Objectives:

1. Standardize a solution of NaOH, using potassium hydrogen phthalate, KHP, as a standard.
2. Determine the purity of an impure sample of KHP, using the standardized solution of NaOH.

Standardization An analytical technique of accurately determining the concentration of a solution, by reacting it with a known amount of a known substance ("***standard****")*

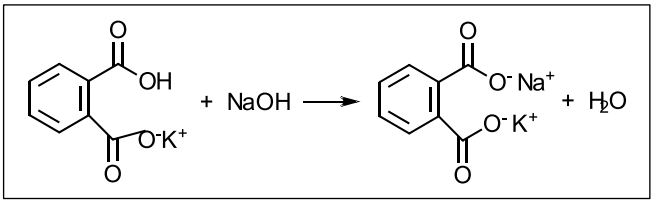
Titration An analytical technique of accurately determining the amount of a substance ("***analyte****")*, by reacting it with a standardized solution ("***titrant***").

In both cases, an ***indicator*** is used to signal the stoichiometric ***endpoint*** of the reaction.

Potassium Hydrogen Phthalate, **KHP**

Formula: $C_8H_5KO_4$

M.W. = 204.2g/mol



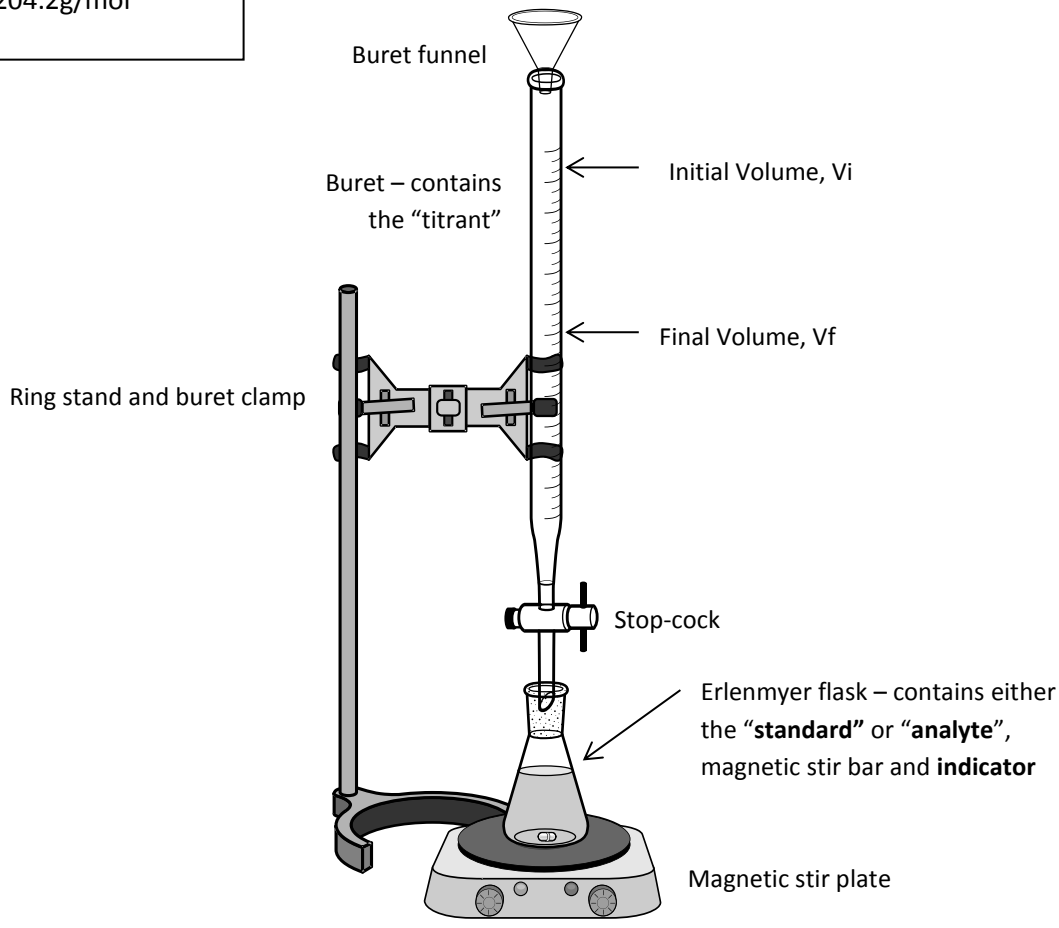


Figure 1 Typical titration apparatus.

Procedure 1 (Standardization of NaOH solution)

1. "Condition" a 50mL buret by repeatedly rinsing with small amounts (about 20mL) of 0.1M stock NaOH solution.
2. Assemble the titration apparatus (Figure 1) such that the "zero" of the buret scale is at the top.
3. Use the short buret funnel to fill the buret with 0.1M stock NaOH solution.
4. Open the stop cock momentarily to allow the solution to fill the entire tip of the buret.
5. Record the initial volume, Vi. It is not necessary (and a waste of time) to attempt to fill the buret exactly to the zero mark.
6. Weigh about 0.3g of KHP into a plastic weighing boat. Record the actual mass.
7. Transfer the KHP into a 250mL Erlenmeyer flask. Use a wash bottle with distilled water to rinse any residual KHP from the weighing boat into the flask.
8. Add about 30mL of distilled water to the flask.
9. Add a small magnetic stir bar to the flask.
10. Add 5 drops of phenolphthalein to the flask.
11. Use the magnetic stir plate to thoroughly stir and dissolve the KHP. Be sure to rinse down the sides of the flask and ensure that ALL of the KHP is in solution.
12. While continuing to stir the solution with the magnetic stir plate, use the stop cock to gradually add NaOH solution to the KHP solution until the endpoint is reached as indicated by a FAINT but PERSISTANT pink color. Use care not to exceed the endpoint. About 15 mL of NaOH solution should be required to reach the endpoint. So, quickly add about 12 mL, and then carefully approach the endpoint. When the endpoint is very near, a single additional drop of NaOH can cause a dramatic change in the indicator color, so be careful.
13. Read and record the final volume, Vf, from the buret.
14. Calculate the molarity of the NaOH solution (eq. 1).
15. Repeat 3-14 for a total of 4 runs.

$$Molarity\ of\ NaOH = \frac{(\quad)gKHP}{1} \times \frac{1\ mol\ KHP}{204.2g\ KHP} \times \frac{1\ mol\ NaOH}{1\ mol\ KHP} \times \frac{1}{(Vf-Vi)\ mL\ NaOH} \times \frac{1000mL}{1L} \qquad \textbf{eq. 1}$$

Table 1 Standardization of the NaOH titrant solution

Run #	Mass of KHP (g)	Vi (mL)	Vf (mL)	Molarity of NaOH Eq. 1 (M)
1				
2				
3				
4				
			Average	
			Std. Dev.	
			95% CI	

Procedure 2 (Purity of Unknown)

1. Use the short buret funnel to fill the buret with the NaOH solution.
2. Open the stop cock momentarily to allow the solution to fill the entire tip of the buret.
3. Record the initial volume, Vi. It is not necessary (and a waste of time) to attempt to fill the buret exactly to the zero mark.
4. Weigh about 0.5g of unknown into a plastic weighing boat. Record the actual mass and ***unknown number***.
5. Transfer the unknown into a 250mL Erlenmeyer flask. Use a wash bottle with distilled water to rinse any residual unknown from the weighing boat into the flask.
6. Add about 30mL of distilled water to the flask.
7. Add a small magnetic stir bar to the flask
8. Add 5 drops of phenolphthalein to the flask
9. Use the magnetic stir plate to thoroughly dissolve the unknown. Be sure to rinse down the sides of the flask and ensure that the entire unknown sample is in solution.
10. While continuing to stir the solution with the magnetic stir plate, use the stop cock to gradually add NaOH solution to the unknown solution until the endpoint is reached as indicated by a FAINT but PERSISTANT pink color.
11. Read and record the final volume, Vf, from the buret.
12. Calculate the mass of KHP in the unknown (eq. 2) and the purity of the unknown (eq. 3).
13. Repeat 3-12 for a total of 4 runs.

$$\text{Mass of KHP} = \frac{(\text{Vf}-\text{Vi})\ \text{mL NaOH soln}}{1000\text{mL/L}} \times \frac{(\ \text{Table 1 Average}\ )\ \text{mol NaOH}}{1\text{L NaOH soln}} \times \frac{1\text{mol KHP}}{1\ \text{mol NaOH}} \times \frac{204.2\text{g KHP}}{1\ \text{mol KHP}} \qquad \textbf{eq. 2}$$

$$\text{Purity of Unknown} = \frac{\text{Mass of KHP}}{\text{Mass of Unknown}} \times 100\% \qquad \textbf{eq. 3}$$

Table 2 Purity of Unknown # _____

Run #	Mass of Unknown (g)	Vi (mL)	Vf (mL)	Mass of KHP Eq. 2 (g)	Purity of Unknown Eq. 3 (%)
1					
2					
3					
4					
			Average		
			Std. Dev.		
			95% CI		