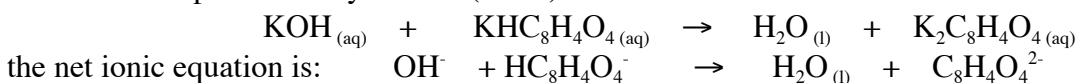


Experiment 20 - Acid-Base Titration: Standardization of KOH and Determination of an Acid Solution

In this experiment, you will determine the precise concentration of a weak acid solution that has an unknown molarity. You will do this by performing a series of titrations. A titration is an experimental technique for determining the concentration of a solution by reaction with something else. 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 buret. 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 $\text{KHC}_8\text{H}_4\text{O}_4$; 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.

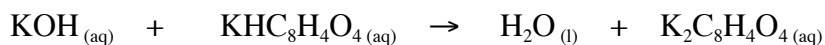
In Part 2, you determine the precise concentration of a potassium hydroxide solution by titration. You might be wondering why you cannot prepare this solution by simply weighing out some KOH and dissolving it in water. This is because KOH is not available in very pure form—it absorbs water from the air.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



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.

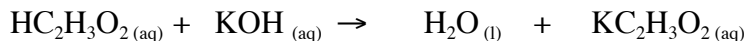
Once you have calculated the concentration of the KOH, you will use your KOH solution in another series of titrations with a weak acid of unknown concentration. This weak acid will either be another KHP solution (Part 3) or vinegar, which contains acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$ (Part 4). Your instructor will tell you whether the class will do Part 3 or Part 4.

If you are doing Part 3, you will use the standardized KOH to titrate a solution of KHP that has an unknown concentration. The reaction for this set of titrations is the same as it was for part 2:



From the titration data, you will calculate the molarity of the unknown KHP solution to 4 significant figures.

If you are doing Part 4, you will use the standardized KOH to titrate two different types of 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:



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 comes into contact with 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 or singly. (Your laboratory instructor will tell you if it is acceptable to work together.) No groups of three or more are allowed. If you work in a pair, for each part of the experiment, you will each do two titrations (a total of four for each part). 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 of 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:

(Remember, ALWAYS show your work and explain your reasoning.)

1. Calculate the approximate mass of KHP you will need to weigh out to make 250 mL of 0.10 M KHP solution. The formula of KHP is $\text{KHC}_8\text{H}_4\text{O}_4$.
2. In Part 1, when you are making the solution of KHP, why can't you use a spatula or scoopula to transfer the KHP to the flask?
3. In Part 1, when you are adding water to the KHP, why must you start over if you add a little too much water?

Procedure

Rinse out a 125 mL flask with deionized water and leave it open in your locker so that it has time to dry completely. You will be collecting your unknown in this flask for part 3 of the experiment. 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, 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. When you have added the approximate mass needed, weigh the vial again. The difference in the two weights is the mass of KHP that was transferred to the flask. In order 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 two weighings. 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.)

Data needed for this part is as follows:

mass of KHP vial

mass of KHP vial after transfer

mass of KHP transferred.

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 of 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.

Calculations for Part 1

From the mass of KHP actually 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

Collect about 350 mL of KOH in a clean 500 mL flask. (It's OK if this flask is wet with a little deionized water.) Stopper it 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 in order to be sure that this KOH solution has exactly 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.

You may use either one or two burets for the titrations in this lab. It is preferable to use two burets, but if there is a shortage of burets, some people will need to use one buret and one 25-mL pipet. If you use two burets, the KHP will go in one buret and the KOH in the other buret. *Follow the directions for "Titration with Two Burets" (see the appendix).* Start with about 20-25 mL of the acid in the titration. Your data will include

initial, final, and net volumes from the buret readings for both burets. **All buret readings must be to the nearest ± 0.01 mL.**

If you are using one buret for the titrations, you will need to use a pipet to measure the volume of KHP used. Pipet 25.00 mL of standardized KHP solution into a 250 mL Erlenmeyer flask. (It's fine if this flask is wet, as long as it's wet with deionized water.) Be sure to use proper pipetting technique, because your results depend on it. *Follow the directions for "Titration with One Buret" (see the appendix).*

After transferring 25 mL of carefully measured KHP solution to a flask from either a buret or a pipet, add 2-3 drops of phenolphthalein indicator. Titrate the KHP solution by adding KOH from a buret. Record the initial volume reading to the nearest ± 0.01 mL. You may add the first 15 mL 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. Record the final reading(s) of the buret(s) to the nearest ± 0.01 mL.

After each titration, you can dump the mixture in the flask down the sink, rinse the flask well with deionized water, and use the flask again without drying it (just shake out the excess water). Do 3 or 4 titrations, and then calculate the molarity of KOH for each titration to four significant figures. Calculate the percent difference between the highest and lowest of the molarities, and if the difference is less than 1.5%, you may go on to the next part of the lab. If the percent difference is more than 1.5%, you need to do more titrations until you have three trials that agree within 1.5%.

Average the best three results. **DO NOT DISCARD THE KOH!** Store it in your locker, stoppered tightly, labeled with its identity and its concentration (for example, "KOH 0.1248 M"). It is absolutely essential that you use the same KOH solution for part 3 or 4 that you standardized in part 2. The whole point of part 2 is to determine the concentration of the KOH to four significant figures, so you can use it to determine the concentration of an unknown to four significant figures.

Calculations for Part 2

For each trial, calculate the molarity of KOH from the data. To do this, start with from the known volume and the calculated molarity of the KHP you made, and calculate the moles of KHP used. Use the balanced equation to relate moles of KHP to moles of KOH used. From the moles of KOH and the volume of KOH used, find the molarity of KOH. Do this calculation separately for each trial. Choose the three trials whose results have the closest agreement, and calculate the percent difference in the molarity between the three trials. If the percent difference is less than 1.5 %, you can go on to part 3 or 4. If not, you need to do more titrations until you do have three trials that agree (but you need

to make sure to save enough KOH to do part 3 or 4 - you cannot refill it from the bottle! Why not?). When you have three results that agree, take the average and use it for the rest of the lab calculations.

Important note: do either Part 3 or Part 4, but not both. Ask your instructor whether you are assigned Part 3 or Part 4.

Part 3: Determination of an Unknown KHP Solution

Give a clean, absolutely dry flask with your name(s) on it to the laboratory instructor to be filled with an unknown KHP solution. Write down the unknown number. Follow the procedure for part 2, but this time, use unknown KHP instead of the standardized KHP. Be sure you use the same KOH solution you used from part 2. (The KHP is pipetted, and the KOH goes in the buret - or the unknown KHP goes in one buret, and the KOH goes in the other buret). You will use the average molarity of KOH from part 2 to calculate the molarity of the unknown KHP for each titration. Again, do four separate titrations, and average the three best results. Calculate the percent difference of the three best results. Your results must agree within 1.5 %. If they do not, you must do more titrations until you have three trials that agree.

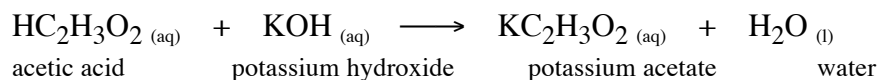
Calculations for Part 3

For each trial, calculate the molarity of the unknown weak acid solution from the data. From the known volume and the average molarity of the KOH, calculate the moles of KOH used. Use the balanced equation to relate moles of KOH to moles of KHP used. From the moles of KHP and the volume of KHP used, find the molarity of the unknown KHP. Again, you need to do this calculation separately for each trial, and the three best trials must agree within 1.5 % - if they don't, you need to do some more titrations until you have three which agree. Calculate the average molarity of your unknown KHP.

Part 4: Mass Percent of Acetic Acid in Vinegars

In Part 4, you determine the precise acetic acid content in a specific brand and type of vinegar by titration with your standardized KOH.

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



Procedure: Your instructor will assign one or two types of vinegar to you. This part will require *at least three titrations for each type of vinegar*, as you will need three results for each vinegar that do not differ by more than 1.5%. For each titration, do the following:

- Weigh a clean, dry Erlenmeyer flask on an analytical balance to a precision of at least ± 0.001 g.

- Add about 2 mL of vinegar (measured with a pipet 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. If the vinegar has a dark color, add more purified water to lighten it.
- Use your standardized KOH solution to titrate the vinegar. Be sure to record the initial buret reading, as well as the buret 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 flask does not have to be absolutely 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 for Part 4

Calculate the mass percent of acetic acid in the vinegar and the molarity of acetic acid in the vinegar to four significant figures. In your calculations, you will need to use the precise concentration of your KOH solution from Part 2. Repeat the procedure until you have three results for each kind of vinegar that do not differ by more than 1.5%. Average the best three results for each kind of vinegar.

Percent Difference Formula:

$$\%difference = \frac{M_{high} - M_{low}}{M_{average}} \times 100$$

In your lab report:

Make sure to include all raw data (actual buret readings) in the Data section. Simple calculations may also be included, such as additions or subtractions. For example, volume delivered or mass of KHP transferred may be included in the data section. Other calculations are done in the Calculations section. Calculations are not data.

Make sure to re-state the calculated molarity of KOH, the unknown number, and the unknown result (molarity or mass percent) in the Summary of Results / Conclusions section. Also include the percent difference for each set of titrations.

In the Evaluation of Results section, discuss possible errors in the lab: errors that you know or suspect you made, errors that may have been likely for anyone doing the lab, and systematic errors inherent in the experiment. ("Human error" is NOT an

acceptable response! You must be specific.) Discuss how each error would affect your results. (Would it make the results too high or too low? Why?)

Questions:

(Remember, ALWAYS show your work and explain your reasoning.)

1. In Part 1, if you used a scoopula to transfer the KHP and some solid stuck to it, how would it affect your result for M_{KHP} ? (Would the calculated (wrong) M_{KHP} be higher or lower than the actual M_{KHP} ?)
2. In Part 1, if you added a little too much water to the volumetric flask, would the calculated M_{KHP} be higher or lower than the actual M_{KHP} ?
3. In Part 2, if there was an air bubble in the tip of the KOH buret that came out during the titration, how would M_{KOH} be affected? (Would the calculated M_{KOH} be higher or lower than the actual M_{KOH} ?)
4. Why is it acceptable to add water to the titration flask?
5. Why can't you add water to either solution at any time before they are transferred to the titration flask?
6. If 25.00 mL of a sulfuric acid solution (H_2SO_4) is titrated with sodium hydroxide, and if it requires 35.88 mL of 0.1127 M NaOH to reach the equivalence point, what is the molarity of the sulfuric acid solution?
7. A sample of 0.495 grams of solid KHP is weighed into an Erlenmeyer flask. This sample is titrated with a sodium hydroxide solution, and 28.56 mL of NaOH are required to reach the endpoint. The sodium hydroxide solution is then used to titrate a sample of phosphoric acid of unknown concentration. It requires 29.88 mL of NaOH to react with 10.33 mL of H_3PO_4 solution. What is the concentration of the phosphoric acid?
8. A 2.353-g sample of vinegar was titrated with 0.08751 M NaOH, and it requires 22.31 mL of NaOH to reach the endpoint. Calculate the mass percent of acetic acid ($\text{HC}_2\text{H}_3\text{O}_2$) in the vinegar sample.