REDOX TITRATION

-University of South Alabama,
T. Gerald Jackson, Josephine VanHook,
and Margaret E. Jackson
Edited by: Patricia M. Davis and Diane H. Roe

EXPERIMENT

30

APPARATUS AND CHEMICALS

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

$Na_2C_2O_4$, sodium oxalate	$KMnO_4$, potassium permanganate
6 M H_2SO_4	Unknown $Na_2C_2O_4$ solution

DISCUSSION

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.

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

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e^- \quad \text{(oxidation half-cell)} \qquad (1)$$

$$8H^+ + MnO_4^- + 5e^- \rightarrow Mn^{2+} + 4H_2O \quad \text{(reduction half-cell)} \qquad (2)$$

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

$$5C_2O_4^{2-} + 16\,H^+ + 2MnO_4^- \rightarrow 10CO_2 + 2Mn^{2+} + 8H_2O \qquad (3)$$

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 Mn^{+7} (purple) to Mn^{+2} (colorless), the reaction must be carried out in an acidic medium through the addition of H_2SO_4. Without the H_2SO_4, the reaction may stall at Mn^{+4}, resulting in the production of 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 $Na_2C_2O_4$ Solution

Weigh approximately 0.5 grams of $Na_2C_2O_4$. Transfer the weighed sample to a 50 ml volumetric flask. Dissolve the $Na_2C_2O_4$ in about 30 ml of deionized water. Make sure the $Na_2C_2O_4$ is completely dissolved. Once all the $Na_2C_2O_4$ is dissolved, dilute the solution to the mark with deionized water. Mix thoroughly. Transfer the 50 ml of $Na_2C_2O_4$ solution to an Erlenmeyer flask and label.

B. Standardization of $KMn0_4$ Solution

Obtain in a clean dry beaker approximately 75 ml of $KMnO_4$ solution to be standardized. Rinse the buret with two 1 ml portions of the $KMnO_4$ solution. Fill the buret with the solution making sure no air remains in the tip (the tip must also be filled with $KMnO_4$ solution). Record the buret reading.
Pipet 10.00 ml of the standard $Na_2C_2O_4$ solution into a 250 ml flask. Add approximately 60 ml of distilled water and 12 ml of 6.0 M H_2SO_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 $KMnO_4$ solution into the $Na_2C_2O_4$ 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 $Na_2C_2O_4$

Obtain an unknown sample of the $Na_2C_2O_4$ 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 H_2SO_4 heat and titrate with KMnO4 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 | **EXPERIMENT**

REDOX TITRATION | 30

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

A. Preparation of Standard $Na_2C_2O_4$ Solution

Mass $Na_2C_2O_4$ ___________g

Moles $Na_2C_2O_4$ ___________mol

Volume of Solution ___________mL

Molarity of $Na_2C_2O_4$ ___________M

B. **Standardization of $KMnO_4$**

	Trial 1	Trial 2
Final buret reading ($KMnO_4$)	__________mL	__________mL
Initial buret reading ($KMnO_4$)	__________mL	__________mL
Volume $KMnO_4$ used	__________mL	__________mL
Volume $Na_2C_2O_4$ titrated	__________mL	__________mL
Molarity of $KMnO_4$	__________M	__________M
Average Molarity of $KMnO_4$	__________M	

C. Determination of Unknown $Na_2C_2O_4$

	Trial 1	Trial 2
Volume $Na_2C_2O_4$	_________mL	_________mL
Final buret reading ($KMnO_4$)	_________mL	_________ mL
Initial buret reading ($KMnO_4$)	_________ mL	_________mL
Volume ($KMnO_4$) used	_________mL	_________mL
Molarity of unknown $Na_2C_2O_4$	_________M	_________M
Average Molarity of unknown $Na_2C_2O_4$	_________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:

$$5C_2O_4^{2-} + 16\,H^+ + 2MnO_4^- \rightarrow 10CO_2 + 2Mn^{2+} + 8H_2O$$

The MnO_4^- salt used is $KMnO_4$, the oxalate salt is $Na_2C_2O_4$ and the acid is H_2SO_4. The Mn^{+2}, K^+, and Na^+ 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_2SO_4 used in the reaction but is necessary to accurately know the volumes of $KMnO_4$ and $Na_2C_2O_4$ 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 MnO_4^- reduced how many moles of 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.

a. $Mn^{2+}_{(aq)} + NaBiO_{3(s)} \rightarrow Bi^{3+}_{(aq)} + MnO_4^-{}_{(aq)}$

______ ______

b. $Cu_{(s)} + NO_3^-{}_{(aq)} \rightarrow Cu^{2+}_{(aq)} + NO_{2(g)}$

______ ______

c. $Cr_2O_7^{2-}{}_{(aq)} + Cl^-{}_{(aq)} \rightarrow Cr^{3+}_{(aq)} + Cl_{2(g)}$

______ ______

.