Determining the Equivalent Mass of an Unknown Acid by Titration

Objectives

- To perform an analytical titration.
- To standardize a basic solution.
- To determine the equivalent mass of an unknown acid.

Background

Titrations are used extensively in analytical chemistry. Some applications of titrations include quality control in the food, cosmetics, beverages, and pharmaceutical industries; quantitative determination of chemical concentrations in environmental samples; and the measurement of various species in biological solutions. In this experiment you will use titrations to determine the molecular mass of an unknown acid.

In analytical chemistry when we use a titration or other precise means to determine the concentration of a solution it is called **standardizing** the solution. In the first part of this experiment you will prepare a sodium hydroxide, NaOH, solution. You will then standardize this solution by titrating it with a standardized hydrochloric acid, HCl, solution of known molarity.

In the second part of this experiment you will use your standardized sodium hydroxide solution to titrate a sample of an unknown solid acid. From your titration data you will determine the number of moles of acid in the sample. From this and the mass of the acid in the sample you will determine the equivalent mass of the unknown acid.

In both parts of this experiment you will perform at least three titrations to ensure the precision of your results.

It is assumed in this experiment that you are already familiar with the basic techniques and calculations used in performing titrations. If not, you should review this material prior to starting this experiment.

Procedure:
Materials and Equipment:

You will need the following additional items from the stockroom for this experiment:

- Two 50-mL burets

Safety:

GENERAL SAFETY: Students must wear safety goggles and a lab coat at all times.
GLOVES: Gloves are needed when handling:

- sodium hydroxide
- Phenolphthalein
- Solid acids (Special caution should be taken to prevent inhalation of the solid acids, especially in the case of a large spill. If this occurs, consult your instructor.)

WASTE DISPOSAL: All waste containing your unknown acid must be disposed of in the hazardous-waste container in the fume hood. All other waste may be disposed on in the sink.

Experimental Set-up and Procedure:

General Notes

This experiment is performed solo. Because being able to perform a precise and accurate titration is important to a variety of fields your work in this experiment will be graded almost entirely on the precision and accuracy of your results. It is strongly suggested that you check your results before disposing of your solutions in case additional trials are required. Your report is due upon completion of the experiment.

All glassware must be properly rinsed before performing this experiment. Your flasks should be rinsed several times with deionized water before use. Do not use soap in your rinsings as this could effect your results in the experiment. Your burets and funnels should be rinsed several times with deionized water and then several more times with ~5-mL samples the solution you will fill them with. Label all solutions to avoid cross contamination.

Burets should be properly clamped into the buret stand so that they are held in a vertical position. Be certain that buret tips are firmly pressed in and that there are no air bubbles in the tips. Also be sure to remove your funnels before beginning you titrations. Finally, It is a good idea to double-check all readings of your buret with your lab partner to ensure accurate results.

A proper endpoint in a titration is reached when a single drop of titrant causes the indicator to change color and the color persists while swirling the solution for at least 30 seconds. If you are unsure whether only a single drop of titrant was dispensed, then you should discard and repeat the trial you are performing (or in some cases you may perform a back titration). The indicator shade being light or dark is not sufficient to determine if a proper endpoint has been reached; some trials may have a darker color than others because the titration curve is very steep in the region of the endpoint. The only accurate determination that the proper endpoint has been reached is if you are certain that only a single drop was required to reach the endpoint signaled by the indicator color change. Any uncertain trials should be discarded.

Part A: Standardization of a Sodium Hydroxide Solution

In this part of the experiment you will prepare and standardize a sodium hydroxide solution.

1. Rinse a 500-mL Florence or Erlenmeyer flask, a 250-mL beaker, a 600-mL beaker, two 50-mL burets, two small funnels, a small graduated cylinder, and three 250-mL Erlenmeyer flasks several times using deionized water. There is no need to dry these items after rinsing.

2. Using the small graduated cylinder, measure approximately 8 mL of 6-M NaOH solution into the 500-mL flask. Using your 600-mL beaker, measure approximately 400 mL of deionized water and add this to the 8 mL of NaOH in your flask. (These measurements do not need to be exact because you will standardize this solution later). Stopper and label this solution, “sodium hydroxide solution”. Swirl your sodium hydroxide solution well before proceeding to be certain it is completely mixed. Leave the stopper in place when you are not pouring the solution into another container.

3. Rinse your 250-mL beaker 2-times with approximately 10-mL samples of the standardized HCl solution, then collect approximately 100 mL of this solution in your beaker. Label this beaker, “standardized HCl solution.” Record the exact molarity of this solution (from the reagent label) on your data sheet. If you need more of this reagent be certain that its molarity is the same.

4. Rinse one of your burets 2 times with approximately 10-mL samples of your standardized HCl solution. Then fill the buret and clamp it vertically in your buret stand. Label this buret, “HCl.” Proceeding in a similar manner, rinse and fill your second buret with some of the "sodium hydroxide solution" you prepared in your 500-mL flask (***NOT*** *the 6M NaOH from the stock bottle*) and label this buret, “NaOH.” Check both buret tips to make sure they are pressed in firmly and there are no air bubbles present. If you used a funnel to fill either buret remove the funnel before proceeding. Keep these funnels next to their respective burets so that you don’t inadvertently cross contaminate either solution.

5. Record the initial volume of HCl solution in your buret on your data sheet to the nearest hundreth of a milliliter. It often helps to hold a white card or sheet of paper behind the buret to aid in this reading. It is a good idea to have a lab partner double-check your buret readings in this and subsequent steps.

6. Now transfer approximately 20 mL of the HCl solution from your buret to a clean rinsed 250-mL Erlenmeyer flask. (This flask does not need to be dried because the amount of deionized water inside will not change the number of moles of acid added). Record the final volume of HCl solution in your buret on your data sheet.

7. Add 2 or 3 drops of phenolphthalein indicator to the HCl solution in the 250-mL Erlenmeyer flask.

8. Record the initial volume of NaOH solution in your second buret on your data sheet. Use this solution to titrate the HCl solution in the 250-mL Erlenmeyer flask. The titration endpoint is signaled when a single drop of NaOH solution added to your flask causes a faint pink color to persist in the solution for at least 30 seconds while swirling. (See the general notes at the beginning of this procedure for more details about determining the endpoint).

9. If you go past the endpoint in this step you may **back titrate** by adding a small amount of additional HCl from your first buret to the solution in your Erlenmeyer flask. Be sure to record this second buret reading for the HCl solution on your data sheet and cross out your original final buret reading. Now continue titrating with your NaOH solution to the new endpoint.

10. Refill each of your burets and perform two additional titrations in a similar manner.

11. Calculate the molarity of your NaOH solution using the data from each trial. If at least 2 of these trials agree with each other to within 1% you may proceed. If not then perform additional titrations until at least 2 molarities agree within 1%.

12. Calculate the average of all trials that agree within 1%.

Once you have achieved these conditions your sodium hydroxide has been standardized. Record the average (or standardized) molarity of this solution on your data sheet. You will use this value for your calculations involving this solution in Part B.

Part B: Determining the Equivalent Mass of an Unknown Acid

In this part of the experiment you will use your standardized sodium hydroxide solution to titrate an unknown acid and determine its equivalent mass.

1. Clean and rinse a 250-mL Erlenmeyer flask using deionized water. This flask does not need to be dried.

2. Obtain a vial containing your solid acid unknown from your instructor. Handle this vial only with a clean paper towel or tongs to avoid getting moisture, hand cream, or fingerprints on this vial. Record the unknown number of your solid acid on your data sheet.

3. The mass of solid acid you will require for each trial depends on the particular unknown acid sample used and is given on the label of each sample. This should be a value between 0.1 and 0.5 grams.

We will now use a technique called **weighing by difference** to measure this mass of solid acid used in each trial. Weighing by difference is an analytical technique for precisely determining the amount of substance added to a container. Simply placing your flask onto

the balance and then weighing your flask and its contents is not always precise because moisture or other substances on the outside of your flask may result in some error in your measurements.

4. To weigh the unknown solid acid by difference carefully remove the cap from the sample vial and set it aside. Weigh your sample vial and its contents on the analytical balance. Record this mass on your data sheet. Now transfer a small amount of the solid acid from the vial directly into your 250-mL Erlenmeyer flask. Do not use a spatula or other device to transfer the solid as this may result in some loss due to sticking to the spatula. Now reweigh the vial containing the remaining unknown solid acid sample. The difference between the final and initial mass of the vial and its contents is equal to the mass of the solid transferred to the flask. If the mass of solid that you added is less than the amount required, transfer more of the solid to the flask and then reweigh the vial and its contents, repeating as necessary. Proceed cautiously because if you transfer too much of the solid you will need to dispose of it, re-rinse the flask, and start your weighing by difference procedure again. You should weigh by difference for all three trials in this part of the experiment.

5. Now add about 50 mL of deionized water (this amount does not need to be exact) and 2 to 3 drops of phenolphthalein indicator to the contents of this flask. Swirl to mix. Your unknown acid may be relatively insoluble, so don't worry if it does not all dissolve in this step.

6. Refill the buret containing your standardized NaOH solution and remove the funnel.

7. Use your standardized NaOH solution to titrate your solid acid sample. Once again the endpoint occurs when a single drop of NaOH solution added to your flask causes a faint pink color to persist in the solution for at least 30 seconds while swirling.

8. Refill your buret and perform two additional titrations in a similar manner.

9. Using the average molarity you determined in Part A for your standardized NaOH solution, calculate the experimentally determined equivalent mass of your unknown acid for each trial and determine the average of these values.

Clean up:

Only the titrated solutions containing your unknown solid acid need to be poured into the chemical waste container. All other solutions, including any excess standardized HCl or NaOH solution may be disposed of in the sink. Rinse your burets well and then return these to the stockroom. Return any unused unknown solid acid to your instructor making certain that you have recorded the unknown number on your data sheet.

Name: _____ Date: _____

Lab Partner: _____ Lab Section: _____

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Part A – Standardization of a Sodium Hydroxide Solution

Molarity of standardized HCl solution: _____ M

Data	*Trial 1*	*Trial 2*	*Trial 3*	*(Trial 4)**
Initial HCl buret reading:				
Final HCl buret reading:				
(2^{nd} Final buret reading)†:				
Volume of HCl used:				
Initial NaOH buret reading:				
Final NaOH buret reading:				
Volume of NaOH used:				
Calculations	*Trial 1*	*Trial 2*	*Trial 3*	*(Trial 4)**
Moles of HCl used:				
Moles of NaOH titrated:				
Molarity of NaOH:				

* Only three trials are required, but space for a fourth is given if needed.
† Optional: Only required if a back titration is performed.

Average molarity of NaOH solution: _____ M

In the space below, clearly show all calculations for your Trial 1 data only:

Part B – Determining the Equivalent Mass of an Unknown Acid

Unknown Number of Solid Acid: _____

Data	*Trial 1*	*Trial 2*	*Trial 3*	*(Trial 4)**
Initial mass of vial:				
Final mass of vial:				
Mass of acid used:				
Initial NaOH buret reading:				
Final NaOH buret reading:				
Volume of NaOH used:				
Intial HCl buret reading:				
Final HCl buret reading:				
Volume of HCl used:				
Calculations	*Trial 1*	*Trial 2*	*Trial 3*	*(Trial 4)**
Moles of NaOH used:				
Moles of HCl used:				
Moles of H^+ titrated:				
Equivalent mass of acid:				

*Only three trials are required, but space for a fourth is given if needed.

Average equivalent mass of unknown acid: _____ $g \cdot eq^{-1}$

In the space below, clearly show all calculations for your Trial 1 data only: