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Oxidation–Reduction Titrations
AP Chemistry Laboratory #8

Introduction

A common task in analytical chemistry is the determination of the amount of a substance present in a sample or product. If the product contains a substance that can be oxidized, then it is possible to determine the number of moles of that substance by titrating the sample with a solution of a strong oxidizing agent. In this lab, an oxidizing solution will be standardized and then use to determine the number of moles of oxalic acid, a reducing agent.

Concepts

- Oxidation–reduction reaction
- Titration
- Half-reaction
- Equivalence point

Background

Oxidation–reduction reactions occur by electron transfer. The balanced chemical reaction can be written as the combination of two *half-reactions,* representing the oxidation reaction and the reduction reaction, respectively.

For example: If solid iron is placed in a solution of gold(III) ions, the gold(III) ions are reduced to solid gold and the iron oxidized to iron(III) ions, according to the following half-reactions:

$$Fe(s) \rightarrow Fe^{3+}(aq) + 3e^- \qquad \textit{oxidation half-reaction}$$

$$Au^{3+}(aq) + 3e^- \rightarrow Au(s) \qquad \textit{reduction half-reaction}$$

$$Fe(s) + Au^{3+}(aq) \rightarrow Au(s) + Fe^{3+}(aq) \qquad \textit{oxidation–reduction reaction} \qquad \textit{Reaction 1}$$

In this experiment, potassium permanganate, $KMnO_4$, is used as the oxidizing agent. In an acidic solution, the MnO_4^- ion is reduced from Mn(VII) to Mn(II) according to the following half-reaction:

$$8H^+(aq) + MnO_4^-(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l) \qquad \textit{Half-Reaction 2}$$

In Part 1, a solution of $KMnO_4$ is standardized by titration with a solution containing a known concentration of iron(II) ions, (Fe^{2+}).

In the corresponding oxidation half-reaction, the Fe^{2+} ion is oxidized to Fe^{3+}:

$$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^- \qquad \textit{Half-Reaction 3}$$

Combining half-reactions 2 and 3 and balancing the number of electrons transferred gives the overall reaction equation:

$$8H^+(aq) + MnO_4^-(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 5Fe^{3+}(aq) + 4H_2O(l) \qquad \textit{Reaction 4}$$

The balanced equation shows that 5 moles of Fe^{2+} are required to react with 1 mole of MnO_4^-.

For this redox titration, the *equivalence point* occurs when the exact number of moles of Fe^{2+} ions has been added to react completely with all the MnO_4^- ions in solution. At this point:

$$\text{moles } Fe^{2+} = 5 \times (\text{moles } MnO_4^-) \qquad \textit{Equation 1}$$

If the volume and molarity of the Fe^{2+} solution are known, then:

$$V_{Fe^{2+}}M_{Fe^{2+}} = 5\, V_{MnO_4^-}M_{MnO_4^-} \qquad \textit{Equation 2}$$

Rearranging Equation 2 yields the equation for the concentration of the potassium permanganate solution.

$$M_{MnO_4^-} = \frac{(V_{Fe^{2+}})(M_{Fe^{2+}})}{5\,(V_{MnO_4^-})}$$

The indicator for this titration is the MnO_4^- ion itself. The MnO_4^- ion is purple in solution. At the endpoint of the titration, the solution changes from light pink to colorless.

In Part 2, the concentration of an oxalic acid solution is determined by titration with the permanganate solution standardized in Part 1. In this case, the endpoint occurs when the pink color of the MnO_4^- ion persists. The half-reaction for the oxidation of oxalic acid is:

$$2H_2O(l) + H_2C_2O_4(aq) \rightarrow 2H_2CO_3(aq) + 2H^+(aq) + 2e^- \qquad \textit{Half-Reaction 5}$$

The oxidation state of carbon changes from (+3) in $H_2C_2O_4$ to (+4) in H_2CO_3.

Experiment Overview

The purpose of this lab is to standardize a solution of potassium permanganate by redox titration with a standard solution of iron(II) ions. A solution of oxalic acid is then titrated with the permanganate solution to determine the exact concentration of oxalic acid.

Pre-Lab Questions

1. Write the balanced net ionic equation for the reaction between MnO_4^- ions and $H_2C_2O_4$ in acid solution.

2. How many moles of Fe^{2+} ions can be oxidized by 0.043 moles of MnO_4^- ions?

3. 1.630 g of iron ore is dissolved in an acidic solution. This solution is titrated to a pink endpoint with 27.15 mL of a 0.020 M $KMnO_4$ solution.
   a. How many moles of MnO_4^- ions were consumed?
   b. How many moles of Fe^{2+} were in the iron ore sample?
   c. What is the percent of iron in the iron ore sample?

Materials

Buret, 50-mL	Ring stand
Erlenmeyer flasks, 250-mL, 3	Buret clamp
Hot plate	Potassium permanganate, $KMnO_4$, ≈0.02 M, 100 mL
Thermometer	Ferrous ammonium sulfate, $Fe(NH_4)_2SO_4 \cdot 6H_2O$, 0.100 M, 50 mL
Volumetric pipet, 10-mL	Oxalic acid solution, $H_2C_2O_4$, 60 mL
Volumetric pipet, 25-mL	Sulfuric acid, H_2SO_4, 6 M, 50 mL
Beakers, 100-mL, 3	Manganese sulfate, $MnSO_4 \cdot H_2O$, 1.0 M, 5 mL
Graduated cylinder, 10-mL	Water, distilled or deionized
Wash bottle	

Safety Precautions

Sulfuric acid (6 M) is corrosive to eyes, skin, and other tissue; always add acid to water, never the reverse. Potassium permanganate solution may be a skin irritant. The oxalic acid solution is a skin and eye irritant; it is moderately toxic by ingestion. The manganese sulfate solution is a body tissue irritant. Wear chemical splash goggles and chemical-resistant gloves and apron. Wash hands thoroughly with soap and water before leaving the laboratory.

Procedure

Part 1. Standardization of a Potassium Permanganate Solution

1. Obtain approximately 80 mL of the potassium permanganate solution in a 100-mL beaker. Obtain 50 mL of the 0.100 M ferrous ammonium sulfate solution in another 100-mL beaker. Label both beakers.
2. Set up a clean, 50-mL buret in the ring stand and buret clamp.
3. Rinse the buret with approximately 10 mL of distilled or deionized water and then with two 5 mL portions of the MnO_4^- solution (potassium permanganate solution, $KMnO_4$).
4. Close the stopcock and fill the buret to above the zero mark with MnO_4^- solution.
5. Open the stopcock to allow any air bubbles to escape from the tip. Close the stopcock when the liquid level is between the 0- and 10-mL marks.
6. Record the precise level of the solution in the buret in the Part 1 Data Table. This is the initial volume of the MnO_4^- solution. (See Figure 1 for reading buret level.)
7. With the volumetric pipet, transfer 10 mL of the 0.100 M Fe^{2+} solution to a clean 250-mL Erlenmeyer flask. Record this volume in the Part 1 Data Table.
8. Measure out 10 mL of the 6 M H_2SO_4 into a clean 10-mL graduated cylinder and add this to the Erlenmeyer flask. Swirl to mix.

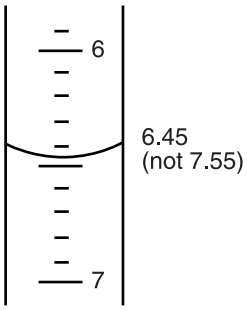

Figure 1.

Procedure

9. Position the flask under the buret so that the tip of the buret is within the flask but at least 2 cm above the liquid surface.

10. Titrate the ferrous ammonium sulfate solution with the MnO_4^- solution until the first trace of pink color persists for 30 seconds. Remember to swirl the flask and to rinse the walls of the flask with distilled water before the endpoint is reached.

11. Record final buret reading as the final volume of the MnO_4^- solution in the Part 1 Data Table.

12. Repeat the standardization titration two more times.

Part 2. Determination of Concentration of an Oxalic Acid Solution

1. Obtain approximately 60 mL of the oxalic acid solution in a clean 100-mL beaker.

2. With a 25-mL volumetric pipet, transfer 25 mL samples of the oxalic acid solution to each of two clean 250-mL Erlenmeyer flasks. Record the volume in the Part 2 Data Table.

3. Add 5 drops of the 1.0 M $MnSO_4$ solution to each flask. *(The Mn^{2+} ion acts as a catalyst for the reaction.)*

4. Measure out 10 mL of 6 M H_2SO_4 into a graduated cylinder and add this amount to each of the 250-mL Erlenmeyer flasks. Add 20 mL of distilled water to each flask and swirl.

5. Warm the first flask to about 85 °C on the hot plate.

6. Immediately titrate this solution with the standardized MnO_4^- solution from Part 1. Record both the initial and final buret readings in the Part 2 Data Table.

7. Repeat steps 5 and 6 with the second flask.

Disposal

Your instructor will provide disposal instructions.

Data Tables

Part 1

Molarity of Fe^{2+} _____ M

	Trial 1	Trial 2	Trial 3
Volume of Fe^{2+} solution titrated	mL	mL	mL
Initial volume of MnO_4^- solution	mL	mL	mL
Final volume of MnO_4^- solution	mL	mL	mL
Volume of MnO_4^- added	mL	mL	mL

Part 2

Molarity of MnO_4^- solution _____ M

	Trial 1	Trial 2
Volume of $H_2C_2O_4$ solution titrated	mL	mL
Initial volume of MnO_4^- solution	mL	mL
Final volume of MnO_4^- solution	mL	mL
Volume of MnO_4^- added	mL	mL

Molarity of $H_2C_2O_4$ solution _____ M

Post-Lab Calculations

1. From the Part 1 standardization data, calculate the molarity of the MnO_4^- solution for each trial. Average the values and enter the average in the Part 2 Data Table.
2. From the Part 2 titration data, calculate the molarity of the $H_2C_2O_4$ solution for each trial. Average the values and enter the average in the Part 2 Data Table.
3. How many moles of Fe^{2+} ions and MnO_4^- ions were titrated in each Part 1 trial?
4. How many moles of oxalic acid, $H_2C_2O_4$ were titrated in each Part 2 trial?