Experiment 5: Determining the Stoichiometry and Products of a Redox Reaction

Reading: Chapter sections 4.4-4.6 and 20.1-20.2 in your course text and this lab handout

Ongoing Learning Goals:

- To use a scientific notebook as a primary record of procedures, data, observations, and example calculations
- To make scientific measurements
- To present your formal results through a laboratory report along with proper citations
- To use Excel to tabulate, calculate, analyze, and graph scientific data
- To apply balanced chemical equations and stoichiometric relationships to quantitative measurements
- To relate measured chemical properties to the reactions of chemical species
- To evaluate the uncertainty (error) in scientific measurements, and understand the causes of the underlying uncertainty

Additional Learning Goals for Experiment 5:

- To perform titrations that quantitatively relate to unknown chemical processes
- To understand and balance oxidation-reduction reactions

Introduction

A titration is a process in which a solution of known concentration is mixed with a solution of unknown concentration and a specific chemical reaction between the two reactants is carried just to completion. The point at which the titration reaction is complete, with no excess of either reactant, is called the **equivalence point**. Effective titrations require accurate methods for measuring solution volumes, and detecting the "end point" of the reaction. The "end point" of the reaction is an observable change in a measurable quantity of the reaction solution, often a color change. The "end point" (observable change) should be very close to the equivalence point at which an exactly stoichiometric amount of the known reactant has been added.

You performed titrations last week in the laboratory to determine the relationship between conductivity and concentration in aqueous solutions. This week you will use another type of titration to explore the stoichiometry of the reaction of potassium permanganate ($KMnO_4$) with iron (II). In this titration, you will quantitatively add a potassium permanganate solution (**the titrant**) to an iron (II) solution (**the analyte**), but instead of monitoring the reaction throughout the titration, you will only need to measure the volume added to get to the end point (*very* close to the **equivalence point**). You will be able to visually determine this end point from a faint but persistent pink color due to the presence of a trace amount ($< 2 \times 10^{-6}$ M MnO_4^-) of unreacted permanganate (Figure 1). *Your titration will allow you to determine the stoichiometry of the reaction, and from that stoichiometry you will be able to determine the identities of the reaction products and the specific redox reaction (out of 4 possibilties) that has taken place.*

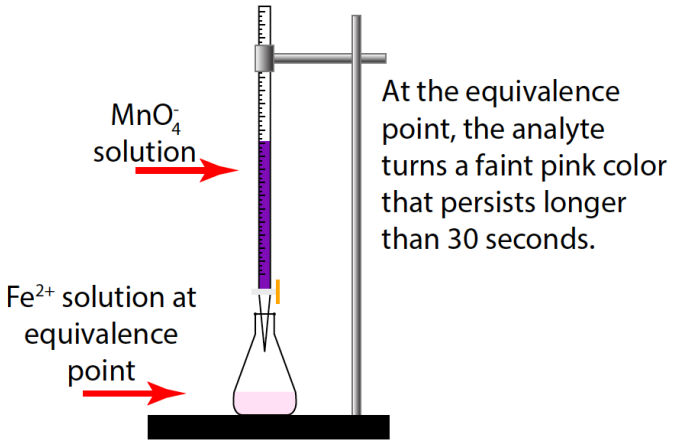

Figure 1. Diagram of the titration assembly. The MnO_4^- solution is quantitatively added from a buret to the Fe^{2+} solution in an Erlenmeyer flask.

Procedure for experiment

You will independently prepare the analyte solution and perform replicas of the titration to achieve a high degree of precision.

A. **Preparation of the 0.01 M $KMnO_4$ titrant**: Into a weigh bottle, approximately weigh the previously determined mass of $KMnO_4$ (from your prelab) to make 250.0 mL of a 0.010 M solution. All masses should be recorded to 4 decimal places. Using the deionized water bottle, transfer the $KMnO_4$ into a 250-mL volumetric flask using a long-stemmed funnel. Carefully rinse the weigh bottle into the flask several times with deionized water. Bring the volume up to the mark, adding the last few milliliters with a Pasteur pipet to avoid going over the mark. Put in the stopper, and turn the flask up and down several times to thoroughly mix.
B. **Weigh a sample of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$:** Weigh about 0.5 g of iron (II) ammonium sulfate hexahydrate into a weigh boat. Record mass to 4 decimal places. Transfer this sample into a 200 or 250 mL Erlenmeyer flask. Add approximately 25 mL of 0.5 M sulfuric acid and 25 mL of 2.0 M phosphoric acid. Add a stir bar, and turn on the stir plate. Make sure all crystals have dissolved.
C. **Titrate sample to a faint pink endpoint:** Rinse the buret with about 3 mL of the titrant added using the small plastic funnel, then fill the buret. Flush bubbles from the buret tip. Remove any drops from the tip. Record the initial volume (from the bottom of the meniscus) to hundredths of a mL. Insert the tip of the buret well inside the neck of the Erlenmeyer flask. Add titrant until the appearance of a very faint pink color that persists. As you approach this endpoint, the pink color will begin to persist for longer periods of time, before disappearing. Once any pink shows, it is advisable to add titrant slowly. Partial drops can be added by rinsing the buret tip and the wall of the flask.
D. **Perform replicate experiments:** Do two more titrations and check your percent error. Use fresh samples of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ each time. Calculate the percent error in the mole ratio. If percent error is not less than 2%, perform a fourth titration. See the Error Analysis document for help calculating percent error from replicas.

Data Analysis

Following the template on the next page of this lab handout, create an Excel spreadsheet containing all the mass, volume, and molar relationships from your experimental data. Keep in mind that you first need to determine the average ratio for the moles of Fe^{2+} to the moles of MnO_4^-. Once you have determined this molar ratio for all your trials, you will be able to calculate values for error/uncertainty. In addition, your molar ratio rounded to the nearest whole number will serve to determine which of the four potential redox reactions (from your pre-lab assignment) most likely corresponds to the chemistry taking place during your titration.

What should be in your laboratory notebook?

1. Calculate the concentration of your $KMnO_4$ solution from the exact mass of $KMnO_4$ you weighed out.
2. All masses and volumes (initial, final, and difference) for each titration.
3. Attach the Excel spreadsheet you created with all the required information as shown on the following page.

Laboratory report: Use the **Report Form** for Experiment 5.

Excel data table to create for your Experiment 5 Report

Measured mass of $KMnO_4$, g _____

Calculated molarity of $KMnO_4$, M _____

Trial	**$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ mass, g**	**Fe^{+2} moles***	**$KMnO_4$ volume, mL***	**MnO_4^- moles***	**$Fe^{2+} : MnO_4^-$ molar ratio***
1					
2					
3					

Average molar ratio: _____:_____

Standard deviation of molar ratio: _____

Standard deviation of the mean (SDOM) of molar ratio*: _____

Percent error (also called percent relative error) of molar ratio*: _____

Percent precision of molar ratio*: _____

Theoretical molar ratio from one of the four possible redox reactions: _____:_____

% Accuracy*: _____

***Show example calculation in your lab notebook**

Reminder:

$$\textit{Percent accuracy} = 100 - \frac{|\textit{theoretical value} - \textit{experimental value}| * 100}{\textit{theoretical value}}$$

An Example Analysis

The approach used in this experiment can be illustrated with a parallel example using perchlorate ion, ClO_4^-, as the oxidizing agent. In the titration of Fe^{2+} with ClO_4^-, the two possible chlorine-containing products are Cl_2 and Cl^-. The possible half reactions are shown as equations 1 to 3 and the balanced equations for the two possible reactions are equations 4 and 5 (verify the balancing of these reactions as practice). From these balanced reactions, we can see that the stoichiometry of the reaction in terms of moles Fe^{2+} to moles ClO_4^- can be used to determine the products of the reaction: 7:1 for Cl_2 as the product or 8:1 for Cl^- as the product.

$Fe^{2+} \rightarrow Fe^{3+} + 1e^-$ oxidation half-reaction (1)

$ClO_4^- \rightarrow 1/2\ Cl_{2\ (g)}$ reduction half-reaction (2)

or

$ClO_4^- \rightarrow Cl^-$ reduction half-reaction (3)

Adding reaction (1) [oxidation] to either reaction (2) or (3) [possible reductions] yields:

$7\ Fe^{2+} + ClO_4^- + 8\ H^+ \rightarrow 7\ Fe^{3+} + 1/2\ Cl_{2\ (g)} + 4\ H_2O$ (4)

or

$8\ Fe^{2+} + ClO_4^- + 8\ H^+ \rightarrow 8\ Fe^{3+} + Cl^- + 4\ H_2O$ (5)

To determine the actual stoichiometry, the titration experiment was carried out. A carefully weighed sample of 0.3532 g of ferrous sulfate $FeSO_4 \cdot 7H_2O$ (F.W. 278.03 g/mol) was titrated with a 0.01062 M solution of $KClO_4$. The endpoint was reached when 14.99 mL of $KClO_4$ was added. This result was used to determine the stoichiometry of the reaction as shown below. The resulting stoichiometry of 8:1 indicates that the product of the reaction was Cl^- and for every mole of ClO_4^-, 8 electrons were transferred.

Problem Solving: (keeping at least one extra significant figure and rounding at the end)

The number of moles of Fe^{2+} is:

$$0.3532\ \text{g of } FeSO_4 \cdot 7H_2O \left(\frac{1 mole}{278.03 g}\right) = 1.2704 \times 10^{-3}\ \text{mol } Fe^{2+}$$

The number of moles of ClO_4^- added is:

$$14.99\ \text{mL of } KClO_4 \left(\frac{1\text{L}}{1000\text{mL}}\right) \left(\frac{0.01062\text{mol}}{\text{L}}\right) = 1.5919 \times 10^{-4}\ \text{mol } ClO_4^-$$

The ratio of Fe^{2+} to ClO_4^- is:

$$\frac{\text{moles } Fe^{2+}}{\text{moles } ClO_4^-} = \frac{1.2704 \times 10^{-3} \text{ mol}}{1.5919 \times 10^{-4} \text{ mol}} = 7.980$$

The stoichiometry is, therefore, 8:1, so reaction (5) is the correct reaction.