Lab 4: Iron Redox Titration

Before lab (week 1):

1. Prepare your lab notebook and data tables
2. Examine MSDS sheets on website: $KMnO_4$

Week 2:

1. Do Week 1 calculations
2. Prepare your lab notebook and data tables

Introduction

Oxidation-reduction ("redox") titration is a type of titration based on the loss or gain of electrons between the analyte and the titrant. The endpoint of this titration is where the # of equivalence of the reducing agent is equal to the number of equivalence of the oxidizing agent.

Potassium permanganate, $KMnO_4$, is a strong oxidizing agent. Permanganate, MnO_4^-, is an intense dark purple color. Reduction of purple permanganate ion to the colorless Mn^{+2} ion, the solution will turn from dark purple to a faint pink color at the equivalence point. No additional indicator is needed for this titration. The reduction of permanganate requires strong acidic conditions.

In this experiment, permanganate will be reduced by oxalate, $C_2O_4^{2-}$ in acidic conditions. Oxalate reacts very slowly at room temperature so the solutions are titrated hot to make the procedure practical. The unbalanced redox reaction is shown below.

$$MnO_4^- + C_2O_4^{2-} \rightarrow Mn^{2+} + CO_2 \quad \text{(acidic solution)}$$
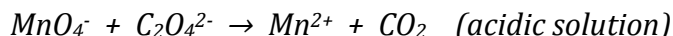
Based on this reaction above, a potassium permanganate solution can be standardized against a sample of potassium oxalate; therefore the exact *normality** (eq/L) of the permanganate solution can be determined. This is very useful because now the standardized permanganate solution can be used to find the concentrations of iron (II) in an unknown solution. The unbalanced redox reaction for the determination of an unknown iron (II) concentration is shown below.

$$MnO_4^- + Fe^{2+} \rightarrow Mn^{2+} \ Fe^{3+} \quad \text{(acidic solution)}$$
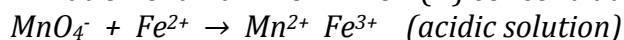
This lab will be split up into two parts: standardizing the potassium permanganate solution (Day 1) and determining the concentration of Fe(II) in an unknown solution (Day 2).

*This lab utilizes a chemical term, *normality*, to describe the concentration of a solution. Normality is a useful scale to describe the transfer of electrons in a redox reaction. The units of normality are *# of equivalents/liter*, which for this lab can be related to the # of electrons/liter.

Procedure

Week 1: Prepare and Standardize 0.01 N $KMnO_4$ Stock Solution

On an analytical balance, weigh approximately 0.1 g $KMnO_4$ crystals on a piece of weighing paper. Add the crystals to a 500 mL Erlenmeyer flask.

3. Add approximately 350 mL of distilled water to the flask.
4. Heat the solution with occasional swirling to dissolve the $KMnO_4$ crystals. Do not boil the solution. This may take about 30 minutes.
5. Allow the solution to cool and stopper with a rubber stopper. You will need this solution for both day 1 and day 2.

6. On weighing paper, weigh approximately 0.05 g of potassium oxalate ($K_2C_2O_4 \cdot H_2O$) on the analytical balance. Record the exact mass to 4 decimal places. Transfer the sample to a 250 mL Erlenmeyer flask.
7. Rinse and fill the buret with the $KMnO_4$ solution.
8. Add 50 mL of distilled water and 5 mL of 6 N H_2SO_4 to the oxalate sample in the Erlenmeyer flask. Swirl to dissolve the solids.
9. Heat the acidified oxalate solution to about 85°C. Do not boil the solution.
10. Record the initial buret reading. Because the $KMnO_4$ solution is strongly colored, the top of the meniscus may be read instead of the bottom.
11. Titrate the hot oxalate solution with the $KMnO_4$ solution until the appearance of a very faint pink color.
12. Record the final buret reading and calculate the volume of $KMnO_4$ used in the titration.
13. Discard the titration mixture in the appropriate waste container in the fume hood as instructed by the TA and repeat the titration with a new sample of oxalate for a total of 3 trials.
14. An oxalic acid solution may be used to wash the buret and the titration flask if a brown stain remains in the glassware.

Week 2: Quantifying Fe²⁺ in unknown solutions
1. Pipet a 25 mL sample of the unknown Fe(II) solution into a 250 mL Erlenmeyer flask.
2. Add 50 mL of distilled water and 12 mL of 6 N H_3PO_4 into the flask.
3. Fill a buret with the standard KMnO4 solution and record the initial buret reading.
4. Titrate the sample with the standard $KMnO_4$ to a faint pink end-point and record the final buret reading. Calculate the volume of $KMnO_4$ used.
5. Discard the Fe solution in the appropriate waste container as instructed by the TA and repeat the titration with a new sample of the unknown Fe (II) solution for a total of 4 trials.
6. When finished with all trials, discard the purple permanganate solution in the appropriate waste container in the fume hood.
7. Oxalic acid may be used to remove any brown stains left on the glassware.

Discussion Question
What is the largest source of random error in these measurements? And what could be done to minimize it?

Lab write-up
1. **Abstract:** Background, purpose, method, results, what it means! A 250-word trash can!
2. **Introduction:** Give a brief introduction to the problem being addressed. Be sure to cite any materials you use.
3. **Results:** Briefly discuss your results in text, shown in figures and tables:
4. **Discussion:** respond to the questions above in paragraph form (not Q1: answer). Devote 1-2 paragraphs to discuss each question from week 2 and 1-2 paragraphs for the whole class data.
5. **Conclusion:** Write a short paragraph concluding what you learned.
6. **References**
7. **Group dynamics:** let me know how you feel your group worked together.
8. **Appendix 1- Calculations:** Send a NEAT and ORGANIZED spreadsheet to instructor.

Week 1: Calculations

1. Using the half-reaction method, write a balanced redox equation for the reaction of permanganate with oxalate in an acidic solution.

2. Calculate the equivalent weight of the oxalate reducing agent from the molar mass of the oxalate sample and the equivalence of electrons lost by the reducing agent in the oxidation half-reaction.

3. Use the sample mass that you weighed out of the oxalate solid and the equivalent weight to calculate the number of equivalents of oxalate in each sample.

4. At the equivalence point, the equivalence of the reducing agent is equal to the equivalence of the oxidizing agent.

5. Using the equivalence of the oxidizing agent from the step before, and the volume of permanganate solution you used in the titration, calculate the normality of the KMnO4 solution.

6. Calculate the average normality of the permanganate solution for the four trials.

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Week 2: Calculation of the concentration of the unknown Fe solution

1. Using the half-reaction method, balance the redox reaction of permanganate with iron(II) in acidic media.

2. Calculate the equivalence of $KMnO_4$ titrated.

3. At the equivalence point, the equivalence of the oxidizing agent is equal to the equivalence of the reducing agent.

4. Determine the normality of the ferric reducing agent.

5. Calculate the molarity (mol/L) of the ferrous solution

6. Calculate the mass concentration (mg/L, aka 'ppm') of iron in the unknown solution by multiplying the molar mass of iron by the molarity of the ferrous solution.

7. Calculate the average mass concentrations for the ferrous unknown solutions for the four trials.