

USE BLUE/BLACK INK!!!!

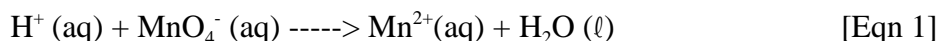
Goal:

The goal of this lab is to determine the molarity of an oxalic acid solution by titrating it with a standard potassium permanganate solution.

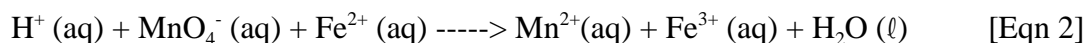
Introduction/Background:

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, a solution of an oxidizing agent will first be standardized and then used to determine the number of moles of oxalic acid, a reducing agent.

In acidic solution, manganese is reduced according to the following unbalanced half-reaction:



During the standardization part of the lab, ferrous ammonium sulfate will be oxidized to iron (III) ions in acidic solution according to the following unbalanced equation:



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

$$\text{moles Fe}^{2+} = (\text{mole ratio of Fe}^{2+} \text{ to MnO}_4^- \text{ from equation})(\text{moles of MnO}_4^- \text{ in solution})$$

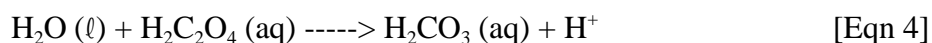
If the volume and molarity of the Fe^{2+} solution are known, then

$$V_{\text{Fe}^{2+}} \times M_{\text{Fe}^{2+}} = (\text{mole ratio of Fe}^{2+} \text{ to MnO}_4^- \text{ from eqn}) \times V_{\text{MnO}_4^-} \times M_{\text{MnO}_4^-} \quad [\text{Eqn 3}]$$

Instead of phenolphthalein or some other acid-base indicator, the indicator for this titration is the permanganate ion itself. The permanganate ion is purple (violet) in solution. At the endpoint of the titration, the solution changes from light pink to colorless.

In the second part of the lab, the concentration of an oxalic acid solution will be determined by titration with the standardized potassium permanganate solution. In this case, the endpoint occurs when the pink color of the permanganate ion persists.

The unbalanced half-reaction for the oxidation of oxalic acid is



NOTE: This lab will take two days.

(c) What is the percent of iron in the iron ore sample?

6) List the names and formulas for all the chemicals that will be used during both days of the lab:

7) (a) What is a catalyst? _____

(b) What catalyst will be used during this lab? _____

8) Summarize what will happen and/or the goal of Day 1: _____

9) Summarize what will happen and/or the goal of Day 2: _____

Materials - Day 1:

50 mL 0.100 M ferrous ammonium sulfate

50 mL 6 M H₂SO₄

2 50 mL burets

1 ringstand

1 10 mL graduated cylinder

1 test tube brush

2 250 mL beakers (for cleaning and waste)

100 mL approx. 0.02 M KMnO₄

2 100 mL beakers

1 double buret clamp

3 250 mL Erlenmeyer flasks

1 permanent marker

2 buret funnels

1 30 mL beaker

Materials - Day 2:

2 100 mL beakers

2 250 mL Erlenmeyer flasks

1 10 mL graduated cylinder

2 250 mL beakers (for cleaning and waste)

1 30 mL beaker

1 25 mL graduated cylinder

1 thermometer
5 mL 1.0 M MnSO_4
60 mL approx. 0.02 M KMnO_4

1 hot plate
60 mL 0.25 M oxalic acid

Hazards:

The student safety contract applies. _____

Procedure - Day 1:

1. Clean beakers, flasks, and the graduated cylinder
2. Label glassware
 - a. label one 100 mL beaker KMnO_4 and initials
 - b. label another 100 mL beaker $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ and initials
 - c. label the first buret KMnO_4
 - d. label the second buret $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$
 - e. label the 30 mL beaker H_2SO_4 and initials
 - f. label two 250 mL beakers waste
3. Obtain solutions
 - a. obtain approximately 80 mL of potassium permanganate solution in a 100 mL beaker
 - b. obtain approximately 50 mL of ferrous ammonium sulfate solution in a 100 mL beaker
 - c. obtain approximately 10 mL of sulfuric acid in a 30 mL beaker
3. Set up two 50 mL burets
4. Prepare the burets
 - a. use the 250 mL beakers for waste
 - b. rinse the burets with about 10 mL of distilled water
 - c. rinse the first buret twice with about 5 mL of potassium permanganate solution
 - d. rinse the second buret twice with about 5 mL of ferrous ammonium sulfate solution
5. Fill the burets
 - a. fill the first buret to the zero mark with potassium permanganate solution
 - b. fill the second buret with the remaining ferrous ammonium sulfate solution
6. Using the second buret, transfer approximately 10 mL of ferrous ammonium sulfate solution into the first Erlenmeyer flask
 - a. record the starting volume of ferrous ammonium sulfate
 - b. record the ending volume of ferrous ammonium sulfate

7. Acidify the solution
 - a. measure out 10 mL of 6 M H_2SO_4 into the graduated cylinder
 - b. add it to the Erlenmeyer flask
 - c. swirl to mix
8. Titrate the ferrous ammonium sulfate
 - a. position the Erlenmeyer flask under the buret of potassium permanganate so that the tip of the buret is in the flask but at least 2 cm above the surface of the liquid
 - b. slowly add potassium permanganate solution until the first trace of pink color persists for 30 seconds
 - c. while adding the permanganate, swirl the flask and wash down the walls of the flask with distilled water
 - d. record the final buret reading
9. Repeat steps 6-8 twice more for a total of three trials
10. Clean up
 - a. empty both burets into a 250 mL beaker
 - b. fill the ferrous ammonium sulfate buret with distilled water
 - c. drain into waste beaker
 - d. add hydrogen peroxide to the potassium permanganate buret
 - e. drain into waste beaker
 - f. rinse the potassium permanganate buret with distilled water again

Procedure - Day 2:

1. Clean the beakers, Erlenmeyer flasks, and graduated cylinder
2. Label
 - a. label a 100 mL beaker oxalic acid and initials
 - b. label a 100 mL beaker potassium permanganate and initials
 - c. label a 30 mL beaker sulfuric acid and initials
2. Obtain solutions
 - a. obtain about 60 mL of oxalic acid solution in a 100 mL beaker
 - b. obtain about 60 mL of potassium permanganate solution in a 100 mL beaker
 - c. obtain about 20 mL of 6 M H_2SO_4 in a 30 mL beaker
3. Set up two 50 mL burets
4. Prepare the burets
 - a. use the 250 mL beakers for waste
 - b. rinse the burets with about 10 mL of distilled water
 - c. rinse the first buret twice with about 5 mL of oxalic acid solution
 - d. rinse the second buret twice with about 5 mL of potassium permanganate solution
5. Fill the burets
 - a. fill the first buret with oxalic acid solution
 - b. fill the second buret with potassium permanganate solution
6. Measure oxalic acid
 - a. Record starting volume of oxalic acid
 - b. Transfer 25 mL of oxalic solution to the first Erlenmeyer flask
 - c. Record ending volume of oxalic acid
 - d. Repeat steps a-c for the second Erlenmeyer flask
7. Add 5 drops of manganese sulfate solution to each flask (to act as a catalyst)
8. Acidify the solution
 - a. measure out 10 mL of 6 M H_2SO_4 into the graduated cylinder
 - b. add it to the first Erlenmeyer flask
 - c. add 20 mL of distilled water to the flask
 - d. swirl to mix

- e. repeat steps a-d for the second flask
9. Warm the first flask to 85 °C on the hot plate
10. Titrate the solution in the flask with the potassium permanganate solution until the pink color of the solution persists
11. Repeat steps 9 and 10 on the second flask.
12. Clean up
 - a. empty both burets into a 250 mL beaker
 - b. fill the oxalic acid buret with distilled water
 - c. drain into waste beaker
 - d. add hydrogen peroxide to the potassium permanganate buret
 - e. drain into waste beaker
 - f. rinse the potassium permanganate buret with distilled water again

Data:

Data Table I (Day 1)

	Trial 1	Trial 2	Trial 3
Initial volume of Fe ²⁺			
Final volume of Fe ²⁺			
Volume of Fe ²⁺ used			
Initial volume of MnO ₄ ⁻			
Final volume of MnO ₄ ⁻			
Volume of MnO ₄ ⁻ added			

Data Table II (Day 2)

	Trial 1	Trial 2
Initial volume of oxalic acid		
Final volume of oxalic acid		
Volume of oxalic acid used		
Initial volume of MnO ₄ ⁻		
Final volume of MnO ₄ ⁻		
Volume of MnO ₄ ⁻ added		

****RECORD ADDITIONAL DATA ON A SEPARATE SHEET****

Post-lab calculations (answer on a separate sheet and attach; no need for a conclusion):

- 1) Calculate the molarity of the ferrous ammonium sulfate solution from Day 1
- 2) Use the day 1 data and Eqn 3 from the introduction/background to calculate the molarity of the potassium permanganate solution. Calculate the molarity for each trial and report the average.
- 3) Calculate the molarity of the oxalic acid solution for each trial on Day 2 using Eqn 3 (but replacing Fe²⁺ with oxalic acid). Calculate the molarity for each trial and report the average.

Lab handout based on the experiment "Oxidation-Reduction Titrations" in Laboratory Experiments for Advanced Placement Chemistry (Second Edition) by S.A. Vonderbrink (Flinn Scientific, 2006)