**INTRODUCTION:**

Bases react with acids. OH\(^{-}\) is a strongly basic ion, and NaOH is the most common strong base. When the generic acid, HA, reacts with NaOH, the reaction is:

Phosphoric acid, H\(_3\)PO\(_4\), is a polyprotic ("many hydrogen") acid. You will react sodium hydroxide with this acid while carefully monitoring the reaction for indications that stoichiometric quantities of the reactants have been used. "Stoichiometric" means that the quantities of reactants equal the amounts required in balanced chemical equations. "Carefully monitoring" means to repeatedly add one reactant in small quantities while watching for some physical sign that shows that the proper stoichiometric amount has been added. This process is called "titration". In this experiment, the reactants are dissolved in water. The solutions have been carefully prepared and their concentrations are marked on the labels. You will monitor the quantities of reactants used in the reaction by measuring the volumes of the solutions used. Two volumetric measuring tools will be used: a buret and a pipet. *(Refer to the "how to use" sheets on pages 103 and 104 for proper use.)* You will monitor the moment when the reaction has reached stoichiometric quantities of reactants using two different indicators and a pH meter. An "indicator" is a chemical that changes color as conditions in the solution change. You will use two different acid-base indicators that change color as the pH of the solution changes. Methyl orange changes from red to yellow as the pH changes from 3 to 5. Phenolphthalein changes from colorless to pink as the pH changes from 8 to 10. The "pH meter" is an instrument that measures the pH of a solution by checking the voltage output of a special battery (the pH electrode) which puts out various voltages in proportion to the pH of the solution surrounding the immersed end of the battery.

**EQUIPMENT:**

Work in pairs. All solutions may be discarded in the sink.

- Buret stand with one buret
- 10 ml pipet
- Magnetic stirrer
- pH meter with electrode
- pH 4 and 7 buffers, 5 ml each
- Stir bar and pipetting bulb
- 50 ml of 0.1M H\(_3\)PO\(_4\) in a clean, dry 50 ml beaker
- 100 ml of 0.1M NaOH in a clean, dry 150 ml beaker

**PROCEDURE:**

**Indicator titrations:**

Set up the equipment as shown in the diagram.

Rinse the buret with two 5 ml quantities of NaOH. Make sure the tip is filled with the NaOH and no bubbles are present in the tip. Fill the buret with NaOH to 1 cm above the top graduate mark. Do not have the flask under the buret while filling the buret. Open the stopcock to let the NaOH solution drain into a waste container until the bottom of the meniscus is at or under the top graduate mark. Record the volume reading. Rinse the pipet twice with small quantities of H\(_3\)PO\(_4\) solution. Pipet 10 ml of the acid solution into the flask. Add about 50 ml of deionized water for bulk.
**Methyl Orange:**
Add three drops of methyl orange indicator solution. Turn the magnetic stirrer up to give a small whirlpool without splashing. Add NaOH from the buret until the methyl orange changes color. Carefully observe the color changes that take place right at the point of entry of the stream of liquid from the buret into the liquid in the flask. Note how the solution is yellow at that point, but the color quickly dissipates with distance from the entry point. This color change is due to a localized excess of base. As the solutions mix, the total excess of acid in the flask causes the methyl orange to regain its red color. As the titration proceeds, the yellow at the entry point more and more slowly dissipates back to red. Finally, when the base is in excess, the color stays yellow. To perform a good titration, no more than one drop (!) of base should be added in excess. To achieve this, the rate of addition of liquid from the buret gets slower and slower as you approach the end point, until you are adding the titrant drop by drop, watching the color of the liquid in the flask. When the color has changed permanently (for 30 seconds), you have reached the endpoint of the titration. Record the final volume. Note that it is faster to overshoot the endpoint and to gain experience that will allow you to repeat the titration a second time with confidence, than to run through a titration with tedious, drop by drop addition from beginning to end.

**Phenolphthalein:**
Rinse out the flask. Pipet 10 ml of H$_3$PO$_4$ solution into the flask, and add about 50 ml of deionized water as you did for the first titration. Add 3 drops of phenolphthalein. Record the liquid level in the buret as the starting volume. Titrate until a permanent pink color appears. (Remember--only one drop of excess base.) Record the final volume on the buret.

**pH Meter Titration:**
Calibrate the pH meter according to the meter directions. Fill the buret to the zero mark with NaOH solution. Set up the equipment as shown in the diagram. Notice especially that a special support rod with a clamp for the electrode is clamped to the ring stand on the rubber stopper at the top of the support rod. Pipet 10 ml of acid into the beaker. The electrode should be as low as possible in the beaker, but above the path of the stir bar. Add enough deionized water to the beaker to cover 1 cm of the electrode. Record the buret reading and the pH.
You will add base from the buret into the flask in either 1 ml or 0.2 ml increments as directed below. You will see why there is this variation when you graph the data.

Add 1 ml of base from the buret and record the buret reading and the pH. Repeat the procedure until the buret reads 9 ml. Then add 0.2 ml increments of base, recording the buret reading and pH after each addition until the buret reads 11 ml. Return to 1 ml increments until 19 ml have been added. Then begin 0.2 ml additions once more until the 21 ml mark. Finally, add 1 ml increments until a total of 25 ml have been added.

At this point, clean up. Rinse the pipet and buret 3 times with deionized water. Fill the buret with deionized water and return it on its stand to the shelf. Make sure the stopcock is tight enough so the buret doesn’t leak. If the pipet bulb had liquid sucked into it, tell the instructor so the bulb will be rinsed out. Dispose of excess acid and base in the sink. Rinse the electrode and set it in the soaking beaker with deionized water. Turn the pH meter off.

**CALCULATIONS AND GRAPH:**
In order to interpret the two indicator titrations and the pH titration, you will calculate the mole ratio of base to acid for each indicator, and relate these calculated values to the graph of the variation of the pH of the solution with the volume of base added.
DATA

Indicator titrations:

<table>
<thead>
<tr>
<th></th>
<th>Methyl Orange</th>
<th>Phenolphthalein</th>
</tr>
</thead>
<tbody>
<tr>
<td>final volume</td>
<td>____ml</td>
<td>____ml</td>
</tr>
<tr>
<td>initial volume</td>
<td>____ml</td>
<td>____ml</td>
</tr>
<tr>
<td>volume used</td>
<td>____ml</td>
<td>____ml</td>
</tr>
<tr>
<td>ratio, $V_{\text{base}}/V_{\text{acid}}$</td>
<td>____</td>
<td>____</td>
</tr>
</tbody>
</table>

Since the NaOH and the $\text{H}_3\text{PO}_4$ solutions are the same strength, 0.100M, the ratio of the volumes reacted is the same as the ratio of the moles reacted. (note: 10.0 ml acid was used)

pH titration: graph data on the grid below.

<table>
<thead>
<tr>
<th>ml</th>
<th>pH</th>
<th>ml</th>
<th>pH</th>
<th>ml</th>
<th>pH</th>
<th>ml</th>
<th>pH</th>
<th>ml</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

pH vs ml NaOH added grid.
QUESTIONS

1. Write out the balanced equation for the reaction of 1 mole of NaOH with 1 mole of H$_3$PO$_4$:

2. Write out the balanced equation for the reaction of 2 moles of NaOH with 1 mole of H$_3$PO$_4$:

3. Explain how the volume data from the indicator reactions corresponds to the dual steep slopes on the pH vs volume of base curve.

4. Explain how the pH range for the change of color of the two indicators corresponds with the shape of the pH curve.

5. Write out the net ionic reaction for 1 mole of OH$^-$ reacting with one mole of H$_3$PO$_4$. Below the first equation, line up and write out the net ionic reaction for 1 mole of OH$^-$ reacting with the non-water product of the first reaction:

6. Given similar concentrations, the stronger the acid, the lower the pH. Comment on the relative strengths of the acids H$_3$PO$_4$ and H$_2$PO$_4$$^-$. 