EXPERIMENT 6

"OH HOW BITTER A THING IT IS..." ACID BASE TITRATIONS

Techniques

- dilution
- standardization
- volume titration
- unknown determination

MSDS available for

- sodium hydroxide, NaOH (aq)
- sulfuric acid, H_2SO_4 (aq)
- hydrochloric acid, HCl (aq)

Principles

- acidity/basicity
- concentration
- equivalence point, endpoint
- weight percent

Recommended Advanced Reading

- **Chapters 16,17 in Silberberg, Lavieri and Venkateswaran, 1st CE, McGraw-Hill, 2013.**

INTRODUCTION

The beginning

This is the first lab experiment where you will work with unknown solutions. You will use the techniques learned in the lab today to determine the concentrations of some unlabelled solutions. While you will not be graded solely on the correct answer, it is important to remember that if you work carefully, you should have no problem getting very close to the real value, and that will be an important consideration in the assignment of a grade. In today's session you will:

- prepare a solution of sodium hydroxide, NaOH, by dilution
- standardize the NaOH solution
- use your standardized NaOH solution to determine the concentration of an unknown acid
- use your standardized NaOH solution to determine the concentration and weight percent of acid in a juice sample

Please note that you will be working ALONE in this experiment. This is a general overview of what you will be accomplishing in this experiment.

EXPERIMENT 6: *Acid Base Titration*

Introduction

"As a beginning chemist....make a habit of weighing carefully, of pouring from one vessel to another without spilling and without missing the last drop, and of observing the small details...."(J. J. Berzelius, 1779-1848)

What are Acids and Bases?

Acids and bases are substances that are commonly encountered, not only in the laboratory, but also in our daily lives. It is well known that substances that are acidic are sour in taste (as we know from soft drinks, vinegar, and lemonade!), and that strong acids are corrosive. It is less well known that bases are bitter in taste and that strong bases are corrosive as well. Why is it that we know more about acids than bases, and what is it that distinguishes them from one another?

Acids and bases may be defined in many different ways. The Arrhenius definition of an acid is the simplest and states that **acids** are substances that behave as **proton donors** when dissolved in water, and that **bases** are substances that behave as **hydroxide ion donors** when placed in water. What is a proton? A proton is better known as a hydrogen ion, H^+ (aq). The hydroxide ion is the species OH^- (aq). Thus, HNO_3, which dissolves in water according to the following equation,

$$HNO_3\,(aq) \rightarrow H^+\,(aq) + NO_3^-\,(aq) \qquad [1]$$

can be seen to donate a proton when it is dissolved and is thus called an acid. On the other hand, NaOH dissolves in water as follows:

$$NaOH\,(aq) \rightarrow Na^+\,(aq) + OH^-\,(aq) \qquad [2]$$

NaOH donates a hydroxide ion upon dissolution in water and is thus classified as a base. Arrhenius' definition was found to be limited when substances such as HCO_3^- (aq) and CO_3^{2-} (aq) were found to behave as bases as well, but did not donate hydroxide ions in solution. The slightly expanded Brønsted-Lowry definition remains essentially the same for acids, but expands to add that **bases** are also substances that **accept hydrogen ions** when placed in water, thus explaining the behaviour of HCO_3^- (aq) and CO_3^{2-} (aq). However, subsequently, many substances that have been found to display acid/base behaviour do not even contain hydrogen ions! The most expanded definition at present is thus the Lewis definition of acids and bases in which **acids** are defined as being **electron pair acceptors** and **bases** are defined as being **electron pair donors**. For the purpose of this experiment, the Arrhenius definition is sufficient, as the acids and bases used in this lab contain hydrogen ions or hydroxide ions.

Acids and bases can normally be described in two ways: according to their strength and their concentration. A **strong** acid or base is one that dissociates almost to completion when the acid or base is placed in water. Thus in the case of Equations [1] and [2], there is very little HNO_3 or NaOH remaining once these species have been placed in water: all or almost all of it has been dissociated to produce H^+, NO_3^-, Na^+, or OH^- dissolved in water. Thus, HNO_3 and NaOH are examples of a strong acid and a strong base. A **weak** acid or base is one that does not dissociate completely when placed in water. Acetic acid and ammonia are examples of a weak acid and a weak base. In this lab, we will be dealing mainly with strong acids and bases.

The concentration of an acid or base will depend on the amount of acid or base added to a given volume of water. If there is **more acid/base** and **less water**, the solution is **more concentrated**. If there is **less acid/base** and **more water**, the solution is **less concentrated**. How do we measure concentration? In general, the amount of the acid/base, in this case called the **solute**, is measured in ***moles***, and the volume of the **solution** (which comprises the solute AND the solvent) is measured in ***litres***. The unit for concentration is thus ***moles per litre, mol/L***. This relation can be expressed by the following equation:

$$\text{Concentration}\left(\text{mol} / \text{L}\right) = \frac{\text{amount of solute (mol)}}{\text{volume of solution (L)}} \qquad [3]$$

where the number of moles of solute can be found by dividing the mass of the solute (in grams) by the molar mass of the solute (in grams per mole).

Titration of Acids and Bases

Acids and bases react to form a salt and water. This is the general reaction that is termed a **neutralization** reaction. The amount of acid required to neutralize a base is determined by the stoichiometry of the reaction. The **equivalence point** of the reaction is the point at which the amount of acid added is exactly enough to titrate the amount of base present. For example, if sulfuric acid, H_2SO_4 (aq) is reacted with sodium hydroxide, NaOH (aq), the neutralization reaction that occurs may be written as,

$$H_2SO_4\,(aq) + 2\,NaOH\,(aq) \rightarrow Na_2SO_4\,(aq) + 2\,H_2O\,(l) \qquad [4]$$

From the equation as written, it can be seen that **1 mole of H_2SO_4** reacts with **2 moles of NaOH**. If we had 25 mL of a 0.5 mol/L solution of NaOH and we were titrating it with 0.5 mol/L H_2SO_4, what volume of acid would be required? The solution may be divided into simple steps, so that one equation may be formulated that will provide an easy methodology for solving such a problem.

Step 1. Find the amount (mol) of base

$$\begin{aligned} n_{base} &= Concentration_{base} \times V_{base} \\ &= 0.5\ mol/L \times 25\ mL \\ &= 0.5\ mol\ L^{-1} \times 25 \times 10^{-3}\ L \\ &= 1.25 \times 10^{-2}\ mol \end{aligned}$$

Step 2. Determine the relationship between the amount (mol) of acid and base

<u>1</u> mole of <u>acid</u> reacts with <u>2</u> moles of <u>base</u>, therefore

$$\mathbf{n_{base} = 2\ n_{acid}} \qquad [5]$$

(Remember!!! that the amount (mol) of base is **twice** as big as the amount (mol) of acid!)

Step 3. Calculate the amount (mol) of acid required

$$n_{acid} = 0.5\, n_{base} \text{ (rearrangement of Equation 5)}$$
$$= 0.5 \times 1.25 \times 10^{-2} \text{ mol}$$
$$= 6.25 \times 10^{-3} \text{ mol}$$

Step 4. Calculate the volume of acid required

$$V_{acid} = n_{acid} / \text{ concentration}_{acid}$$
$$= 6.25 \times 10^{-3} \text{ mol} / 0.5 \text{ mol L}^{-1}$$
$$= 1.25 \times 10^{-2} \text{ L}$$
$$= 12.5 \text{ mL}$$

We can simplify the process by recognizing that the entire process hinges on the stoichiometry between the acid and the base, that is Step 2. Remember that the amount (mol), n, is equal to the concentration x volume. In Equation [5], if we replace "n" by the expression "concentration x volume", we can substitute directly into one equation and isolate the quantity we require.

$$\mathbf{n_{base} = 2\ n_{acid}}$$
$$\mathbf{concentration_{base} \times V_{base} = 2\ concentration_{acid} \times V_{acid}}$$

Using ***c*** as our symbol of concentration, we can write this as

$$\mathbf{c_{base} \times V_{base} = 2\ c_{acid} \times V_{acid}} \qquad [6]$$

This equation is valid for the stoichiometry of Equation 4, but not for all stoichiometries! A more general form of the equation would be

$$c_{base} \times V_{base} = \frac{b}{a} c_{acid} \times V_{acid} \qquad [7]$$

where **b** is the stoichiometric coefficient of the base and **a** is the stoichiometric coefficient of the acid in the chemical equation.

Endpoint

Now we know that the equivalence point is when equal amounts of acid and base are present in the solution, and we know how to calculate what the equivalence point is, but how do we know we have actually reached this point? Acids and bases are generally (and the ones we will be working with are definitely) colourless, and aside from generating a small quantity of heat, there are no bells or whistles when we reach the point where the amount of acid and base are the same. So how do we know?

We make use of an **indicator**. An indicator is itself a weak acid that dissociates to different extents in a solution depending on the acidity of the solution. The important quality of an indicator is that it is **coloured**. Generally the undissociated and the dissociated species of the indicator have different colours. If the indicator is carefully chosen, the colour of the indicator will change very close to the point at which equal amounts of acid and base are present in the solution. Thus, in a good titration, the **equivalence point** and the **endpoint** are very close.

Concept of the Experiment

In this experiment, there are four main parts. First, you will prepare a solution of NaOH by diluting a given solution of NaOH. Second, you will determine the exact concentration of your prepared NaOH solution by standardizing it against an acid solution of known concentration. Third, you will use your standardized base solution to titrate an unknown acid solution using a volumetric titration. Finally, you will titrate a juice sample and determine the percent by mass of acid in the juice.

To standardize your base solution, you will use a known volume of your base solution, and add an acid solution of known concentration until you obtain a visible endpoint. You can then use Equation 7 to calculate the concentration of your base solution.

The percent by mass of acid in a juice sample can be determined once the concentration of the acid in the juice has been found. The density of the juice sample will be supplied and can be used to determine the percent by mass of acid in the juice.

Safety Precautions

1. **Wear approved eye protection at all times**.

2. The base solution supplied is concentrated. Concentrated bases are **corrosive**.

3. Acids and bases, even in dilute solutions, are **corrosive**. Ensure that if you spill any acid or base, you clean up immediately (it is easy to mistake these solutions for water). Rinse thoroughly with water for at least 15 minutes if any acid or base is spilled on skin or clothing,

(TTD) Things to Do

- Complete the prelab exercises before coming to the laboratory.
- Prepare a diluted NaOH solution from the concentrated reagent provided.
- Standardize the diluted NaOH solution using an acid of known concentration.
- Use the standardized NaOH solution to determine the concentration of an unknown acid sample by volumetric titration.
- Titrate a juice sample and determine the concentration and the percent mass of acid in the juice.
- Use safe laboratory procedures at all times.

PLEASE NOTE: **You will be working individually in this experiment and not with your partner. When preparing for this experiment, please read and plan accordingly!!**

PROCEDURE

Equipment and chemicals needed

Chemicals	*Equipment*
6 mol/L NaOH	3 - 150 mL beakers
Standard Acid solution	100 mL beaker
Unknown Acid solution	2 - 25 mL burets
juice sample	2 funnels
phenolphthalein	3 - Erlenmeyer flasks
	10 , 50 mL graduated cylinders
	400 or 600 mL beaker

Acid Base Titration

Starting the experiment

Preparing a Solution of NaOH by Dilution

1. Rinse a 600 mL beaker with distilled water and dry it.

2. Use the 10 mL graduated cylinder to measure approximately 8-9 mL of concentrated NaOH. NOTE: Do **NOT** obtain the NaOH in a graduated cylinder! Use a small beaker and share with the people in your work area!!! Note the concentration of the NaOH AS WRITTEN.

3. Add approximately 480 - 490 mL of distilled water to the 600 mL beaker. Add the concentrated NaOH to the water WHILE stirring well. Continue to stir for 2 min.

4. Pour the excess concentrated NaOH solution down the drain (if nobody else in your area can use it!), flushing with plenty of water.

Calculations 1

5. Calculate the approximate concentration of the diluted NaOH solution.

Moving on

Standardizing the Diluted NaOH Solution

6. Label one buret "NaOH" and a second buret with the name of your standard acid. The standard acid is **MONOPROTIC**.

7. Fill the burets with the solutions as labelled using the funnels (BE CAREFUL not to mix up burets and solutions!!).

8. Record the initial buret reading and then transfer 20 - 25 mL of the standard acid into a clean Erlenmeyer flask. NOTE the final reading on the buret and calculate the EXACT volume of acid that was transferred. REMEMBER: The buret can be read to 2 DIGITS AFTER THE DECIMAL. Add 2-3 drops of phenolphthalein to the flask.

9. Record the initial buret reading on the buret containing the NaOH solution. Add the diluted NaOH to the flask containing the acid slowly, swirling constantly, until the first pale pink colour that lasts for 30 SECONDS is obtained. HINT: Place the flask on a piece of white paper to see the colour clearly!

10. Record the volume of base needed to reach the endpoint.

11. Repeat steps 7 - 9 at least three times or until there is no more than a 0.2 mL difference between your results.

Calculations 2

12. Calculate the concentration of the diluted NaOH. Is the result reasonable?

Determining the Concentration of an Unknown Acid

13. Using a buret transfer 20 - 25 mL of the unknown acid sample into a clean Erlenmeyer flask. Note the EXACT volume of the acid. Add 2 - 3 drops of phenolphthalein. The unknown acid is **DIPROTIC**.

14. Repeat steps 9 and 10 using the standardized NaOH solution.

15. Repeat steps 13-14 at least three times, or until reproducible results are obtained.

Calculations 3

16. Determine the concentration of the unknown acid sample.

Almost there!

Determining the Mass Percentage of Acid in a Juice Sample

17. Ask your demonstrator to assign you a juice sample. NOTE: All the juices are a type of citrus juice; they contain citric acid, $\mathbf{H_3}C_6H_5O_7$. The bolded hydrogen atoms are titratable, and you should be able to determine the correct ratio of **b/a** in Equation [7]. The acid in the juice is **TRIPROTIC**.

Finishing the experiment

18. Titrate the juice as described in steps 13 - 16 to determine the concentration of acid in your juice sample. Do at least three trials, or enough to obtain reproducible results.

19. Obtain the density of your juice from the demonstrator.

Calculations 4

20. Calculate the concentration of the acid in the juice and the mass percentage of acid in the juice.

Cleaning Up!

21. Rinse the flasks, and all the burets and beakers used thoroughly with warm water and then with distilled water. All solutions may be poured into the sink and flushed with plenty of water. Clean the counters of all paper towels before leaving.

22. Remember to get your raw data, written in PEN, signed by your TA and to attach this raw data to your report in order to receive a grade!

Calculations

1. Use the initial volume of NaOH, its initial concentration, and the total volume to determine the approximate concentration of your NaOH solution, in mol/L, using $c_1 V_1 = c_2 V_2$ (Remember: the total amount (mol) is the same!)

2. Use the exact volume of the standard acid, the exact concentration of the standard acid, and the exact volume of

your base used in the titration to determine the EXACT concentration of your NaOH, using Equation 7.

3. Use the exact concentration of your NaOH and the volume you used in your titration, as well as the volume of your unknown acid to determine the concentration of your unknown acid using Equation 7.

4. Use the exact concentration and volume of your NaOH solution and the volume of your juice to determine the concentration of the acid in your juice using Equation 7.

5. Use the density of the juice, the calculated concentration of the acid in your juice, the molar mass of the acid in your juice and the formula below to determine the mass percent of acid in the juice.

$$\text{Mass Percent of Acid in Juice} = \frac{(c_{acid}, mol/L)(MM_{acid}, g/mol)}{(density_{juice}, g/mL) \times 1000} \times 100\,\%$$

Points to Ponder

- Why is there a factor of 1000 in the equation for the mass percent of acid in the juice?
- Does it matter what volume of concentrated NaOH you use in the beginning?
- Why do we determine the concentration of the NaOH just before we use it?
- What are the sources of error inherent in the experiment? How does each source of error contribute to the result (ie. does it increase or decrease the value of the concentration?)

Lab Report

- *Refer to page iii in the FYI section and the lab report section in experiment 1.*

- Complete all the calculations, as outlined in the procedural section, and don't forget to include significant figures. **Failure to include significant figure values will result in substantial reduction of the report grade!**