EXPERIMENT

Titration of the Weak Acid Potassium Hydrogen Phthalate (KHP)

INTRODUCTION

Materials generally considered to possess acidic and/or basic properties are widely distributed in nature and range from simple inorganic materials through organic and biological molecules of great complexity. Since acid-base equilibrium is a general phenomenon, it is advantageous to use it as an analytical tool.

The reaction between an acid and a base is called **neutralization**. The products of an acid-base neutralization reaction are water and an ionic compound. The general reaction involved in neutralization titrations can be depicted as follows:

\[
\text{HA} + \text{MOH} = \text{H}_2\text{O} + \text{MA}
\]

(reactant) (reactant)

A commonly used method of expressing concentration is **molarity** (M). The equation defining molarity is:

\[
\text{Molarity (M)} = \frac{\text{mol of solute}}{\text{volume of solution in L}}
\]

Because **titration** is a quantitative, volumetric procedure used to determine the concentration of a solute in a solution, it is possible to determine the concentration of an acid in a solution of the acid by measuring the volume of base needed to just consume the acid.

The point at which the hydronium ion from the acid is just completely neutralized is called the **equivalence point** of the titration. At the equivalence point, the number of moles of hydronium ion neutralized and number of moles of hydroxide ion added are equal.

**Indicators**, water-soluble dyes that have one color in acidic solutions and another in basic solutions allow us to know when the equivalence point is reached. In this experiment, the indicator that works best is phenolphthalein (feen-ohl-thay-leen). It changes from colorless on the acid side to red on the basic side.

In this experiment you will determine the exact concentration of the strong base NaOH by measuring the volume of base needed to just neutralize a precisely weighed amount of the weak acid, **potassium hydrogen phthalate** (KHP). The reaction of KHP with sodium hydroxide is shown below.

\[
\begin{align*}
\text{O} & \quad \text{K}^+ \\
\text{C} & \quad \text{O} \\
\text{C} & \quad \text{O} \\
\text{C} & \quad \text{OH} \\
\text{O} & \quad \text{C} \\
\end{align*}
\begin{align*}
+ \quad \text{NaOH} & \rightarrow
\begin{align*}
\text{O} & \quad \text{K}^+ \\
\text{C} & \quad \text{O} \\
\text{C} & \quad \text{O} \\
\text{C} & \quad \text{Na}^+ \\
\text{O} & \quad \text{H} \\
\end{align*}
+ \quad \text{H}_2\text{O}
\end{align*}
\]
At the equivalence point:

\[
\text{Number of moles of KHP neutralized} = \text{number of moles of NaOH added}
\]

Restated in an equation which holds at the equivalence point:

\[
\frac{g \text{KHP}}{204.23 \text{ g/mol}} = V_{\text{NaOH in liters}} \times M_{\text{NaOH}}
\]

In the titration experiment, all terms are known except \(M_{\text{NaOH}}\). The equation is solved for \(M_{\text{NaOH}}\).

\[
M_{\text{NaOH}} = \frac{\text{mass KHP (g)}}{V_{\text{NaOH in liters}} \times (204.23 \text{ g/mol})}
\]

A. GENERAL PROCEDURE

1. Each student, CHECK-OUT a buret, magnetic stirrer, and magnetic stir bar from the Chemistry stockroom.

2. Clamp the buret to a stand using a buret clamp.

3. Obtain about 125 mL of the approximate 0.1 M NaOH solution in a clean beaker. Cover the beaker with a watchglass.

B. CONDITIONING THE BURET

1. Remove the cork from the buret and pour out the distilled water into the sink by inverting the buret and opening the stopcock.

2. Close the stopcock (horizontal position) and fill the buret with 5-10 mL of the standardized NaOH solution. This will replace the water coating with a coating of the NaOH solution.
   a. Put the cork back on the buret and carefully tilt the buret, rolling it between your fingers in order to coat the entire length of the buret with NaOH.
   b. Remove the cork and open the stopcock to allow the NaOH solution to drain through the tip of the buret.
   c. Repeat this procedure one more time.
   d. At this time, DISCARD THE CORK IN THE TRASH BIN. A new cork will be issued when the buret is returned to the stockroom.

3. Secure the buret back on the ring stand making sure that the buret is not crooked. Always make sure the buret is clamped in a perfectly VERTICAL position before taking any readings.

SODIUM HYDROXIDE IS CORROSIVE!

WEAR GOGGLES AT ALL TIMES AND IMMEDIATELY CLEAN UP ANY SPILLS.
C. TITRATION PROCEDURE

You are to determine the true molarity of the NaOH solution to four significant figures by titration of a known mass of KHP as specified on the drill sheet.

1. Using a small funnel, fill the rinsed buret with the NaOH solution above the zero mark as shown in Figure 1.

![Figure 1 Filling a buret.](image)

2. Adjust the level of the solution so that the bottom of the meniscus is at or slightly below the zero mark on the buret. To do this:
   a. Fully open the stopcock. To EXPEL ANY AIR BUBBLES FROM THE TIP, gently tap the side of the buret tip while solution is flowing as shown in Figure 2. If an air bubble is present during titration, VOLUME READINGS MAY BE IN ERROR!

![Figure 2 Removing air bubbles from buret tip.](image)

   b. Close the stopcock and visually check to be sure all air bubbles are removed.

3. Record the initial reading to the nearest 0.1 mL. Read the bottom of the meniscus as shown in Figure 3. Be sure your eye is at the level of meniscus, not above or below. Reading from an angle, rather than straight on, results in a parallax error. Remember, read DOWN a buret.

![Figure 3 Reading a buret.](image)
4. Place 50 mL of dH₂O into a 250 mL Erlenmeyer flask.

5. Use a CLEAN scoopula to weigh the exact mass of KHP specified on your drill sheet. Record this mass on the Data Sheet as shown on the balance.

**NOTE:** IF YOU SPILLED ANY KHP DURING THE WEIGHING PROCESS, CLEAN IT UP BEFORE CONTINUING.

6. Add the KHP to the 250 mL Erlenmeyer flask containing the 50 mL of dH₂O and add 2-3 drops of the Phenolphthalein indicator as shown in Figure 4. Place the small, white magnetic stir bar obtained from the stockroom in the flask.

![Figure 4 Preparing the solution for titration.](image)

7. Unwrap the magnetic stirrer’s electrical cord and plug into electrical outlet. Leave the Velcro cable wrap attached to the electrical cord. **DO NOT LOSE THE VELCRO CABLE WRAP!**
   a. Obtain a circular sheet of white paper from the bin and put it on the top of the magnetic stirrer.
   b. Put the 250 mL Erlenmeyer flask and its contents atop the magnetic stirrer.

8. Adjust the buret height, so that the tip of the buret is slightly below the rim of the titration flask. This will prevent the loss of titrant by splashing outside the flask.

9. Turn magnetic stirrer **ON** and proceed to mix the contents of the flask until the KHP has dissolved. Adjust the speed control knob to the desired position in order to accomplish this.

10. Begin titrating by opening the stopcock and allowing a stream of NaOH to run into the KHP solution while it is stirring. The solution should be delivered quickly until a couple of mL from the endpoint. You will see the indicator change color when the titrant (NaOH) hits the solution in the flask, but the color quickly dissipates upon stirring as shown in Figure 5.

![Figure 5 Delivering a stream of titrant.](image)
11. Approach the endpoint more slowly and watch the color of your flask carefully. Use a wash bottle to rinse the sides of the flask and the tip of the buret, to be sure all titrant is mixed in the flask as shown in Figure 6.

![Figure 6 Rinsing flask walls and buret tip.](image)

12. As you get nearer to the endpoint (i.e. when you start noticing that the desired color is persisting for a couple seconds), slow down the rate at which you add NaOH to about one drop at a time, so that you don’t overshoot the endpoint as shown in Figure 7.

![Figure 7 Adding drops near endpoint.](image)

13. Near the endpoint, the trail of pink color from each drop spreads out further and lingers for longer periods of time. **STOP** when one drop of base causes the entire solution to turn a PALE PINK color and the color persists for at least 30 seconds as shown in Figure 8. This is the endpoint.

![Figure 8 Endpoint.](image)
14. Addition of too much titrant leads to a deeper color coloration that won’t go away, even with swirling. If the flask looks like Figure 9, you have gone too far!

![Figure 9: Past the endpoint.](image)

15. Once you have reached the endpoint, record the final volume of NaOH from the buret on the Data Sheet to the nearest 0.1 mL as shown in Figure 10. Remember, read **DOWN** a buret. The volume of NaOH required to reach the endpoint is the difference between the initial and final volumes.

![Figure 10: Final buret reading.](image)

16. Refill the buret and perform the titration once more on another sample of KHP, recording your data on the Data Sheet. **DON’T FORGET TO ADD THE PHENOLPHTHALEIN INDICATOR.** Both titration determinations should agree to within 0.5 mL.

   a. To prevent the white magnetic stir bar in the Erlenmeyer flask from going down the drain, hold the **ROUND MAGNETIC RETRIEVER** against the bottom of the Erlenmeyer flask and proceed to empty its contents (Figure 11).

![Figure 11: Using a magnetic retriever](image)
D. BURET CLEANING INSTRUCTIONS

1. With the stopcock closed, add some distilled H₂O to the buret. Tip and roll the buret, allowing the water to have contact with the entire inside surfaces. Open the stopcock and allow the water to drain through the tip.

2. Repeat the rinse TWICE more with distilled H₂O. Use a wash bottle to rinse the OUTSIDE of the buret tip.

3. After the final rinse, COMPLETELY FILL THE BURET AS WELL AS THE TIP with distilled H₂O.

4. Wipe the buret DRY with paper towel before returning it to the stockroom.

5. Return to the stockroom for testing. The technician will perform a LITMUS TEST to ensure the cleanliness of the buret and will also issue a new cork at this time.

E. OTHER INSTRUCTIONS

1. CLEAN THE BOTTOM OF THE RINGSTAND WITH THE 409 PROVIDED.

2. Return to the stockroom all 3 items that were checked out at the beginning of the lab.
   a. Before returning the magnetic stirrer to the stockroom, NEATLY WRAP the cord with the Velcro strip provided.

3. REFILL water bottles up to the BLUE LINE with distilled water.
   a. Faucet is located at the front of the lab next to the whiteboard.

4. Return goggles to the GOGGLE CABINET by neatly arranging them on the racks. This is necessary for proper sterilization.

5. AFTER ALL EXPERIMENT SECTIONS HAVE BEEN COMPLETED, PLEASE WIPE THE BENCHES DRY THEN WIPE WITH THE 409 PROVIDED.

6. LOCK your locker before leaving the lab.
Known mass of KHP (on drill sheet) ________________

**Part C: WEIGHING OF KHP SAMPLE** (Show correct number of significant figures as well as units!)

<table>
<thead>
<tr>
<th>Trial 1</th>
<th>Trial 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actual Mass of KHP Used</td>
<td></td>
</tr>
</tbody>
</table>

**Part C: TITRATION OF KHP AGAINST NaOH** (Read the buret to the nearest 0.01 mL and show the correct number of significant figures as well as units)

<table>
<thead>
<tr>
<th>Trial 1</th>
<th>Trial 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial Buret Reading</td>
<td></td>
</tr>
<tr>
<td>Final Buret Reading</td>
<td></td>
</tr>
<tr>
<td>Volume NaOH Used in L</td>
<td></td>
</tr>
<tr>
<td>Molarity of NaOH Solution Determined by Titration</td>
<td></td>
</tr>
</tbody>
</table>

Average molarity of NaOH from titrations ________________

**CALCULATIONS**

1. Determine $M_{NaOH}$ for Trial 1 (show your work).

$M_{NaOH}$ for Trial 1 ________________
2. Determine $M_{\text{NaOH}}$ for Trial 2 (show your work).

\[ M_{\text{NaOH}} \text{ for Trial 2 } \]

4. Determine the average $M_{\text{NaOH}}$ for Trials 1-2 (show your work).

\[ \text{Average } M_{\text{NaOH}} \]