

8—Oxidation-Reduction Titration



Name: _____

Date: _____

Section: _____

Lab Professor: _____

Objectives

- Develop skills in the reliable measurement of mass and volume
- Learn proper use of analytical balance for mass determination
- Learn proper use of buret for volume determination
- Learn and reinforce the concepts of titration as an analytical method
- Determination of significant figures for laboratory measurement methods
- Evaluation of uncertainty of measurements
- Calculation the average, deviation, average deviation and relative average deviation
- Develop skills with substances that may be hazardous
- Gain experience with stoichiometry in balanced reactions
- Observe a reduction oxidation process
- Understand quantitative analysis

Pre-Laboratory Requirements

- Read Section 21.1 and review sections 4.5-4.6 in Silberberg.
- Pre-Lab Questions (if required by your instructor)
- Laboratory Notebook—prepared before lab (if required by your instructor)

Safety Notes

- Eye protection must be worn at all times!!!
- Sulfuric acid is a corrosive, dangerous acid – your instructor will provide special safety instructions for its use and handling. Disposable gloves are available.

Discussion

In this experiment, you will perform a titration for quantitative analysis. In past lab experiments you may have performed titrations based on acid-base reactions. Stoichiometry for the acid base titrations was most likely 1:1 with an indicator dye used to find an equivalence volume when a color change occurred. However in this lab experiment, you will perform titrations for an oxidation-reduction reaction (often called “redox” reaction) and will find that the stoichiometry is not 1:1 and that the reaction is self-indicating; that is, there is no indicator needed.

Potassium permanganate (KMnO_4) is a common chemical found in most laboratories. It can be used for a variety of purposes and although hazardous in concentrated solutions it is relatively safe in small concentrations. It is best known for its deep purple color that can be seen with the naked eye at low concentrations [note: if you are color blind, you must inform your instructor for assistance with this lab

experiment]. It dissolves in water as a strong electrolyte to give K^+ and MnO_4^- ions. Manganese is in the +7 oxidation state (the highest) in $KMnO_4$. Therefore it can only be reduced (gain electrons) to lower oxidation states in redox reactions. Although it is possible to make +4, +3, 0 and other oxidation states, the most common reaction is a five electron reduction to +2; that is Mn^{2+} which occurs as a hydrated ion in water. The reduction half reaction is:



The half reaction requires acid (H^+) to make water, and the acid speeds up the reaction. It is not an “acid-base” reaction in the strict sense as there is no neutralization. Yet the above half reaction cannot occur without a complimentary oxidation half reaction. There are many choices but one of the best is based on a chemical called sodium oxalate ($Na_2C_2O_4$). This compound has weak base properties, and in strong acid it is immediately converted to oxalic acid ($H_2C_2O_4$) but that’s not what we are interested in for this experiment. The sodium form dissolves to give 2 Na^+ and one oxalate ($C_2O_4^{2-}$): a trio of ions. The oxidation half reaction may then be written as:



The two half reactions have different numbers of electrons, five for the reduction and two for the oxidation. The rules for balancing redox reactions include that electron must be canceled out when adding and stoichiometry are done. If the first reaction is multiplied by 2 and the second by 5, then the two are added together we get the balanced, overall redox reaction:



This reaction tells us that five oxalate ($C_2O_4^{2-}$) and two permanganate (MnO_4^-) on a mole basis go together to make products in the presence of a lot of acid (H^+)!

A titration is a process of combining two liquids – a titrant and an analyte – in a manner so that stoichiometric equivalence is achieved. When that occurs the following equation must be true for the above overall redox reaction:

$$2 \text{ mol permanganate} = 5 \text{ mol oxalate}$$

This equation tells us that if you want to convert moles of permanganate into moles of oxalate (stoichiometry) you must multiply by 5/2. In previous lab experiments you learned that molarity (M) times volume (L) gives mol. Suppose you make a solution that contains a known molarity of permanganate. Put that in a buret, deliver it into an oxalate solution until something happens that tells you that the reaction stoichiometry has been met. Then read the volume delivered and you may calculate the moles of oxalate from:

$$(5 \text{ mol oxalate} / 2 \text{ mol permanganate}) \times M(MnO_4^-) \times V(MnO_4^-) = \text{mol oxalate } (C_2O_4^{2-})$$

Experimental Overview

In this experiment, you will use a pre-prepared solution of $KMnO_4$. The lab assistants have dissolved solid $KMnO_4$, then filtered it to remove impurities and undissolved solid. You will have two solids available for use: pure 100% $Na_2C_2O_4$ which will be your “standard” and impure $Na_2C_2O_4$ which will be your “unknown.” The staff will know the actual weight percent of $Na_2C_2O_4$ in your unknown and your mission will be to find out what that is by careful measurement as follows. Be sure to write down any identification on the unknown bottle, otherwise we won’t know the composition for grading.

Permanganate solutions stain glassware and require stringent cleaning before and after use. If you see a brown material form, that is most likely MnO_2 which is hard to remove. Be sure to clean stopcocks and tips of burets and thoroughly rinse all glassware with water. Sulfuric acid (H_2SO_4) that contains a small amount of MnSO_4 as a catalyst is used to provide acid in the above reaction. Be extra careful as it can badly burn skin and damage eyes. If you get some on your hands, etc, rinse immediately with water, then tell the instructor. Heat will be generated in parts of this lab when solutions are mixed.

The first part of this lab is known as standardization. The permanganate solution has an unspecified concentration and you must find out what it is. The second part of this lab is the analysis of the unknown using the “standardized” permanganate. If for any trial you observe brown precipitate formation, you have made a mistake and must discard and redo the trial.

This titration is fun because it is self-indicating. Permanganate will first form a pink purple solution that is readily seen with the naked eye when it is added to the oxalate solution until reaction occurs. It will then react and disappear. This will continue until you have added enough to achieve equivalence. One drop after equivalence will give the solution a tint of color. If this color persists for more than a minute, there is no more oxalate left to react.

Good titration technique includes rinsing the sides of the receiving vessel, thorough and continuous mixing and making sure that the solid is initially totally dissolved. You will see CO_2 bubbles form as reaction occurs.

Experimental Procedure

Part I. Standardization of Prepared Permanganate Solution

1. Weigh 0.250 g of $\text{Na}_2\text{C}_2\text{O}_4$ into a 250 mL beaker or flask (you do not need to be exact, just know exactly what it is, e.g. 0.245 grams; Range 0.230-0.270 grams).
2. Dissolve this salt in about 100 mL of DI water, and acidify the solution with 25 mL of 3 M H_2SO_4 (Be Careful!).
3. Fill the buret to the zero line and make sure there is no air bubble in the tip.
4. Titrate the solution with the prepared permanganate solution, remembering to stir constantly (**Note: be sure not to add more than 1 mL of solution before stopping and waiting for the solution to clear with each addition**).
5. Stop the titration when the solution becomes slightly colored (the purple pink first appears) and the color lasts a minute or longer after thorough mixing.
6. Record the volume delivered to the nearest 0.01 mL.
7. If your solution turns a permanent muddy brown, this is an indication of MnO_2 formation and you should discard the entire trial and start again.
8. Calculate the mol $\text{Na}_2\text{C}_2\text{O}_4$ for each mass used from its molecular weight.
9. Convert mL to L for the observed volume used in the titration
10. Calculate the molarity of the KMnO_4 titrant and record in your notebook and the data table below.
11. Repeats steps 3-8 at least two more times. If you have poor results discard the data and do another trial.

Part I:

	Trial 1	Trial 2	Trial 3
Mass of $\text{Na}_2\text{C}_2\text{O}_4$ (g)			
Moles of $\text{Na}_2\text{C}_2\text{O}_4$			
Initial buret reading (mL)			
Final buret reading (mL)			
Total volume used (mL)			
Total volume used (L)			
Molarity of KMnO_4 solution			
Average molarity (M)			
Relative average deviation in M			

Calculations:

Part II. Analysis of an Unknown Oxalate

1. Weigh 0.750 g of the unknown solid into a 250 mL beaker (you do not need to be exact, just know exactly what it is, e.g. 0.755 grams; Range – 0.730 to 0.770 grams)
2. Follow the same procedure (steps 3-8 above) as for the standardization to calculate the mass and percentage of Na₂C₂O₄ in the unknown sample
3. Your instructor will check calculations and give you instructions on clean up and waste disposal.

Part II: Unknown number or letter ID: _____

	Trial 1	Trial 2	Trial 3
Initial buret reading (mL)			
Final buret reading (mL)			
Total volume used (mL)			
Total volume used (L)			
Moles of KMnO ₄			
Moles of Na ₂ C ₂ O ₄ in unknown			
Mass of Na ₂ C ₂ O ₄ in unknown (g)			
Mass of unknown (g)			
Percent of Na ₂ C ₂ O ₄ in unknown			
Average percent of Na ₂ C ₂ O ₄ in unknown			
Relative average deviation			

Calculations: