of KOH and Determination of the Molarity and/or Percent Composition of an Acid Solution

In this experiment, you will determine the molarity and percent composition of a weak acid using titration. You will do this by performing a **titration**.[1] A titration is an experimental technique used to determine the amount of material in an **analyte**. An analyte is substance analyzed by the titration process. We use a **titrant** to run the analysis. The titrant is the substance in which we know the amount of moles. For our experiment, the titrant will be a solution of potassium hydrogen phalate (KHP) which is behaving as an ACID. This will be a standard solution. We will use this standard solution to obtain the concentration of a BASE (either KOH or NaOH) solution. We will use the base solution to titrate the vinegar solution. Since we are using a known concentration of base, the technique is also referred to as ***alkalimetry.***

To perform a titration, a carefully measured amount of one reactant is added to an Erlenmeyer flask. An **indicator** is added that will signal the endpoint of the titration by a visible color change. Then the other reactant is added slowly to the flask using a burette. When the indicator changes color, the reaction is complete and the volume is measured. Titration, then, becomes a way for us to count reactant moles.

In this lab, you will start with monoprotic acid. When an equivalent amount of OH^- is added to the reaction vessel, the indicator will change color, and the reaction is considered done. This point, the point at which the indicator has changed color, is called the endpoint. For the indicator to change color, there must be a slight excess of hydroxide ions. The change occurs around the **endpoint**, but **not** at the point at which the moles of acid = the moles of base (the precise equivalence point). If we choose an indicator that changes close to the equivalence point, and stop the reaction at the same color for each reaction, we can be reasonably certain that the equivalence points for all the titrations will be about the same.

When the indicator changes color, the reaction is complete and the volume is measured. If you know the volumes of both solutions used and the concentration of one of the solutions, you can calculate the concentration of the other solution using stoichiometry.

In **Part 1**, you will make a *standard* solution of potassium hydrogen phthalate. (The chemical formula of potassium hydrogen phthalate is $KHC_8H_4O_4$; it is often abbreviated as "KHP".) The concentration of a standard solution is known very precisely. Since KHP is available in very pure form, you can make a standard solution by weighing a precise amount of KHP and dissolving it in water to make a precise volume of solution. You will calculate the exact concentration of your KHP solution to four significant figures.

In **Part 2**, you determine the precise concentration of a potassium hydroxide solution by titration. You might be wondering why you cannot prepare this solution by simply weighing out some KOH and dissolving it in water. This is because KOH is not available in very pure form—it absorbs water from the air. To determine the precise concentration of a KOH solution, you must

[1] Sandell, E. B. & West, T. S., Recommended Nomenclature for Titrimetric Analysis, London Butterworths, A*nalytical Chemistry Division, Commission on Analytical Nomenclature, 429 (unknown date)*

titrate it with another standard solution, such as KHP. You will use the KHP standard solution that you made in Part 1 in a series of titrations with potassium hydroxide (KOH).

The reaction is as follows:

The molecular equation: $KOH_{(aq)} + KHC_8H_4O_{4\,(aq)} \rightarrow H_2O_{(l)} + K_2C_8H_4O_{4\,(aq)}$

The net ionic equation:: $OH^- + HC_8H_4O_4^- \rightarrow H_2O_{(l)} + C_8H_4O_4^{2-}$

From the results of your titrations, you will be able to determine the precise concentration of the KOH solution. This process is called "**standardization**" of the KOH solution.

Once you have calculated the concentration of the KOH, you will use your KOH solution in another series of titrations with a weak acid of unknown concentration. This weak acid will be vinegar, which contains acetic acid, $HC_2H_3O_2$.

You will use the standardized KOH to titrate vinegar. Vinegar is a complex mixture that contains acetic acid as its acidic component. The equation for the reaction in Part 4 is as follows:

$$HC_2H_3O_{2\,(aq)} + KOH_{(aq)} \rightarrow H_2O_{(l)} + KC_2H_3O_{2\,(aq)}$$

You will use the titration data to calculate the mass percent acetic acid in the vinegar and the molarity of acetic acid in the vinegar.

For this experiment, you will be graded on the accuracy of your results: how close you come to determining the actual concentration of the unknown weak acid. Proper laboratory technique is therefore essential.

General Procedural Notes:

In this lab, as in any precise titration, you must be very careful not to alter the concentration of the solutions in any way before their volumes have been measured. Any glassware that will be used the solutions must be either absolutely clean and dry or rinsed with 3 small portions of the solution to be used in it (being sure to wet the entire inner surface of the glassware each time). If you accidentally use a piece of glassware that is wet, the small amount of water on the inside of the glassware will dilute the solution slightly and alter its concentration. If this happens, you will need to start over.

After the volumes of the solutions have been measured precisely (once they reach the reaction flask), it is perfectly fine to dilute the mixture with deionized water. (Once they are mixed, it's the number of reactant molecules present that matters. Adding water cannot change the number of reactant molecules.)

You will be working in pairs (three if you must) or singly. (Your laboratory instructor will tell you if it is acceptable to work together.) No groups of more than three students are allowed. If you work with partners, each person in the group will do two titrations such that there is agreement among the samples. If you work alone, you need three trials that agree for each part. Average the best three results from the titrations. If you need to do additional trials, include all the data. Make sure to clearly indicate which trials are being averaged and which are being thrown out. Your three best results must agree within 1.5 % of each other. If they don't, you will need to do additional titrations.

Safety Precautions:

- Wear your safety goggles.
- If any acid or base solution splashes on you, rinse it off immediately.

Waste Disposal:

- Waste from this experiment may be safely discarded down the drain using plenty of running water.

Prelab Questions and Calculations: See pre-lab handout for questions

Procedure

When you have standardized the KOH (when you have finished part 2 and have three trials which agree), turn in your small flask to the instructor to be filled with unknown KHP solution. It must be absolutely dry. (If it's not, its concentration will be slightly altered by the water drops.) Make sure to write down the unknown number.

Part 1: Preparation of Standard KHP Solution

You will need to make 250 mL of approximately 0.1 M KHP. Clean a 250 mL volumetric flask, and rinse it several times with deionized water. Make sure to wet the inner walls of the flask completely each time.

Note: all masses must be measured to ± 0.001 g or ± 0.0001 g. Make sure to use a balance that has glass doors. Go to the balance with your volumetric flask, a clean funnel with a **wide neck** (you will probably need to borrow one from the stockroom-most of the funnels in the lab drawers have narrow necks), and a small vial of KHP.

Put the funnel into the neck of the volumetric flask. Weigh the vial of KHP, tare the balance, then gently tap out some KHP from the vial into the funnel. You need to transfer the approximate mass needed into the funnel, so you may need to check a few times to see how much you have added. Each time you weigh the vial, the mass will register as a negative value on the balance. For this mass to be accurate, you must not lose any KHP during the transfer! *Do not use a scoopula or a spatula or any implements at all to transfer the solid!* Also, take care not to let any of the solid spill at any time between the weightings. It is essential that all of the KHP that leaves the vial goes into the flask. If you spill any of the solid, you must start over.

Note: you do not need to use the exact mass of KHP that you calculated: any mass that is within 0.5 g of what you calculated will be fine. The important thing is that you know exactly how much was used. (Use all digits that the electronic balance gives you.)

Rinse the KHP in the funnel into the flask with a jet of deionized water from a wash bottle. When the funnel has been rinsed thoroughly and all the KHP is in the flask, remove the funnel and add more deionized water to the volumetric flask. When you have added about three-quarters of the water you need, swirl the flask for a few minutes to dissolve the solid. Make sure no solution splashes out of the flask. If it does, you will need to start over. When the solid has dissolved, add more water, rinsing down the inner walls of the flask. Add water up to the mark on the neck of the flask. The bottom of the meniscus must be precisely on the line.

IMPORTANT: The most common mistake is accidentally adding just a little too much water. If you do this, there is no way to correct for it, and you must start over. To insure that this doesn't

happen, when you get within about an inch from the mark on the neck of the flask, start using a clean dropper to add the water, and be very careful.

Once the volume of the solution reaches the mark, stopper the flask securely and then mix the solution continuously for 5 minutes. (Invert and swirl, invert and swirl, etc.) Make sure to keep this solution stoppered when not in use. Transfer this solution to a 250mL Florence flask.

Calculations for Part 1

From the mass of KHP used and the volume of the volumetric flask, calculate the molarity of the standard KHP solution. The volume of the volumetric flask is 250.0 mL. Calculate the molarity of the solution to 4 significant figures.

Part 2: Standardization of KOH

You are using two burettes for this part of the lab. One burette will hold the KHP solution from part 1; the other will hold the KOH solution (analyte).

The KOH solution (the analyte for this titration): Collect the KOH solution in a 500 mL Florence Flask. Check that the stopper fits the flask BEFORE you add the KOH solution for storage. Rinse 500-mL Florance flask with UK KOH solution using 5 mL aliquots. Collect about 350 mL of KOH in a clean 500 mL flask.

Stopper the flask well, and shake it continuously for a few minutes. Label it (Example: "0.1 M KOH"). **Important**: make sure to collect enough KOH solution to last for the entire experiment, and make sure NOT to refill this KOH solution. This is to be sure that this KOH solution has the same concentration throughout the experiment. After the first day, the refill bottle of KOH might have been prepared in a different batch, and thus it will have a different concentration, and you will have to start all over.

Preparing for the titration

You will need: a ring stand, a working burette clamp, and two burettes, and two funnels, 4-5 Erlenmeyer flasks (there are extras under the hood-please return them at the end of the lab day), and a water bottle filled with distilled water. The titration technique we will use in this part of the experiment is often called '**back titration**'. In a back titration, we add extra titrant to the analyte until the indicator registers pink, then add analyte until the indicator returns to clear. We continue until the process reaches a steady state, where one drop of acid for example turns the pink solution clear, and one drop of base turns the clear solution pink.

Procedure:

Label burette 1 with acid (titrant), and burette 2 with base (analyte). Rinse each burette with appropriate solution three times. Clip them to the burette clamp. Fill each burette with the appropriate solution up to around zero mark and read and record the initial volumes of acid and base to the nearest ± 0.01mL.

Transferring 15-25 mL of of standardized KHP solution to a flask from acid burette. into a 250 mL Erlenmeyer flask. (It's fine if this flask is wet, as long as it's wet with deionized water.) Add 1-2 drops of phenolphthalein indicator to the flask with acid.

Titrate the KHP solution by adding KOH from the base burette until the solution turns light pink. Swirl the flask and periodically wash down the inner walls of the flask with a jet of water wash bottle.

Switch to the KHP burette and slowly add acid drop by drop, until the solution turns clear. Switch to the base burette and slowly add base drop by drop, until the solution turns light pink. You want to reach a steady state of the drops of acid to turn clear and the amount of base to turn pink. The endpoint is reached when one drop of base added changes the solution from colorless to a light pink that persists for 20 seconds or more and 1 drop of acid changes back to clear.

Stop with the addition of base and read the FINAL volume of acid and the FINAL volume of base add Record the final reading(s) of the burette(s) to the nearest ± 0.01 mL. After each titration, dump the mixture in the flask down the sink. Rinse and use the flask again without drying it (just shake out the excess water). Do 4-6 titrations, and then calculate the molarity of KOH for each titration to four significant figures. A white paper under the reaction flask is helpful for detecting the faint pink color. Your data will include initial, final, and net volumes from the burette readings.

- **<u>DO NOT DISCARD THE KOH!</u> YOU WILL NEED IT FOR PART 3.**

Store it in locker, stoppered tightly, labeled with its identity and its concentration.

<u>MODIFIED CALCULATIONS:</u>

The **MODIFIED** calculation to determine if you need more trials: calculate the ratio of the acid volume to the base volume for each trial and the average of the ratios. We can make this approximation because the molarity of the KHP is constant. Since we are using the formula $M_{KOH}V_{KOH}=M_{KHP}V_{KHP}$, the M_{KOH} is proportional to $V_{KHP} \div V_{KOH}$. Once you have the ratios, calculate the % difference. If the difference is less than < 1.5%, move on to Part 3. If the difference is > 1.5%, do more titrations such that at least trials are <1.5%. This calculation can be done in your book in the DATA & OBSERVATION section, or in a designated section after the procedure for PART 2.

In the **Calculation Section** of the report: Calculate the Molarity of the KOH using data from the trials you kept for your % difference calculations. Calculate the average molarity of the KOH solution.

<u>Part 3: Mass Percent and Molarity of Acetic Acid in Vinegars</u>

In Part 3, you will determine the precise acetic acid content in a specific brand and type of vinegar by titration with your standardized KOH. For this titration, you will use a single burette technique. The vinegar will be in a burette at the VINEGAR station.

The equation for the reaction of acetic acid with KOH is:

$$\underset{\text{acetic acid}}{HC_2H_3O_{2\,(aq)}} + \underset{\text{potassium hydroxide}}{KOH_{(aq)}} \longrightarrow \underset{\text{potassium acetate}}{KC_2H_3O_{2\,(aq)}} + \underset{\text{water}}{H_2O_{(l)}}$$

Weigh a clean Erlenmeyer flask on an analytical balance to a precision of at least ± 0.001 g. Go to the VINEGAR station, and add about 2 mL of vinegar (measured with a pipet or burette and recorded to ± 0.01 mL) to the flask, and weigh it again. Record the mass of the flask with the vinegar. Subtract to determine the mass of vinegar used. Add a few drops of phenolphthalein indicator into the flask with the vinegar.

Use your standardized KOH solution to titrate the vinegar. Most titrations at the concentration of KOH that we are using, require about 20 mL of base. You may add the first 15 mL of base quickly,

but slow down as you approach the endpoint so that you are eventually adding base one drop at a time to the flask. Swirl the flask well to mix during the titration, and periodically wash down the inner walls of the flask with a jet of water from your wash bottle. (One indication that you are near the endpoint is that the pink color of the indicator will persist for a longer time when you are swirling the flask.) You have reached the endpoint when one drop of base added changes the solution from colorless to a light pink that persists for 20 seconds or more. A white piece of paper under the flask is helpful for detecting the faint pink color. Be sure to record the initial burette reading, as well as the burette reading at the endpoint, to two decimal places.

After the first titration, dump the contents of the flask down the sink, rinse the flask well with deionized water, shake it dry, and carefully dry off the outside of the flask with a paper towel. Weigh the flask again, and continue with the rest of the procedure for subsequent titrations. (The inside of the flask does not have to be dry, but it should be dry on the outside. You will need to weigh it before each trial, because it will contain a slightly different amount of water each time.)

Calculations Section: **NOTE: Modification Calculations do not belong here.**

Part 1:

Calculate the molarity of the standard KHP solution.

Part 2:

- Calculate the volume of base used for each trial, the moles of base for each titration trial using the standard molarity from Part 1, and the molarity of the KOH solution.
- Calculate the average, and the % difference<1.5% for the molarity of the KOH.

Part 3:

- Calculate the mass of vinegar used for each trial, the volume of base used for each trial, the moles of base for each titration trial using the average molarity from Part 2, the moles of acetic acid in vinegar, the mass of acetic acid in vinegar.
- Use the mass of the solution and the mass of acetic acid in vinegar to calculate the mass percentage of acetic acid in vinegar for each trial.
- Calculate the molarity of acetic acid for each trial using the volume of vinegar used and the moles of acetic acid.
- Calculate the average molarity of the vinegar solution, calculate the average % by mass of acetic acid.
- Calculate the % difference <1.5% using the mass percentage.
- Calculate the % error using the data from the bottle (5%, white vinegar).
- Present the average using the three best data

Percent Difference & Error Formulae:

$$\frac{Highest\ value - Lowest\ value\ in\ data\ set}{average\ of\ the\ data\ set} x100 = \%\ difference$$

$$\frac{Accepted\ value - Best\ average}{Accepted\ value} x100 = \%\ error$$

Questions: SEE PRE-LAB FOR POST LAB QUESTIONS