

Introduction

As you know, one common type of reaction in chemistry is oxidation-reduction. It involves the transfer of electrons from one species to another. A species undergoes oxidation when a loss of electrons occurs, and a substance undergoes reduction when it gains electrons. These two processes always occur simultaneously because there must be a transfer of electrons from one species to another. A *reducing agent* is a species that causes reduction by donating the electrons to another species, causing its oxidation number to decrease. Therefore, a reducing agent is itself always oxidized and its own oxidation number is always increased. Conversely, an *oxidizing agent* causes oxidation in another species by accepting that species' electrons, allowing oxidation (and an increase in oxidation number) of the other species. Therefore the oxidizing agent is itself always reduced and its own oxidation number decreases.

There are many different types of oxidation-reduction reactions that you are already familiar with. They include single replacement reactions, in which a substance in elemental form is a reactant or a product: combustion reactions, some synthesis reactions, and some decomposition reactions (never precipitation reactions nor acid base neutralizations). In this lab we will work another type of redox reaction that occurs in solution. In these type of redox reactions atoms will not be found in there elemental state, but instead some metal cations will increase their oxidation number, while other elements in common oxidizing agents will be reduced. One common oxidizing agent that will be used in this lab is the permanganate ion, MnO_4^- . In particular, we will use potassium permanganate, but throughout the lab, the potassium ion will be a spectator ion.

Procedure Preview

First in this lab, you need to titrate a solution of iron(II) ions with a known number of moles against a permanganate ion solution whose concentration you need to determine in order to use in Parts 2 & 3. You will have a general idea of the concentration, but you'll need to do this initial titration to confirm or *standardize* the solution. In this lab, you will standardize the potassium permanganate solution by titrating it into a solution of iron(II) sulfate heptahydrate.

Next in this lab, you will take a commercial iron tablet and make a solution by crushing the tablet, putting it into solution, then titrating it with the standardized potassium permanganate solution to quantify the actual amount of iron in the tablet and comparing it to the manufacturer's claim.

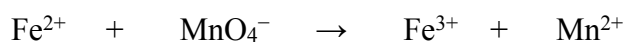
Finally, you will titrate the standardized permanganate solution with hydrogen peroxide to analyze a commercial product in order to determine the actual amount of hydrogen peroxide in the solution sold in the brown bottle in the store.

Pre-LAD - This must be done before class and this page will be turned in.

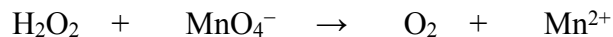
1. Read the Procedure and Processing the Data and then make a Data/Results Table -- one side/page for each of the three Procedures so you will have room to make notes and calculations at the bottom of each table.

Answer the following questions on another separate piece of paper. Show all work as appropriate.

2. The net ionic equation for Procedure 1 and 2 is shown below. It occurs in an acidic solution.
 - a. Determine the oxidation number for all the elements.
 - b. Determine what species is oxidized and which is reduced.
 - c. The reaction above is not balanced, so you must balance it.



3. The net ionic equation for Procedure 3 is shown below. This reaction also occurs in an acidic solution.
- Determine the oxidation number for all the elements.
 - Remember that in all “peroxides,” oxygen has the unusual (and unstable) oxidation number of -1 , not -2
 - Determine what species is the oxidizing agent, and which species is the reducing agent.
 - It is important to note that the oxygen in the the permanganate ion is NOT oxidized. The oxygen in the permanganate ends up as oxygen with the same -2 oxidation number in water that you will add into the equation in part ii. of step c
 - Balance the redox reaction below.



4. Do a search to find out what it means to standardize a solution. Explain briefly.
5. In procedure 1 you will use iron(II) sulfate heptahydrate. Write out a chemical formula for this compound and calculate its molar mass.
6. What mass of iron(II) sulfate heptahydrate would completely react with approximately 10 ml of 0.01 M KMnO_4 ?
7. What volume of 0.010 M potassium permanganate solution would be required to completely react with 1.0 ml of 3% hydrogen peroxide solution. (The solution is 3% by mass of pure H_2O_2 in an aqueous solution. Assume the density of the peroxide solution is 1.02 g/ml)

Materials on each tray

- dispensing flask with ~0.01 M KMnO_4
- bottle with dropper attached that contains - 6 M H_2SO_4
- brown bottle of commercial hydrogen peroxide with 1 ml glass pipet and bulb
- 1x vial of approx 1.0 g of iron(II) sulfate heptahydrate with a scoop (to share with lab bench mates)
- 2x Mortar and pestle
- 2x Buret & clamp
- 2x stirring plate & bar
- 2x deionized water squirt bottle
- 250 ml waste beaker - Labeled WASTE
- 2x 125 ml flasks
- 2x 50 ml flasks
- 2x 25 ml volumetric flask with plastic pipet
- 4x iron tablets

Procedure 1 Standardization of Potassium Permanganate Solution

Goggles must be worn at all times.

Use the cleaner on top of the cabinet if there are too many fingerprints on your goggles.

- A. Measure out approximately 0.1500 g of iron(II) sulfate heptahydrate into a clean, tared 50 ml flask. It need not be exactly 0.15 g, but you do need to know exactly how much you have.
- B. Dissolve the iron(II) sulfate in approximately 20 ml of deionized water. You may put in the stirring bar to stir and help it dissolve.
- C. Prepare the 50 ml buret for the potassium permanganate solution. To do this rinse the buret with about 5 ml of KMnO_4 solution, allowing some to flow through the tip and twirling the buret and draining it out the top into the sink as demonstrated in class. Then fill the buret with approximately 50 ml of MnO_4^- solution, allow a quick gush to flow through the tip into the waste beaker - in an attempt to remove air bubbles from the glass tip of the buret. Make sure the volume is not above the graduated increments at the top, and record the initial volume.
- D. Add approximately 1 ml of 6 M H_2SO_4 to the 50 ml flask to acidify the solution and provide any H^+ ions needed for the reaction to proceed.
- E. Using the stirring plate, titrate the iron(II) sulfate heptahydrate solution with MnO_4^- solution until a faint purple color persists for at least 30 seconds. Record the final volume of MnO_4^- solution in the buret.
- F. Repeat as at least once more, and a third time if necessary. You must rinse and should dry the outside of the flask in between trials. Perform your calculations after the second trial, and if your results are within 5% of the first, you need not repeat a third time.

Processing the Data – The reaction for this titration is the in the PreLAD #2

1. Calculate the number of moles of iron(II) sulfate heptahydrate.
2. Determine the number of moles of Fe^{2+} in solution.
3. Use stoichiometry to calculate the number of moles of MnO_4^- used to oxidize the iron.
4. Calculate the volume of the MnO_4^- solution used knowing your initial and final volumes.
5. Calculate the molarity of the MnO_4^- solution in your buret. remember: *Molarity = moles / Liter of solution*

Procedure 2 Analysis of Commercial Hydrogen Peroxide by Redox Titration

Goggles must be worn at all times. - Use cleaner on top of cabinet if there are too many fingerprints on your goggles.

- A. Obtain a 125 ml flask. No need to dry it. Using the buret on the center lab bench with the commercial hydrogen peroxide, measure out approximately 1.00 ml of H_2O_2 . It need not be exactly 1 ml, but whatever it is, you need to know exactly.
- B. Add approximately 3 ml of 6 M H_2SO_4 to acidify the solution and provide any H^+ ions needed for the reaction to proceed.
- C. Put your flask on the stirring plate, put in the rinsed stirring bar, and establish a gentle stirring rate. You should add some deionized water as necessary to get enough volume of liquid to allow smooth mixing.
- D. Refill your buret with MnO_4^- solution and record the starting volume. Using the standardized MnO_4^- solution from Procedure 1, – DON'T PANIC at the initial pink color, it will disappear – Continue to titrate the solution in the flask until a pale persistent pink remains. Again, you know you have reached the endpoint when the pale purple persists for about 30 seconds before fading. Record the final volume.
- E. Repeat. You must rinse the flask in between trials. Perform your calculations after the second trial, and if your results are within 5% of the first, you need not repeat a third time.
- F. To complete the calculations, we need to know the density of the H_2O_2 solution. Tare a clean DRY 25 ml volumetric flask. Fill to the etched line with the hydrogen peroxide solution and then mass the 25 ml of hydrogen peroxide solution.

Processing the Data - The balanced equation for this titration is in the PreLAD #3.

1. Knowing initial and final volumes, calculate the volume of MnO_4^- solution used.
2. Using the concentration of the permanganate solution that you determined in procedure 1, calculate the number of moles of MnO_4^- used.
3. Use the stoichiometry of the balanced equation to calculate the number of moles of H_2O_2 in the solution sample.
4. Use the molar mass of H_2O_2 to calculate the mass of pure H_2O_2 in the solution sample.
5. From Procedure F above, calculate the density of the hydrogen peroxide solution.
6. Using the density that you calculated in #5 above, calculate the mass of the volume of the sample that you used.
7. Calculate the mass percent of pure H_2O_2 in the solution sample. Compare it to the percentage listed on the bottle.

Procedure 3 Analysis of an Over-the-Counter Iron Supplement by Redox Titration

Goggles must be worn at all times. - Use the lens cleaner on top of cabinet if there are too many fingerprints on your goggles.

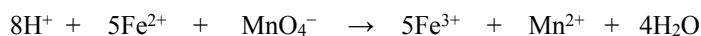
- A. Mass the iron supplement tablet.
- B. Crush it carefully with the mortar and pestle. Carefully put all of the crushed tablet into the 125 ml flask. Rinse the mortar and pestle with a small amount of deionized water to capture any remaining residue, pouring it into the flask. Bring the volume up to about 25 ml of deionized water. Use the rinsed stirring bar to aid in the dissolving process.
- C. Acidify the solution with approximately 2 ml of H_2SO_4 to provide any H^+ ions needed for the reaction to proceed.
- D. Refill the permanganate buret again, recording the starting volume. Titrate the iron tablet solution with the standardized solution to a pale persistent purple. Record the final volume.
- E. Repeat as necessary. You must rinse and should dry the outside of the flask in between trials. Perform your calculations after the second trial, and if your results are within 5% of the first, you need not repeat a third time.

Processing the Data The reaction for this titration is the same as Procedure 1

1. Knowing initial and final volumes, calculate the volume of MnO_4^- solution used.
2. Using the concentration of the permanganate solution that you determined in procedure 1, calculate the number of moles of MnO_4^- used.
3. Use the stoichiometry of the balanced equation to calculate the number of moles of Fe^{+2} that were in the tablet solution.
4. Using the molar mass of Fe, calculate the number of *milligrams* of Fe^{2+} in the tablet. Compare that to the mass listed on the tablet bottle (on the center lab bench).

Post LAD Questions

1. Back in the day when you were young chemists, and we only concerned ourselves with atoms when we balanced equations, you would have balanced the first equation (shown below) without the 5's on the Fe ions. Why, from a redox point of view do we need those 5's?



2. When you performed the calculations, why it was important to know that the iron(II) sulfate we used was a heptahydrate? How would your molarity of permanganate results have been different (higher or lower) if you had unwittingly forgotten it was a hydrate?
3. In each of the procedures, a small amount of 6 M sulfuric acid was added. Why is it an approximate quantity and an exact amount is not important?
4. The point at which you know when to stop a titration is called the "endpoint". How did we employ the color of the permanganate ion and the color (or lack thereof) of the other species in the reaction to tell us when we had arrived at the endpoint?
5. Using the mass of iron in your tablet and the mass of the tablet itself, calculate the mass percent of iron in the tablet. Show your work clearly
6. Obviously the tablet is not 100 % iron ions, some of the remaining percentage is the anion that is part of the iron compound - what is the anion? What might be the purpose of any other materials that are in the tablet?

7. The box labels the pills as iron tablets. The label does not report the presence of iron ions. When we report the mass of Fe, does it really matter if we label it Fe or Fe^{2+} ? Is there any significant difference in the mass of iron atoms or iron ions? Explain.

8. Why might it be difficult to analyze a gel-coat tablet? Or a tablet that has a bright color coating?

9. Your mother has been taking iron tablets and her doctor just told her to “upgrade” to the extra strength tablets. The regular tablets cost \$22.95 for 100 tablets which contain 65 mg of iron, and the extra strength tablets cost \$18.95 per 30 tablets which contain 130 mg of iron. You want to help your mother get the best value for her money, should you advise her to buy the extra strength tablets or just take a double dose of regular tablets. Show your calculations that support your answer.

10. It is important to dry the outside of the flask between trials so that when handling it on and off the balance, the mass does not change due to water inadvertently wiped off, nor do we want to get the balance pans wet. But, why is it not important if the inside is dry?

11. During the third procedure, water is added to the hydrogen peroxide in flask to provide enough water for good stirring action. Surely this water would dilute the hydrogen peroxide solution. Why does this water not affect the lab results?

12. You calculated % H_2O_2 by mass for the solution which we were able to compare to the label on the bottle, but you could calculate molarity. Determine the molar concentration of the hydrogen peroxide solution that you tested. Show your calculations clearly.