

# REDOX TITRATION

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## EXPERIMENT

# 30

### Apparatus

25 mL Buret	10 mL pipet
rubber bulb	hot plate / stirrer
magnetic stir bar	3 - 250 mL Erlenmeyer flasks
50 mL volumetric flask	25 mL graduated cylinder
100 mL graduated cylinder	

### Chemicals

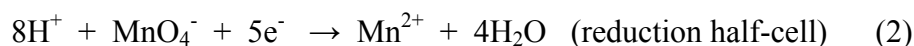
Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub> , sodium oxalate	KMnO <sub>4</sub> , potassium permanganate
6 M H <sub>2</sub> SO <sub>4</sub>	Unknown Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub> solution

### APPARATUS AND CHEMICALS

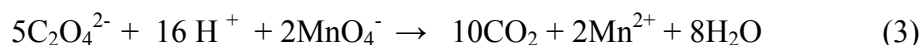
Oxidation is defined as the loss of electrons producing a more positive product. The transfer of electrons represents the simplest kind of oxidation – reduction reaction, a reaction frequently referred to as a redox reaction. In such a reaction the number of electrons lost must equal the number of electrons gained. Atoms of different elements may lose or gain varying numbers of electrons; therefore the oxidation or reduction capacity of any atom or ion must be calculated for a particular reaction. Knowing that information you can determine the stoichiometric relationship between the reactants.

### DISCUSSION

Sodium oxalate and potassium permanganate react in an acid medium according to the following half-cell reactions:



The two half-cell reactions are combined to obtain the complete redox reaction:



From this balanced equation, you can see that there is a 5:2 stoichiometric ratio between the moles of oxalate and the moles of permanganate.

To facilitate the reduction of  $\text{Mn}^{+7}$  (purple) to  $\text{Mn}^{+2}$  (colorless), the reaction must be carried out in an acidic medium through the addition of  $\text{H}_2\text{SO}_4$ . Without the  $\text{H}_2\text{SO}_4$ , the reaction may stall at  $\text{Mn}^{+4}$ , resulting in the production of  $\text{MnO}_2$  (greenish-brown). If this should occur, the reaction would not proceed further and would have to be repeated using the proper conditions.

In today's exercise you will use the relationship between sodium oxalate and potassium permanganate to determine the molar concentration of a permanganate solution. You will then use the standardized permanganate solution to determine the molar concentration of an unknown oxalate solution.

## PROCEDURE

### A. Preparation of Standard $\text{Na}_2\text{C}_2\text{O}_4$ Solution

Weigh approximately 0.5 grams of  $\text{Na}_2\text{C}_2\text{O}_4$ . Transfer the weighed sample to a 50 ml volumetric flask. Dissolve the  $\text{Na}_2\text{C}_2\text{O}_4$  in about 30 ml of deionized water. Make sure the  $\text{Na}_2\text{C}_2\text{O}_4$  is completely dissolved. Once all the  $\text{Na}_2\text{C}_2\text{O}_4$  is dissolved, dilute the solution to the mark with deionized water. Mix thoroughly. Transfer the 50 ml of  $\text{Na}_2\text{C}_2\text{O}_4$  solution to an Erlenmeyer flask and label.

### B. Standardization of $\text{KMnO}_4$ Solution

Obtain in a clean dry beaker approximately 75 ml of  $\text{KMnO}_4$  solution to be standardized. Rinse the buret with two 1 ml portions of the  $\text{KMnO}_4$  solution. Fill the buret with the solution making sure no air remains in the tip (the tip must also be filled with  $\text{KMnO}_4$  solution). Record the buret reading.

Pipet 10.00 ml of the standard  $\text{Na}_2\text{C}_2\text{O}_4$  solution into a 250 ml flask. Add approximately 60 ml of distilled water and 12 ml of 6.0 M  $\text{H}_2\text{SO}_4$ . Heat the solution to almost boiling. (**NOTE: The reaction is slow and requires heat to proceed: the hotter the better, but do not allow the solution to boil.**) Titrate slowly the  $\text{KMnO}_4$  solution into the  $\text{Na}_2\text{C}_2\text{O}_4$  solution until a faint pink color remains for at least 30 seconds. Record the final buret reading. Perform a second titration.

### C. Determination of Unknown $\text{Na}_2\text{C}_2\text{O}_4$

Obtain an unknown sample of the  $\text{Na}_2\text{C}_2\text{O}_4$  from the instructor. Pipet 10 ml of unknown into a 250 mL Erlenmeyer flask, add 60 ml of deionized water, 12 ml of 6 M  $\text{H}_2\text{SO}_4$  heat and titrate with  $\text{KMnO}_4$  as before. Perform a second titration.

## REVIEW QUESTIONS

1. REDOX is an acronym for what type of reaction?
2. Define the term oxidizing agent; reducing agent.

3. What is the oxidizing reagent in today's exercise? the reducing agent?
4. What is the oxidation state of the manganese before and after the reaction?
5. What is the name of the laboratory technique used in today's exercise?
6. Why is it not necessary to use an indicator in this reaction?  
How will you know when the reaction is complete?

Name \_\_\_\_\_ Desk \_\_\_\_\_

Date \_\_\_\_\_ Laboratory Instructor \_\_\_\_\_

**REPORT SHEET**  
**REDOX TITRATION**

**EXPERIMENT**

**30**

Show ALL calculations on separate paper and attach to report sheet.

**A. Preparation of Standard Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> Solution**

Mass Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> \_\_\_\_\_ g

Moles Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> \_\_\_\_\_ mol

Volume of Solution \_\_\_\_\_ mL

Molarity of Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> \_\_\_\_\_ M

**B. Standardization of KMnO<sub>4</sub>**

	Trial 1	Trial 2
Final buret reading (KMnO <sub>4</sub> )	_____ mL	_____ mL
Initial buret reading (KMnO <sub>4</sub> )	_____ mL	_____ mL
Volume KMnO <sub>4</sub> used	_____ mL	_____ mL
Volume Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub> titrated	_____ mL	_____ mL
Molarity of KMnO <sub>4</sub>	_____ M	_____ M
Average Molarity of KMnO <sub>4</sub>	_____ M	

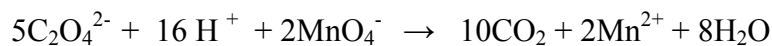
**C. Determination of Unknown Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>**

	Trial 1	Trial 2
Volume Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	_____ mL	_____ mL
Final buret reading (KMnO <sub>4</sub> )	_____ mL	_____ mL
Initial buret reading (KMnO <sub>4</sub> )	_____ mL	_____ mL
Volume (KMnO <sub>4</sub> ) used	_____ mL	_____ mL
Molarity of unknown Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	_____ M	_____ M
Average Molarity of unknown Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	_____ M	

Show ALL calculations on separate paper and attach to report sheet.

**QUESTIONS**

1. Consider the balanced ionic equation for the reaction in today's exercise:



The MnO<sub>4</sub><sup>-</sup> salt used is KMnO<sub>4</sub>, the oxalate salt is Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> and the acid is H<sub>2</sub>SO<sub>4</sub>. The Mn<sup>+2</sup>, K<sup>+</sup>, and Na<sup>+</sup> all react to form three different sulfate salts. Based on these facts, write the balanced molecular equation for the reaction.

2. Why is it not necessary to know accurately the volume of H<sub>2</sub>SO<sub>4</sub> used in the reaction but is necessary to accurately know the volumes of KMnO<sub>4</sub> and Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> solutions?

Name \_\_\_\_\_

3. If the equation for a redox reaction is balanced, will free electrons appear in the equation as either reactants or products? Explain your answer.
4. For every mole of  $\text{MnO}_4^-$  reduced how many moles of  $\text{CO}_2$  are produced?
5. What would have happened if you forgot to put in the sulfuric acid when you ran the reaction?

6. Label the oxidizing/reducing agents in the following reactions. Label them as OX or RED.

