22 Ti TRATION: ANALYSIS OF SODIUM HYDROXIDE

In this experiment, you will learn the concept and technique of titration. You will use a chemical standard (potassium hydrogen phthalate) to determine the concentration of a sodium hydroxide (NaOH) solution.

Titration is a technique used to determine the concentration (molarity) of an unknown solution by allowing it to react with a solution of known concentration. For this experiment, you will determine the concentration a NaOH solution by neutralizing it with an acidic standard (potassium hydrogen phthalate, KHP).

In an acid-base titration, the titrant is dispensed slowly from a buret into a flask containing the analyte and the indicator. Since NaOH will be titrated from the buret, NaOH is the titrant. The indicator signals the reaction completion by changing colours when its environment switches between acidic and basic. In this experiment, phenolphthalein is used as indicator; it is colourless in acid and pink in base. As soon as a change in colour (a very faint pink) is observed (and remains permanent), titration is stopped. The endpoint, the point when the indicator changes colour, serves as a visually detectable close approximation to the equivalence point, the point when the amounts of acid and base are stoichiometrically equal. One must ensure that this approximation holds by carefully performing the titration, adding the titrant dropwise while closely watching for the colour change (endpoint). The last drop of the titrant added must be the drop that brought about the permanent change in colour. The concentration of the analyte is determined from the amount of titrant added required to reach the endpoint.

> It is important not to overshoot the end point. If you overshoot the endpoint, the solution will not be stoichiometrically equal and your calculated concentration will be incorrect.
>
> For this experiment, phenolphthalein changes from colourless to pink. **Once a very faint pink is observed, the endpoint is reached and you should stop the titration immediately.** If your solution turns bright pink, you have overshot the end point. Placing a piece of white paper under the flask helps detect the colour change.

When the titration is close to the endpoint, each drop of titrant will temporarily turn colour when it enters the solution, but the colour will quickly change back. At this point, you should be adding the titrant dropwise and waiting to see it the whole solution changes colour. If it does not, add another drop and repeat.

Standardization of NaOH

Since titration is a quantitative method, the concentration of the standard must be known with good certainty by standardization. To standardize the NaOH solution, it will be neutralized by **potassium hydrogen phthalate** ($KHC_8H_4O_4$, abbreviated KHP) in a titration analysis. KHP is a primary standard, being used to calibrate the standard NaOH solution. The equation for this acid-base reaction is:

$$1\ KHC_8H_4O_4\ (aq) + 1\ NaOH\ (aq) \rightarrow 1\ KNaC_8H_4O_4\ (aq) + 1\ H_2O\ (l)$$

A known mass of KHP ($KHC_8H_4O_4$) crystals is added to an Erlenmeyer flask then dissolved in water. Phenolphthalein is then added to the flask and it is colourless in acidic solution. The NaOH solution is dispensed from the buret until the endpoint is reached when the solution turns pink.

Consider 1.968 g of KHP (molar mass = 204.23 g/mol) dissolved in water requiring 20.06 mL of the NaOH solution to reach phenolphthalein endpoint. The concentration of the NaOH solution and the mass percent of NaOH in the basic solution is calculated as follows:

$$1.968\ \text{g KHP}\left(\frac{\text{mol KHP}}{204.23\ \text{g KHP}}\right) = 0.009636\ mol$$

Note: The number of moles of KHP does not change when dissolved in water. Whether the KHP was dissolved in 10 mL or 10 L of water, the number of moles would remain the same.

$$0.009636\ \text{mol KHP}\left(\frac{1\ \text{mol NaOH}}{1\ \text{mol KHP}}\right) = 0.009636\ \text{mol NaOH}$$

Note: From the balanced chemical equation, KHP and NaOH are 1:1 mole ratio.

$$\text{M} = \frac{0.009636\ \text{mol NaOH}}{0.02006\ \text{L solution}} = 0.4804\ \text{mol/L NaOH solution}$$

By standardization, it is now known that the concentration of the NaOH solution is 0.4804 M.

PROCEDURE

A. Preparation of the KHP Solution

1. Place a clean 125-mL Erlenmeyer flask on a weighing balance then press "tare" or "zero" to zero the readout.

2. Add a little more than 1 g KHP into the flask. Record the mass of the KHP.

3. Add 25 mL of distilled water into the flask, directing water to wash crystals that may be sticking to the walls of the flask.

4. Add a drop of phenolphthalein indicator to the flask.

> **Do not forget to add the indicator.** Without the indicator, you cannot see when you reached the endpoint.

5. Add a magnetic stir bar to the flask and stir the solution on a stirring plate. Note that you are using a combination heat/stir plate; **use only the stirring function**. Keep stirring as you prepare your buret in Part B allowing all the crystals to dissolve.

> IMPORTANT: Do not turn on the heat on the hot plate, only use the stirring function.

B. Preparation of the Buret

1. Obtain ~100 mL NaOH solution in a dry, clean 250 mL beaker. You will use this beaker (and a funnel if you find it necessary), to transfer NaOH solution to the buret. You may refill the beaker with NaOH as needed.

2. Before using a buret for analysis, it needs to be conditioned to minimize errors due to contaminants by rinsing the inside walls of the buret with the titrant, the NaOH solution. Start with the stopcock of the buret closed, or in horizontal position.

> Make sure the stopcock on the buret is closed (horizontal) when pouring the NaOH solution into the buret.
>
> Always fill the buret with a waste beaker under the tip to catch any overflow.

3. Place an empty 400 mL beaker under the tip of the buret and make sure the stopcock is closed (horizontal). Fill the buret halfway (~25 mL) with the titrant (NaOH solution), using a funnel. You may remove the buret from the stand to fill it, but make sure a waste beaker is still under it on the floor while you fill it. Empty the buret by slowly pouring out the solution into the waste beaker while turning the buret so that the solution makes contact with all the inner surface of the buret.

4. Clamp the buret to the stand, making sure it is perfectly vertical. Make sure the stopcock on the buret is closed (horizontal) when pouring the NaOH solution into the buret. Fill with NaOH solution just above the zero mark and place the waste beaker below the buret tip. Open the stopcock to vertical position by turning it 90°. Drain some solution to fill the buret tip, from below the stopcock to the very tip, making sure there are no air gaps or bubbles. You can gently tap the tip with your finger to help release air bubbles.

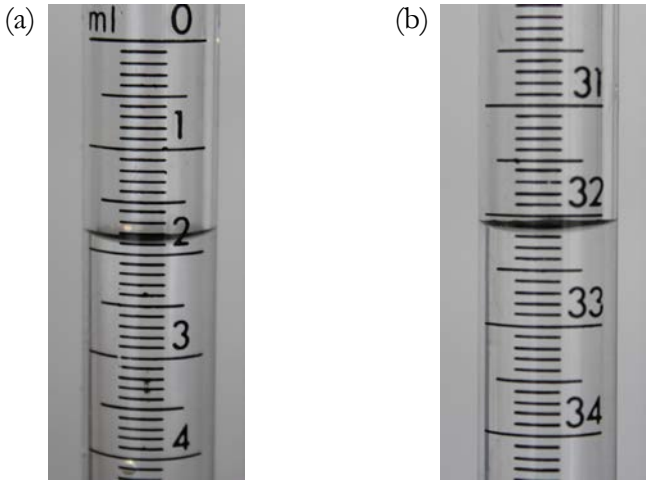
   Note that the stopcock can control how fast the solution flows between the horizontal and vertical positions. The buret is now ready for analysis. At this time, the liquid level should be below the zero mark, within the gradations. You do not need to start with the initial buret reading at zero.

C. Titration of the Standard NaOH Solution

1. Take the initial buret reading and record. The bottom of the meniscus of the NaOH should be between 0 mL and 1 mL. Do not start the buret exactly at 0 mL.

> You need to read the buret at eye level. This means elevating yourself so that your eyes are level with the top of the buret.

Reading the buret. A buret is read at the bottom of the meniscus at eye level. In a buret, the zero mark is at the top and the number markings count up going down towards the tip, as shown in Figure 2a. (Keep in mind that this is opposite to the graduated cylinder where zero is at the bottom because a buret is emptied from the bottom and a beaker is emptied from the top.) For example, the liquid level in Figure 1a is between 1 and 2 mL, but closer to 2 mL; the buret reading is 1.88 mL. **For practice, read the buret in Figure 1b.**



reading = 1.88 mL reading = _____

Volume of titrant used = _____

Figure 1. How to read a buret

The readings are not actual volumes. Buret readings are taken before and after dispensing the titrant during a titration analysis, so the volume of titrant used is the difference between the readings. **If Figures 1a and 1b show liquid levels at the beginning and at the end of a titration analysis, respectively, how many mL of the titrant is used? Ask your instructor to confirm your reading for Figure 1b and the volume of titrant dispensed.**

2. Make sure that all the KHP is dissolved before beginning the titration. This includes any crystals stuck to the sides of the Erlenmeyer flask. **Do not begin the titration until all the KHP has dissolved.**

3. Place the stir plate with the flask containing the KHP solution centered below the buret as shown in Figure 2. Make sure the solution is being stirred at a low speed (avoid splashing of the solution) and the **heat if OFF**. Stirring continues throughout the titration.

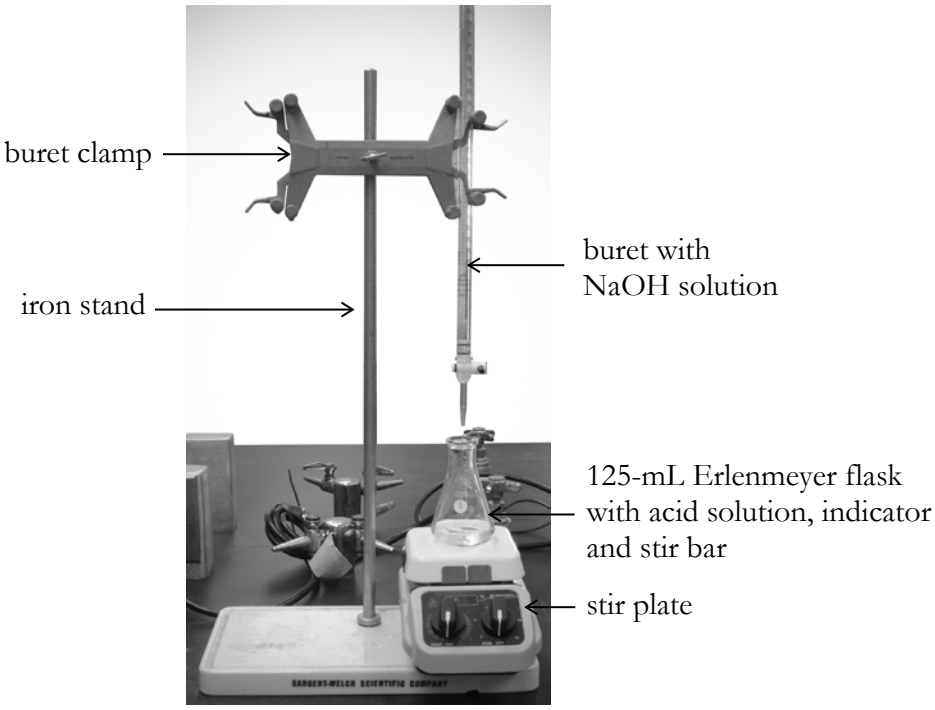


Figure 2. Titration Set-up

4. Open the stopcock to start delivering NaOH to the flask. Initially, the drops can be added quicker, but slow the flow when the pink colour begins to appear. When getting close to the endpoint, drops of the NaOH solution will turn pink when they first enter the KHP solution, then quickly turn colourless. Add the NaOH dropwise nearing the endpoint, making sure the pink colour disappears before adding the next drop.

5. When a drop of NaOH changes the solution from colourless to pink and stays pink with stirring, the endpoint is reached. Record the final buret reading.

> You are aiming for a very light pink, not a bright pink. If your solution is bright pink, you overshot the end point.
>
> You can place a white sheet of paper underneath your flask to help determine if your solution is pink or colourless.

If you emptied the entire buret of NaOH and there was no colour change, you probably forgot to add the phenolphthalein indicator.

Note: Do not allow the liquid level to go below the calibrations at the bottom of the buret. If this is about to occur, record a final reading. Refill the buret, record another initial reading and then another final reading when titration is completed. The total volume of NaOH used is the sum of the volumes used for the first and second sets of readings.

6. Perform two additional trials, using two more KHP solutions. Refill the buret with NaOH solution between each trial or when necessary.

7. Rinse the buret with water when all titrations are completed, drain through the tip into the waste beaker, and invert the buret in the clamp, with the stopcock in the open position (vertical).

8. Calculate the molarity of the NaOH solution.

CLEAN-UP

- Dispose of wastes in the large jug in the front hood.
- Wash all glassware used and return them to their appropriate places.
- Rinse the buret with water and drain by clamping it upside down.
- Return materials where they belong. Return the stir bar on top of the stir plate.

Name: _____ **Date:** _____

Partner's Name: _____

TITRATION: ANALYSIS OF SODIUM HYDROXIDE

Report all your measurements and answers in the correct number of significant figures and units.

	Trial 1	Trial 2	Trial 3
Mass of KHP			
Moles of KHP			
Volume of water added to KHP			
Moles of KHP (after water added)			
Concentration of KHP (Assume 25 mL of solution)			
Initial buret reading (NaOH)			
Final buret reading (NaOH)			
Volume of NaOH solution needed to neutralize KHP			
Moles of NaOH			
Concentration of NaOH solution			
Average concentration of NaOH			

Calculate the concentration of the standard NaOH solution for the three trials and report the results the table above. Show the complete calculations for Trial 1 only. Observe correct number of significant figures and units.

Trial 1 calculation for moles and concentration of KHP:

Trial 1 calculation for moles and concentration of NaOH:

POST-LAB EXERCISES

Show clearly the complete calculations with correct number of significant figures and units.

1. A solution of nitric acid is standardized using 0.851 g of sodium carbonate. Find the molarity of the nitric acid solution if 17.66 mL of it are required to reach the endpoint.

$$\_\_\ HNO_3\ (aq) + \_\_\ Na_2CO_3\ (aq) \rightarrow \_\_\ NaNO_3\ (aq) + \_\_\ H_2O\ (l) + \_\_\ CO_2\ (g)$$

2. Caltrate tablets contains calcium carbonate. If one tablet requires 34.46 mL of 0.291 M solution of hydrochloric acid for neutralization, how many milligrams of calcium carbonate is in this Caltrate tablet?

$$\_\_\ CaCO_3\ (s) + \_\_\ HCl\ (aq) \rightarrow \_\_\ CaCl_2\ (aq) + \_\_\ H_2O\ (l) + \_\_\ CO_2\ (g)$$