Experiment 11 - Acid-Base Titration

Introduction

A **titration** is an experimental technique for determining the **molarity** of a solution by reaction with something else. Recall that molarity of a solution is defined as moles of solute per liter of solution, so a 1 M ("one molar") solution has 1 mole of solute in 1 liter of solution.

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. At this point, you assume that you have added exactly the right number of moles of the second reactant to completely react with the first (the mole ratio used exactly matches the ratio in the balanced equation). Since you know how much of the first you started with, and how much of the second you added (using the buret), you can calculate the concentration of the unknown solution using stoichiometry.

Titrations are often performed to measure concentration of acidic or basic solutions. Acid-base titrations are convenient because there are many appropriate indicators. In this lab, we will use **phenolphthalein** as our indicator. Phenolphthalein is clear (colorless) in acidic solution, but pink in basic solution. In each titration, we will put the acidic solution in Erlenmeyer flask, and titrate in the basic solution from the buret. The solution in the flask will start clear, and turn pink when the reaction is complete.

The ultimate goal of this lab is the measure the concentration of acetic acid in vinegar. However, we will do this in several steps over 2 weeks. In Part 1, we will make a **standard solution** of an acid called potassium hydrogen phthalate, abbreviated KHP. (It is ***not*** potassium hydrogen phosphide—this doesn't charge balance correctly because phosphide ions are 3-.) The chemical formula of potassium hydrogen phthalate is $KHC_8H_4O_4$. This is a solid weak acid that can be measured conveniently and exactly and is available very pure, allowing you to make a solution of precisely-known concentration (to 4 significant figures).

In Part 2, you will use your KHP standard solution to determine the precise concentration of a NaOH solution. The reason we can't simply prepare the sodium hydroxide solution with a concentration known to 4 significant figures is that NaOH is hygroscopic, meaning it absorbs water from the air. Thus, it won't be perfectly pure, so we can't weigh it out and know exactly how much we have. For this reason, we find the NaOH concentration by titrating with the standard solution of KHP. This process is called "standardization" of the NaOH solution. Once you know the concentration of the NaOH solution, you can use it to titrate vinegar, and find the precise concentration of the vinegar.

The reaction between NaOH and KHP is

$$NaOH(aq) + KHC_8H_4O_4(aq) \rightarrow H_2O(l) + NaKC_8H_4O_4(aq)$$

and the reaction between NaOH and vinegar is

$$NaOH(aq) + CH_3COOH(aq) \rightarrow H_2O(l) + CH_3COONa(aq)$$

As you may have noticed, this experiment emphasizes accuracy and precision. Unlike many experiments we do, with titrations it is possible to be extremely accurate and precise, because there are no major limitations in the equipment or the method. Your goal is to achieve the maximum accuracy and minimum uncertainty you can. Since we want our final answer to have 4

significant figures, we care about the difference between, for example, 0.1001 and 0.1008 M. Because this difference is quite small, mistakes that may seem small can have an important effect! For this reason, you have to think carefully about what you are doing, such as when it is acceptable to use wet glassware or add DI water, and when any additional water would destroy your accuracy.

Chemists often use square brackets around a formula to indicate the molarity of that substance. For example, [NaOH] means the molarity of NaOH. So, for example, the KHP solution you prepare should have [KHP] = 0.1 M.

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.

Procedure

You can work in pairs for Part 1, but you must work by yourself for Part 2 and Part 3.

Part 1: Preparation of Standard KHP Solution

In Part 1, you will prepare exactly 250 mL of a solution that is approximately 0.1 M in KHP (the standard acid). The solution doesn't have to be exactly 0.1 M, but you do need to know precisely what the concentration actually is (to at least 4 significant figures). To know the concentration precisely, you will need to know precisely how much KHP you used, and precisely how much solution you made. This means that all the KHP you weigh out must go into the solution, and you must know the total volume of the solution exactly, which you do by using a volumetric flask and filling it exactly to the line.

1. Wash a 250 mL volumetric flask with DI water (it should have a single line on the narrow neck—check if you aren't sure what a volumetric flask is). While you wash it, practice filling it exactly to the line on the neck. Use a squirt bottle with DI water or a dropper to get the meniscus exactly on the line. It's worth practicing because when you make your solution for real, if you add water over the line, you'll have to start over.
2. Collect the required mass (within 0.5 g of what you calculated on the pre-lab) of KHP in a clean, dry weigh boat and record the mass to at least 3 decimal places. (Be sure to tare the weigh boat on the balance before you put the KHP in, so you don't include the mass of the weigh boat.)
3. Very carefully, pour the KHP into your volumetric flask. You can use a clean funnel (rinsed with DI water). Use a squirt bottle with DI water to rinse all the KHP off the weigh boat and funnel and into the flask. Also wash the KHP down the neck of the flask into the bottom of the flask. Make sure you don't spill any KHP or leave any in the weigh boat. If you spill any, you need to wash the flask and start over.
4. Add DI water until the round part of the flask is half full, then swirl vigorously to dissolve the KHP completely. This might take a few minutes, and it's much easier if there is still

plenty of space in the flask. Make sure you don't spill any of the solution! If you do, you need to start over.
5. Add more DI water until the round part of the flask is mostly full, rinsing the sides of the flask and the neck as you add it. Mix again.
6. Add DI water carefully until it gets close to the line on the neck. Then add DI water ***very carefully*** (use a dropper) until the meniscus of the solution is exactly on the line. ***Do not go over*** the line; if you do you need to start over.
7. Use a stopper with parafilm to cover the flask. Invert and swirl the flask continuously for 5 minutes to thoroughly mix the solution. Make sure you don't spill any of the solution before it's fully mixed! If you do, you need to start over.
8. Keep the solution stoppered when you aren't using it.
9. Using the mass of KHP you actually used, calculate the concentration of your solution to 4 significant figures. The final volume (if you got it exactly on the line) is 0.2500 L. (This means you'll need to use at least 5 sig figs in the atomic weights when you find the molecular weight of KHP. Make sure you keep at least 5 sig figs in all the middle steps of the calculation.)

Part 2: Standardization of NaOH

For this part, your job is to measure the concentration of the NaOH to 4 significant figures. To do this, you will titrate the NaOH with your standard KHP solution at least 3 times, until your three best results have an average deviation less than 0.003 M. If they don't, you will need to do additional titrations. Save all the data—don't cross out trials that deviate more, even if you choose not to include them in your averages.

1. Collect about 250 mL of the NaOH solution (roughly 0.1 M) which is provided in a clean 500 mL flask. It's ok if the flask is wet with DI water from cleaning. Mix it thoroughly and stopper it. Label it with your name, the approximate concentration ("0.1 M NaOH") and the number on the bottle (record this in your notebook also). Don't refill this bottle later.
2. Get a small (25 mL) buret. Rinse it with DI water several times, letting the water run out the bottom and rinsing the sides. Then rinse it **3 times** with your KHP solution. Each rinse, use about 5 mL. Roll the buret so the sides are completely rinsed, then let the solution run out the bottom of the buret. This is called "**conditioning the buret**". This ensures that your solution isn't diluted or contaminated by anything when you put it in the buret, so the concentration doesn't change.
3. Once the buret is conditioned, fill it (no higher than the markings) with your KHP solution. You can share the KHP buret with your lab partner (who also shares your KHP solution). Drain 1-2 mL of the solution out the bottom of the buret to make sure there are no air bubbles. (Use a buret clamp to hold it up.)
4. Get another 50 mL (preferably) or 25 mL buret. Condition it again, this time using first water, then your NaOH solution. Fill it with NaOH and drain 1-2 mL out the bottom. You will probably want to have your own NaOH buret separate from your partner.
5. Separately from your partner, get a clean Erlenmeyer flask (100 or 250 mL is fine). You can rinse it with DI water and it's ok if it is wet.
6. In a table in your notebook, record the initial reading of the buret to 2 decimal places (such as 1.24 mL). Then drain 10-15 mL of the KHP into the Erlenmeyer flask. Touch the tip of

the buret to the side of the flask to get the last drop in. Record the final buret reading in the table in your notebook (always to 2 decimal places).

7. Add 2-3 drops of phenolphthalein indicator to your flask. Swirl to mix.
8. Record the initial reading on your NaOH buret. Drain the NaOH into your flask, swirling constantly. You can rinse the sides of the flask sometimes with DI water. Add the NaOH quickly at first, then slower when you expect you are getting close to the end point.
9. When the solution in the flask stays very slightly pink after swirling, you have reached the end point. Record the final volume on the NaOH buret.
10. Rinse the flask and repeat the titration (steps 6-9) at least twice. For each titration, you need to record the initial and final reading on each buret. This is the data you will use for your calculations. Try to get the color of the solution at the endpoint as light as you can (add the NaOH dropwise, swirling in between each drop, when you get close to the endpoint).
11. Do your calculations and check with your instructor. If your trials are not close enough together, you will have to do more trials.
12. Once your instructor approves your results, close the NaOH solution securely and label it with your name and the precise average concentration you calculated. Store it as directed for next week; you will use this same solution for Part 3.
13. Dispose of the KHP solution in the waste bottle in the hood.

Calculations for Part 2

1. For each trial (titration), calculate the concentration of the NaOH to 4 significant figures. (Find the moles of KHP in the flask at the beginning, and assume you added the same number of moles of NaOH.)
2. Calculate the average of your trials and the average deviation. If the average deviation is greater than 0.003 M, do more titrations and be very careful of your technique.

Part 3: 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 NaOH. As in Part 2, you will do at least 3 titrations, until the results do not differ by more than 1.5 %.

Commented [JS2]: Seems excessive. Perhaps 3% relative average deviation would be more achievable considering the relative uncertainty of the vinegar volume is close to 2%.

1. Collect a small amount of vinegar (each person will need about 15 mL for titration and each buret will need about 15 mL for conditioning). Condition a small (20 mL) buret with the vinegar you are using. (See the instructions for Part 2 to remind you how to condition the buret.) You can share the vinegar buret with a partner. Make sure to use the same brand of vinegar for the whole thing.
2. Condition a large buret (if available) with your NaOH solution from last week.
3. Weigh a clean, dry Erlenmeyer flask (100 mL is fine) on an analytical balance to a precision of at least ± 0.001 g and record the mass in your notebook.
4. Record the initial reading of the vinegar buret in your data table. Add about 2.5 mL of vinegar to the flask. Record the final reading of the buret. Weigh the flask again and record the mass of the flask with the vinegar. Subtract to determine the mass of vinegar used.
5. 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.

6. Use your standardized NaOH 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. (Review the titration instructions in Part 2 if necessary.)
7. 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.
8. Repeat the procedure from step 3-7 at least twice. (Mass the flask, add vinegar, re-weigh the flask, and titrate. 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.)
9. Calculate the concentration of the vinegar and check the results with your instructor. If necessary, repeat the titrations until your results are close enough together.
10. Once your instructor approves your results, clean up. The waste can go down the sink.

Calculations for Part 3

1. Calculate the concentration of the vinegar separately for each trial, as you did for Part 2, using the precise concentration you found for the NaOH.
2. Find the % difference between your least similar trials (of 3 trials total). If it is not less than 1.5%, do more trials until you have 3 within 1.5% of each other.
3. Find the average concentration (in molarity) and the average deviation of the vinegar.
4. Find the density of the vinegar from each trial. Calculate the average density and average deviation.
5. Using the average concentration and the average density, find the mass % of acetic acid in the vinegar.
6. Record the density, molarity and mass % with their error ranges (as described in the lab notebook guidelines).

Name: Section:

Experiment 11 Pre-Lab Sheet: First Day

1. (2 pts) Find the mass of KHP (formula $KHC_8H_4O_4$) needed to prepare 250 mL of 0.10 M solution. Show your work in detail, with all the units.

2. (1 pt) Why must you start over if you add too much water to the volumetric flask in Part 1?

3. (1 pt) How many moles of KHP are there in 10 mL of 0.10 M solution? Show your work in detail, with all the units.

4. (1 pt) Write the equation for the reaction that occurs during the titration in Part 2.

5. (3 pts) Explain *in detail* how to calculate the molarity of a solution using titration data. Show that you understand the purpose of titration as a lab technique, and the principle on which it works. You should be able to follow this procedure to do your calculations in lab.

6. (2 pts) How and why must you condition your burets?

Name: Section:

Experiment 11 Pre-Lab Sheet: Second Day

1. (2 pts) What is the purpose of Part 1?

2. (2 pts) What is the purpose of Part 2?

3. (2 pts) What is the purpose of Part 3?

4. (4 pts) Find the concentration of a solution of sulfuric acid if it requires 30.45 mL of 0.1048 M NaOH to titrate 44.81 mL of the sulfuric acid solution. (Hint: write the balanced equation!)