

# Acid-Base Titration

## Learning Goals:

1. Use the technique of acid-base titration to standardize (three significant figures) a basic solution against a primary standard.
2. Determine the molecular weight of an unknown acid (three significant figures) by using the standardized basic solution as a secondary standard in another acid-base titration.

## Abstract:

Acid-base titration is a technique commonly used to determine the moles of acid in a sample by adding a known volume of strong base of a known concentration. The strong base provides the hydroxide ions,  $\text{OH}^-$ , to react quantitatively with the acid. The point at which the acid is completely and exactly consumed (reacted with) the known quantity of base is called the equivalence point and is signaled by a color change in the solution (end point). This color change is created by an indicator dye which is extremely sensitive to the presence of even a small excess of aqueous  $\text{OH}^-$ . From the stoichiometry of the balanced chemical reaction, the number of moles of the unknown acid solution can be determined. If the number of grams of unknown acid is measured, the molecular weight can be calculated.

## Lecture Connections

## Important Equations

**Neutralization equation** for any monoprotic acid, HA, neutralized by strong base, NaOH:



### Equation 1: Neutralization Reaction

Note:  $\text{Na}^+$ <sub>(aq)</sub> and  $\text{A}^-$ <sub>(aq)</sub> are spectator ions in this reaction and are simply written as the salt byproduct,  $\text{NaA}$ <sub>(aq)</sub>.

As more and more of the base is added the excess acid is neutralized as shown in **Equation 1**. When more moles of  $\text{OH}^-$  are added than moles of HA initially present, the final solution contains excess  $\text{OH}^-$  which means the titration has passed the endpoint. If an acid-base indicator, such as Phenolphthalein, is

present there will be a color change at this endpoint. Even a fraction of a drop of excess base is all that is required to trigger the color change and INDICATE the end point of the titration.

✓ The color change at the *endpoint* is the experimental observable that tells us that the *equivalence point* -- equivalent numbers of moles of acid and base present -- has been reached.

**Titration equation** for monoprotic acid/base

**moles acid = moles base** at equivalence point.

To find the moles of base we use the concentration of the basic solution and its volume:

**moles base =  $M_{\text{base}}V_{\text{base}}$**

To calculate the number of moles of a known acid in a weighed sample the molecular Weight of the acid is used:

**moles of acid = grams of acid / Molecular Weight of acid**

For this titration the monoprotic acid to be used as the Primary Standard will be Potassium hydrogen phthalate.

Potassium hydrogen phthalate,  $C_8H_5O_4K$ , ("**KHP**" an acronym, not a chemical formula), is a very pure compound, which is a high molecular weight acid, (204.23 g/mole), does not change mass when exposed to the atmosphere and whose mass can be measured very accurately, is the primary standard for this experiment. A weighed sample of this acid will be titrated to a phenolphthalein endpoint with a solution of Sodium hydroxide that is approximately 0.1M. Sodium hydroxide absorbs water from the atmosphere and therefore can not be used as a primary standard. but a solution of standardized NaOH can be used as a secondary standard in the determination of the molecular weight of an unknown acid.

### **Prelab Assignment**

View the video clips on [How to Weigh](#) and [Redox Titration](#). You will need [QuickTime](#) video player to see them.

In your lab notebook, prepare the following information:

1. A brief (2-3 sentence) introduction to the lab.
2. A table of safety information that includes the chemicals used in the lab and any safety handling precautions. This information can be obtained from the MSDS safety sheets.
3. How many moles of acid are in 0.398 grams of Potassium hydrogen phthalate, "KHP"? (Be sure to show your calculations in the lab notebook.)
4. How many moles of base are in 14.36 mL of 0.105 M NaOH?

Give this information to your TA at the beginning of the lab. You will not be allowed to work in the lab without this information.

### Procedures:

Chemicals	Glassware and Supplies	Instruments
<ul style="list-style-type: none"> <li>• <u>Potassium acid phthalate, <math>\text{KHC}_8\text{H}_4\text{O}_4</math> ("KHP") as the primary standard in a small vial</u></li> <li>• <u>Assigned Unknown acid in a small vial</u></li> <li>• <u>Phenolphthalein indicator</u></li> <li>• <u>1 M Sodium hydroxide (NaOH) solution</u></li> </ul>	<ul style="list-style-type: none"> <li>• 3,250 mL Erlenmeyer flasks</li> <li>• 500 mL plastic bottle</li> <li>• <u>Buret with buret stand</u></li> </ul>	<ul style="list-style-type: none"> <li>• Balance</li> </ul>

## Determination of the Concentration of a Secondary Standard Basic Solution to three significant figures:

### 1. Prepare 3 solutions of primary standard:


Obtain a small vial of potassium acid phthalate (KHP, MW=204.23g/mol). Wipe the outside of the bottle to remove any KHP crystals or any other material that adheres to the bottle (fingerprints, water etc.)


Weigh the vial on the analytical balance and record its weight to the nearest 0.001 g.

Transfer about 0.5 g of KHP to a clean numbered 250 mL Erlenmeyer flask and reweigh the vial. Transfer another 0.5 g sample into a second

numbered Erlenmeyer flask and reweigh the vial. Repeat this one more time and then get the final weight of the vial.


You should now have three samples of KHP of known weight in three flasks. Add about 100 mL of distilled water to each of the flasks to dissolve the KHP. **Add 5 or 6 drops of phenolphthalein indicator to each of the flasks.** Set these aside to allow the KHP to dissolve.

 Record the weights to the nearest 0.001 gram in a data table. Since the weight of each sample will be slightly different, be sure to keep the numbers of the Erlenmeyer flasks organized so that you can tell the samples apart.

 In this step, you prepared acid (KHP) solutions with a known number of moles. Calculate the moles of acid in each flask. The chemical formula for KHP is  $\text{KHC}_8\text{H}_4\text{O}_4$ . These solutions will now be titrated with base (NaOH) to find the *PRECISE* concentration of the base.


## 2. Prepare your NaOH solution:

Fill a 500 mL plastic bottle half full with distilled water. Using a graduated cylinder add 50 mL of 1M NaOH to the bottle, mix the solution, fill the bottle with distilled water, and mix completely. This solution is about 0.1M NaOH.


 You will use the following titration to determine the concentration of the NaOH solution *precisely* to 3 significant figures.

## 3. Rinse and fill buret with NaOH:

Remove the buret from the buret stand. Using a small beaker, pour about 5 mL of the NaOH solution that you have prepared into the buret. Carefully tilt the buret and roll it between your fingers in order to rinse the entire length of the buret tube. Discard the wash solution in a waste container by allowing it to drain through the stopcock. Repeat this wash twice more. Fill the buret with your prepared NaOH solution and replace it on the buret stand. Place an empty beaker under the buret and open the stopcock to allow all of the air bubbles to escape from the tip of the buret. Adjust the level of the solution so that the bottom of the meniscus is slightly below the zero mark on the buret. Record the initial volume of the filled buret to two decimal places.

 Never hold a beaker with solution above eye level since spilling could cause you to get solution into your eyes.

 Record the initial volume of the filled buret to two decimal places.

 **Improve accuracy by *Splitting Drops*.** Open the stopcock very slowly until a drop is suspended from the tip of the buret. Touch the side of the flask to the tip and wash the drop down into the solution with a stream of distilled water from your squeeze bottle. When splitting drops gives you a permanent color change (one that does not fade before 30 seconds), you have reached the endpoint. If you are not sure whether or not you have reached the endpoint, add another split drop.

4. **Standardize the NaOH by titrating the PRIMARY standard acid:**

Place one of the numbered Erlenmeyer flasks containing a measured sample of KHP under the buret. Place a piece of white paper under the flask. This will make it easier to see the end point. Slowly add the NaOH solution in the buret to the flask. If right handed, use your left hand to control the flow from the buret and the right hand to swirl the Erlenmeyer flask. You must continuously swirl the flask during the titration to make sure that the base and acid solutions are thoroughly mixed. When the pink color appears and remains for just a second or two, with swirling, you are close to the endpoint and should add the base drop by drop. To reach the end point very accurately, you can use the **splitting drops** technique. The endpoint is reached when the addition of one drop of the base causes the solution to turn a very pale pink and remain pink, **while swirling**, for at least 20-30 seconds. Allow the solution in the buret to set for about a minute and then record the final buret reading to two decimal places. (The pink color may fade after a few minutes.)

 Record the final volume of the buret to two decimal places.

5. **Repeat the standardization:**

Refill the buret, wait a minute, recording the initial volume, and titrate the other two solutions in exactly the same manner as described above.

**Save the standardized NaOH solution in the plastic bottle for the next part!**



Record the initial and final volumes of the buret to two decimal places for each titration.

## 6. Calculate the molarity of the NaOH:



At the endpoint (when the indicator changed color), the number of moles of NaOH used in the titration is equal to the number of moles of KHP weighed out:

moles KHP = moles NaOH

Moles of acid = grams acid / molecular weight of acid (see sample exercise 3.9 in BLB)

Moles of NaOH = molarity of base ( $M_b$ ) X volume of base ( $V_b$ )

### Example Calculation

If 32.15 mL of NaOH solution is used to reach the endpoint in a titration of 0.985 g of KHP, what is the concentration of the NaOH solution used to titrate the KHP? Remember, at the endpoint, the number of moles of NaOH equals the number of moles of KHP.

$$\begin{aligned} \text{Molarity of NaOH} &= \left( \frac{1 \text{ mol NaOH}}{1 \text{ mol KHP}} \right) \left( \frac{0.985 \text{ g KHP}}{204.2 \text{ g/mol KHP}} \right) \left( \frac{1}{0.03215 \text{ L}} \right) \\ &= 0.150 \text{ M} \end{aligned}$$


Using the data from the three titrations, calculate the molarity of the NaOH solution from each titration to three significant figures. Calculate the average of the three values. Calculate their percent deviations from the average. (See Example.) If any of the three concentration value differs from the average by 5% or more, that value should be discarded and another titration performed. Record the final concentration of the Standardized Sodium hydroxide solution.

## Determination of Molecular Weight of an Assigned Unknown Acid:

### 1. Prepare and titrate the unknown acid solutions:


Weigh out 0.25 g of three samples of your Assigned Unknown Acid to the nearest 0.001g. Remember to weigh by difference as you did for the KHP samples. Dissolve each sample of unknown acid in water and add 5 or 6

drops of phenolphthalein indicator solution. Titrate the sample with your standard NaOH solution using the same procedure as above.

 Record the weights of each acid sample to the nearest 0.001 gram. For each titration, record the initial and final volumes of the buret to two decimal places.

## 2. Final calculations:

Knowing the volume and concentration of NaOH solution allows you to calculate the number of moles of base used to reach the end point. At the end point, the number of moles of base is equal to the number of moles of acid. Using this data and the weight of the sample you can calculate the molecular weight of the unknown acid to three significant figures. Discard and redo any samples that produce one percent (1%) or more error.

 In this step, you are asked to calculate the molecular weight of the an unknown acid.

### Example Calculation

2.134 g of a monoprotic acid was weighed out and dissolved in 100 mL of water. This sample was then titrated with 35.45 mL of a 0.150 M NaOH solution. What is the molecular weight of the acid? Remember, at the end point, the number of moles of NaOH equals the number of moles of monoprotic acid.

The equivalence point is when the solution in the flask has received just enough base to turn a very light pink which remains with mixing.

At the equivalence point there are equivalent numbers of moles of Hydrogen ions,  $H^+$ , and Hydroxide ions,  $OH^-$ .

$$\text{moles of } H^+ = \text{moles of } OH^-$$

If the concentration of the base and the volume needed to reach the equivalence point is known, then the number of moles of base added can be determined.

For this example:

$$\text{moles of } OH^- = (\text{Concentration of base}) \times (\text{Volume of base})$$

$$= 0.150 \frac{\text{moles}}{\text{Liter}} \times 0.03545 \text{ Liter}$$

$$\text{moles of } OH^- = 0.00532 \text{ moles}$$

$$\text{moles of } H^+ = \text{moles of } OH^-, \text{ (only at the equivalence point)}$$

$$\text{moles of } H^+ = 0.00532 \text{ moles}$$

The sample of acid weighed  
2.134 g.

And the molecular weight of the monoprotic acid is the mass per mole of acid

$$\text{MW of acid} = \frac{2.134 \text{ grams}}{0.00532 \text{ moles}} = 401 \text{ g/mole}$$

## Post Lab Assignment

Include the following information in a lab report to give to your TA:

1. Data and calculations from all six titrations.
2. The concentration of the standardized basic solution.
3. The molecular weight of your Assigned Unknown Acid.
4. The number of class results and the class average for your Assigned Unknown Acid.
5. Percentage deviation of your results from the class average result.

Access the "Results section" at the top of your **ICN Progress Page** and complete the following:

1. Choose and click the Submit button for your experiment.
2. Select your Assigned Unknown.
3. Enter your calculated numerical result. (In this case the Molecular Weight for your assigned Unknown Acid)
4. In the comments section enter the correct units for the numerical result.
5. Submit the above information.
6. When a statistically significant number of entries have been entered the class results will be displayed and the average of those results will be posted at the bottom.

Answer the following questions in your report

1. What is gained by weighing your samples by difference as opposed to using weighing paper?
2. After calculating the molecular weight for each run of an assigned acid and finding the average, determine if all of your values fall within 1% of each other and if not how far from the average they are. Present at least one reasonable explanation for an error greater than 1%.
3. If an air bubble is originally trapped in the buret tip but disappears during the titration, how does this affect the calculated molecular weight of acid in the unknown? Explain.
4. If the endpoint in the titration of KHP with NaOH is mistakenly passed (too pink), what effect does this have on the calculated molarity of the NaOH solution? Explain.

[http://cn2.univ.kh.ac.th/edu/newnav/newnavigator/Labs\\_Acid\\_1\\_792.html](http://cn2.univ.kh.ac.th/edu/newnav/newnavigator/Labs_Acid_1_792.html)

## Post Lab Assignment

Include the following information in a lab report to give to your TA:

1. Data and calculations from all six titrations.
2. The concentration of the standardized basic solution.
3. The molecular weight of your Assigned Unknown Acid.
4. The number of class results and the class average for your Assigned Unknown Acid.
5. Percentage deviation of your results from the class average result.

Access the "Results section" at the top of your **ICN Progress Page** and complete the following:

1. Choose and click the Submit button for your experiment.
2. Select your Assigned Unknown.
3. Enter your calculated numerical result. (In this case the Molecular Weight for your assigned Unknown Acid)
4. In the comments section enter the correct units for the numerical result.
5. Submit the above information.
6. When a statistically significant number of entries have been entered the class results will be displayed and the average of those results will be posted at the bottom.

Answer the following questions in your report

1. What is gained by weighing your samples by difference as opposed to using weighing paper?
2. After calculating the molecular weight for each run of an assigned acid and finding the average, determine if all of your values fall within 1% of each other and if not how far from the average they are. Present at least one reasonable explanation for an error greater than 1%.
3. If an air bubble is originally trapped in the buret tip but disappears during the titration, how does this affect the calculated molecular weight of acid in the unknown? Explain.
4. If the endpoint in the titration of KHP with NaOH is mistakenly passed (too pink), what effect does this have on the calculated molarity of the NaOH solution? Explain.