OBJECTIVES:

1. Standardize an aqueous solution of potassium permanganate, $KMnO_4$.
2. Determine the amount of sodium oxalate, $Na_2C_2O_4$, in an impure solid sample.

BACKGROUND

A **Titration** is the analytical method of carefully adding one solution (the "titrant") into another solution (the "analyte") until all of the analyte is consumed by the ensuing reaction.

Titrations are used in 2 analytical scenarios:

1. **Standardization of the titrant** – In this case, the amount of analyte is known and the concentration of titrant is determined.

2. **Analysis of the analyte** – In this case, the concentration of the titrant is known and the amount of analyte is determined.

Oxidation-Reduction ("redox") reactions involve the simultaneous oxidation (loss of electrons) and reduction (gain of electrons) between reactants.

Potassium permanganate, $KMnO_4$, and sodium oxalate, $Na_2C_2O_4$, will undergo a redox reaction with each other in an acidic aqueous solution.

The balanced **net ionic equation** is:

$$16\ H^+ + 2\ MnO_4^- + 5\ C_2O_4^{2-} \rightarrow$$

$$2\ Mn^{2+} + 10\ CO_2 + 8\ H_2O$$

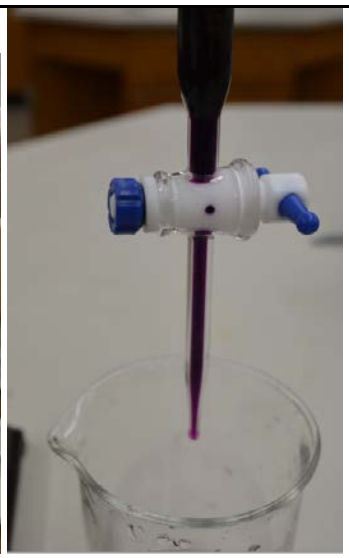
The balanced **molecular equation** is:

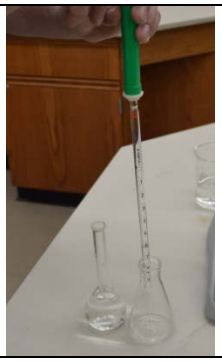
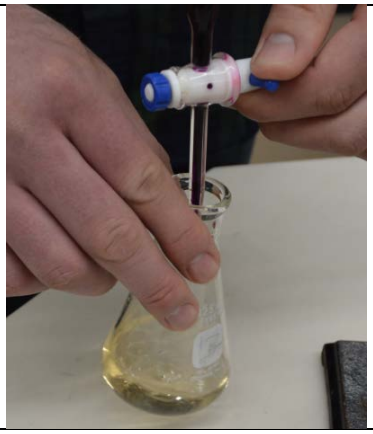
$$8\ H_2SO_4 + 2\ KMnO_4 + 5\ Na_2C_2O_4 \rightarrow$$

$$2\ MnSO_4 + 10\ CO_2 + K_2SO_4 + 5\ Na_2SO_4 + 8\ H_2O$$

In this experiment, an aqueous solution of potassium permanganate will be the titrant, and pure sodium oxalate will be used as the analyte.

PROCEDURE 1 – Preparation and Standardization of potassium permanganate titrant solution.

1. To a 500mL beaker or Erlenmyer flask … a. Add 100 mL 0.130 M $KMnO_4$ b. Add 200 mL DI water c. Stir for a minute d. Label as "**TITRANT**"	 
2. Prepare a 50 mL burette with titrant. a. Rinse burette well with DI water b. Set up burette in stand c. Flush burette with about 10 mL of titrant d. Fill burette with titrant (stay below the end of the funnel). e. Be sure tip is filled.	 (d)  (e)
3. To a 250 mL beaker … a. Add about 2 g of sodium oxalate. Record actual mass (Table 1) b. Rinse any remaining sodium oxalate into the beaker with a few mL of DI water.	 (a)  (b)

4. Add about 50 mL 3 M H_2SO_4 5. Gently heat and stir without boiling, until the sodium oxalate is entirely dissolved	 (4)  (5)
6. Transfer the sodium oxalate solution to a 100 mL volumetric flask. Add 3 M H_2SO_4 until the total volume of the solution is 100 mL. Use a plastic eye dropper when you get close to the 100 mL line. Label this solution as "**STANDARD**".	 
7. Use a 10 mL graduated pipet to transfer 10 mL of the standard into a small Erlenmeyer flask. Heat the flask to about 70-90°C. **Do NOT boil**.	 
8. While the standard solution is still hot, titrate to a persistent faint pink end point. Record Vi and Vf (Table 1).	 
9. Repeat Steps 7 & 8 for a total of 3 good trials.	

PROCEDURE 2 – Determination of the amount of sodium oxalate in an unknown sample.

1. To a 250 mL Erlenmyer flask … a. Add about 0.6 g of unknown. Record unknown sample # and mass (Table 2) b. Add about 20 mL of 3 M H_2SO_4	
2. Gently heat and stir without boiling, until the sample is entirely dissolved.	
3. Heat the flask to about 70-90°C. **Do NOT boil**. Titrate to a persistent faint pink end point. Record Vi and Vf (Table 2)	
4. Repeat Steps 1-3 for a total of 3 good trials.	

Table 1 – Standardization of $KMnO_4$ Titrant Solution (mass of pure $Na_2C_2O_4$ = _____g)

Trial	Vi (mL)	Vf (mL)	V = Vf - Vi (mL)	[$KMnO_4$] (M)
1				
2				
3				
			Average =	
			Std. Dev. =	
			95% CI =	

Table 2 – Determination of weight % of $Na_2C_2O_4$ in unknown sample # _____

Trial	Mass of unknown (g)	Vi (mL)	Vf (mL)	V (mL)	Wt % $Na_2C_2O_4$
1					
2					
3					
				Average =	
				Std. Dev. =	
				95% CI =	

CALCULATIONS

Part 1 – Standardization of $KMnO_4$ Titrant Solution

Procedure 1
Step 3

$$[KMnO4] = \frac{(\quad)\ g\ Na_2C_2O_4}{100\ mL\ sol'n} \times \frac{10\ mL\ sol'n}{1} \times \frac{1\ mol\ Na_2C_2O_4}{133.96\ g\ Na_2C_2O_4} \times \frac{2\ mol\ KMnO_4}{5\ mol\ Na_2C_2O_4} \times \frac{1}{(\quad)\ L\ KMnO_4 sol'n}$$

V = Vf-Vi
Procedure 1
Step 8
(convert mL to L before substituting into the calculation)

Part 2 – Determination of weight % of $Na_2C_2O_4$ in unknown sample

AVG
$[KMnO_4]$
Table 1

Procedure 2
Step 3
(convert mL to L before substituting into the calculation)

$$\text{wt\% } Na_2C_2O_4 = \frac{(\quad)mol\ KMnO_4}{1\ L\ KMnO_4\ sol'n} \times \frac{(\quad)L\ KMnO_4\ sol'n}{1} \times \frac{5\ mol\ Na_2C_2O_4}{2\ mol\ KMnO_4} \times \frac{133.96\ g\ Na_2C_2O_4}{1\ mol\ Na_2C_2O_4} \times \frac{100\%}{(\quad)g\ unknown\ sample}$$

Procedure 2
Step 1