Titration of NaOH with potassium hydrogen phthalate to determine the molarity of NaOH (Text reference for titration: 19.4)

**Titration** is a common lab procedure that gives highly reproducible results for a variety of chemical analyses. A titration is often used to determine the concentration of a solution such as an acid or a base. It makes use of a known reaction between two chemicals, where one solution of unknown concentration is reacted with a precisely measured amount of another chemical whose molar concentration is known. An appropriate **indicator** is used to signal when chemically **equivalent amounts** of each chemical are combined, according to the **exact molar ratio** shown in the balanced equation for the reaction. This is known as the **equivalence point**. To measure solution volumes accurately, finely calibrated **burets** are used.

In this activity you will determine the exact molar concentration of sodium hydroxide solution recently prepared in class. Although you planned that the solution would be 0.5\(M\), the actual concentration will be different due to chemical interaction of the hydroxide ion with other dissolved substances in water (particularly, CO\(_2\)). Assuming the molarity is 0.5\(M\) would lead to inaccuracies in subsequent titrations where the concentration of the NaOH solution must be known with great accuracy. The process you will follow to learn the exact concentration of your NaOH solution is commonly called **“standardizing”** the base.

There are several “housekeeping” and safety steps to learn when doing titrations. You will find that most academic and professional laboratories have similar protocols.

**ALWAYS WEAR GOGGLES DURING ACID/BASE ACTIVITIES.**
**Work with clean glassware; use deionized water for all rinsings/mixings.**

**Preparing the base buret:**
1) Burets may be stored upside-down, often in pairs. We will use only one buret today – the one labeled for a BASE (should be on the right as you face the buret stand). Carefully place it right-side up, secured in the buret clamp. Be sure the valve (called a stopcock) is closed. Be careful that the tip of the buret does not hit other objects.

2) Fill the buret with deionized water and let it drain out into a “waste beaker”. This cleanses the interior of the buret and readies it to receive the solution to be titrated. If water runs smoothly along the inside of the buret, you know that the glass is clean. (In many labs you would be expected to wash the buret with detergent and a buret brush, followed by rinsing with tap and finally distilled water.) While the water is draining out, you should practice controlling the rate of fluid delivery. See if you can deliver liquid one drop at a time. Study the volume markings on the buret and try to read the level of the meniscus to the nearest 0.01 ml. **Always read buret volume markings at eye level.**

3) After thoroughly rinsing the buret add approximately 5 ml of NaOH solution to the buret. (It is advised that you dispense 50 – 60 ml of NaOH stock solution into a **very clean** beaker for easier pouring.) Drain this through the
tip of the buret to remove water and to coat the inside of the buret with base. Some of the solution may remain in the tip.

4) Fill the buret to slightly above the zero line. Drain some base through the tip to clear the buret tip of air. Stop when liquid is between the markings for 0.0 and 2.0 ml. Be sure to remove the hanging drop of base at the tip of the buret by touching the tip to the inside of the waste beaker. Record the initial buret reading as the line on which the meniscus sits.

Preparing the acid:

5) Clean/rinse a 250-ml Erlenmeyer flask with deionized water. It is important that your glassware be scrupulously clean!

6) Using a clean plastic weighing dish, obtain between 0.60 g and 0.70 g of crystalline potassium hydrogen phthalate (“KHP”). Record the exact mass of KHP that you use. **** Be patient with the weighing procedure – this mass of acid is critical.****

7) Transfer all of the KHP to a very clean Erlenmeyer flask along with approximately 100 ml of deionized water. You may squirt some deionized water into the weighing dish to make sure all of the KHP is transferred into the flask. Swirl the flask to dissolve the crystals – this may take several minutes.

8) Add two or three drops of phenolphthalein indicator to the flask and swirl to mix.

(Note: We are using a crystalline acid compound instead of an acid solution. If you titrate with an acid solution, you would prepare an acid buret the same way you prepared the base buret. You would deliver 15 – 20 ml of acid from the buret into a clean flask, add approximately 15 – 20 ml of deionized water and phenolphthalein, and proceed as described below.)

The Titration

9) Place the flask with the KHP solution and phenolphthalein under the buret. The buret tip should be about 1 cm inside the mouth of the flask to avoid outside loss of base.

10) Drip the base into the flask while swirling the flask. You can add 3-4 mL of the base quickly at first, but as the pink color starts to last longer, control the delivery of base so that you add only 1-2 drops at a time. Swirl the flask continuously, and rinse down splashes on the inside of the flask with water from a wash bottle.

11) When the faintest pink color persists for at least 15s, stop and record the volume marking on the buret.

12) Discard the solution in the flask down the drain. Rinse the flask with deionized water. You may repeat the titration one more time, using approx. the same amount of KHP. Be conservative with the amount of base you are using – you will need it for more labwork.
Clean-up

13) Ordinarily, unused solutions are not returned to stock bottles because of the risk of contamination. However, you need the NaOH for other labwork. Dispense unused NaOH back into your stock bottle for use in other activities.

14) Rinse burets with one complete rinsing with tap water, followed by another rinsing with deionized water. Place the buret securely upside-down, with the stopcock left open.

Data and Calculations:

<table>
<thead>
<tr>
<th></th>
<th>Trial 1</th>
<th>Trial 2</th>
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<tbody>
<tr>
<td>Mass of weighing dish and KHC₈H₄O₄</td>
<td>_____ g</td>
<td>_____ g</td>
</tr>
<tr>
<td>Mass of empty weighing dish</td>
<td>_____ g</td>
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<tr>
<td>Initial reading of base buret</td>
<td>_____ mL</td>
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</tr>
<tr>
<td>Final reading of base buret</td>
<td>_____ mL</td>
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Molar mass of KHC₈H₄O₄ | _______ g/mole |

Balanced equation: KHC₈H₄O₄ + NaOH → H₂O + NaKC₈H₄O₄
“KHP” + NaOH → H₂O + Na“KP”

1) Calculate the number of moles of KHP used for each titration.
   Trial 1: 
   Trial 2: 

2) Use the moles of KHP and the balanced equation to determine the number of moles of NaOH present at each equivalence point (“end point”):
   Trial 1: 
   Trial 2: 

3) Determine the volume of NaOH used in the titration: (express as liters)
   Trial 1: 
   Trial 2: 

4) Use the moles and volume of NaOH to calculate the molarity of your NaOH:
   Trial 1: 
   Trial 2: 

5) Compare your results for molar concentration of NaOH – determine the correct average molarity of the stock solution of NaOH. (Your two trials should give similar results.) Re-label your stock bottle with the accurate molarity of NaOH.