LAD B3 (pg 1 of 6) Analysis by Redox Titration

Name_____Per____

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. Atoms undergo oxidation when a loss of electrons occurs, and an atom undergoes reduction when it gains electrons. These two processes always occur simultaneously because there must be a transfer of electrons from one atom to another.

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 with another type of redox reaction that occurs in solution. In these type of redox reactions atoms will not be found in their elemental state, but instead atoms or ions (perhaps as part of a larger ion or molecule) will increase their oxidation number and be oxidized, while other atoms or ions (also perhaps as part of a larger ion or molecule) will decrease their oxidation number and be reduced. A common ion that we will use in this lab, the permanganate ion, MnO_4^-, is very often used to oxidize other substances. 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 will *standardize* a potassium permanganate solution that you will then use for parts 2 & 3. This *standardization* is done by titrating a solution of iron(II) ions with a known number of moles against a permanganate ion solution whose concentration you need to determine. You will have a "rough" idea of the concentration, but you'll need to do this initial titration to "confirm the molarity" or *standardize* the solution. In this lab, you will *standardize* the potassium permanganate solution by titrating the permanganate into a measured mass 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 water, then titrating it with the standardized potassium permanganate solution to analyze the actual amount of iron in the tablet and then 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 with your LAD.*

1. Read the Procedure and Processing the Data and then make a Data/Results Table -- be sure and make three (3) separate sections to coincide with the three different procedures.
   Those 3 sections should be stacked on top of each other NOT side by side.

2. The skeleton equation for Procedure 1 & 2 is shown below. It occurs in an **acidic** solution. If the solution were not acidified, a different oxidation state of manganese would result (resulting in Mn^{4+}, forming an orange ppt MnO_2, instead of colorless Mn^{2+}).

   a. Determine the oxidation number for all the elements. List them above the species in the equation below.

   b. *Identify* which species is oxidized and which is reduced.

$$Fe^{2+} + MnO_4^- \rightarrow Fe^{3+} + Mn^{2+}$$

oxidation half reaction, balance

reduction half reaction, balance

overall balanced equation

adapted from Lab Investigations by David Hostage & Martin Fossett

3. The skeleton equation for Procedure 3 is shown below. This reaction also occurs in an acidic solution.
   a. Determine the oxidation number for all the elements. List them above the species in the equation below.
      - Remember that in all "peroxides," oxygen has the unusual (and unstable) oxidation number of −1, not −2
      - 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, and has the same −2 oxidation number.
   b. Identify which species is oxidized and which is reduced.

$$H_2O_2 + MnO_4^- \rightarrow O_2 + Mn^{2+}$$

oxidation half reaction, balance

reduction half reaction, balance

overall balanced equation

4. In procedure 1 you will use iron(II) sulfate heptahydrate. Write out a chemical formula for this compound and calculate its molar mass.

5. What mass of iron(II) sulfate heptahydrate would completely react with approximately 10 ml of 0.010 M $KMnO_4$? *Calculations must be clearly shown.*

6. What volume of 0.010 M potassium permanganate solution would be required to completely react with 0.50 g of hydrogen peroxide solution. The commercial solution is 3% by mass of pure H_2O_2 in an aqueous solution. *Calculations must be clearly shown.*

Materials on each tray

- dispensing flask with ~0.01 M $KMnO_4$
- bottle with dropper attached that contains − 6 M H_2SO_4
- dropping bottle with 6 M H_3PO_4
- 1x vial ~ 2.0 g of iron(II) sulfate heptahydrate
- scoop
- 2× Mortar and pestle
- 2× Buret & clamp
- 2× stirring plate & bar
- 2× deionized water squirt bottle
- 250 ml waste beaker − Labeled WASTE
- 2× 10 ml beakers to cover burets
- 2× 125 ml flasks
- 2× 50 ml flasks
- 2× 25 ml volumetric flask with plastic pipet
- bottle of iron tablets
- brown bottle of commercial hydrogen peroxide with 1 ml pipet

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 0.15 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 tap water. Put in the stirring bar to stir and help it dissolve.

C. Prepare or "season" 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 a good sized squirt 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 to the correct oxidation state of manganese. Also add several drops of concentrated H_3PO_4 to complex the Fe^{2+} and keep any insoluble $Fe(OH)_2$ from forming.

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. Perform your calculations, then repeat as at least once more, and a third time if your calculations do not match closely enough. You must rinse and should dry the outside of the flask in between trials.

G. SPECIAL TRIAL not to be recorded on your data table. A demonstration trial will be run using approximately 0.15 g of iron(II) sulfate heptahydrate but without using any acid. (*This demonstration will be helpful for PostLAD Question # 3.*)
   - What observational differences did you notice compared to your trials?

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 to reach the endpoint knowing your initial and final volumes.
5. Calculate the molarity of the MnO_4^- solution in your buret. Remember: $Molarity = \frac{moles}{Liters\ of\ Solution}$

Procedure 2 Analysis of an Over-the-Counter Iron Supplement by Redox Titration

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

A. Read the label of your iron tablets and report the milligrams of iron listed to your data table.

B. Mass the iron supplement tablet.

C. Crush the tablet 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 tap water if necessary to capture any remaining residue, pouring it into the flask. Add enough water to just cover the stir bar. Use the rinsed stirring bar to aid in the dissolving process.

D. Acidify the solution with a good sized squirt of H_2SO_4 to provide any H^+ ions needed for the reaction to proceed. Also add several drops of H_3PO_4 to complex the Fe^{2+} and keep any insoluble $Fe(OH)_2$ from forming.

Read the tablet bottle and record the # mg of iron (in your data table) and read the ingredients to see if there are any carbonates present. If there are carbonates, you will need an extra squirt or two of acid.

E. 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.

F. Repeat only after completing Procedure 3.

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 to reach the endpoint.
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 *milli*grams of Fe^{2+} in the tablet.
5. Compare #4 to the mass of iron listed on the tablet bottle and calculate percent error.
6. Calculate the percentage of iron in the tablet.

Procedure 3 Analysis of Commercial Hydrogen Peroxide by Redox Titration

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

A. Obtain a 125 ml flask and tare it. Using the pipet and bottle of H_2O_2 mass between 0.5 − 0.8 g.

B. Add a small squirt of 6 M H_2SO_4 to acidify the solution.

C. Put your flask on the stirring plate, put in the rinsed stirring bar, and establish a gentle stirring rate. You should enough tap water as necessary to just cover the stir bar.

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 if you get a pink color with just a few drops, it will disappear as you proceed – CONTINUE ADDING TITRANT, after a couple of milliliters the pink will disappear, then you can 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.

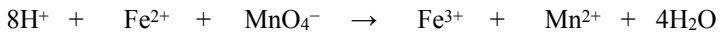
E. Repeat. You must rinse the flask in between trials, be sure and dry it off on the outside, though no need to dry the inside. Perform your calculations after the second trial, and if your results are "close enough", you need not repeat a third time.

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^- used to reach the endpoint.
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. Knowing the molar mass of H_2O_2, calculate the mass of pure H_2O_2 in the solution sample.
5. Calculate the mass percent of pure H_2O_2 in the solution sample.
6. Compare it to the percentage listed on the bottle by calculating percent error.
7. Alternatively, you could calculate molarity. Determine the molar concentration of the hydrogen peroxide solution that you tested.

Post LAD Questions

1. Back in the day when you were young chemists, and you only concerned yourselves with atoms when you balanced equations, you might have balanced the first equation (shown below) without the 5's on the iron ions. Now you might say, "Oh we need the 5's to balance the charges." But why, from an *electron* point of view do we need those 5's?



$$8H^+ + Fe^{2+} + MnO_4^- \rightarrow Fe^{3+} + Mn^{2+} + 4H_2O$$

2. When you performed the calculations, why it was important to know that the iron(II) sulfate we used was a heptahydrate? Would the molarity of permanganate have been higher, lower, or unchanged if you had unwittingly forgotten that the iron(II) sulfate was a hydrate?

3. In each of the procedures, a small amount of 6 M sulfuric acid was added. Why was it necessary? Refer to PreLab #2 and Special demo trial Procedure 1 G. Why is it an approximate quantity and an exact amount is not important?

4. What ingredient in the iron tablet may have caused your procedure to require extra sulfuric acid in Procedure 2 as compared with Procedure 1. Write a reaction that shows the use of extra acid to justify your response.

5. 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? Most importantly, what do we now know has happened at this endpoint?

6. The compound listed on the front of the tablet bottle is ferrous sulfate. This is old-school naming for iron(II) sulfate. Obviously the tablet is not 100 % iron ions (does Part 2 process data #6 support this?), some of the remaining percentage is the anion that is part of the iron compound − what is the anion? The remaining percentage of the tablet are other ingredients listed on the bottle, from our in-class discussion, what might be the purpose of other materials that are listed as ingredients 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. It is important to dry the outside of the flask between trials so that when handling the flask 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 does it not matter if the inside is wet?

9. During the third procedure, water is added to the hydrogen peroxide in the 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?

10. If you had not “seasoned” your buret which may have been rinsed with distilled water by students from the previous class, when doing Procedure 1 would this lack of preparation of the buret produce a calculated molarity that was higher, lower or have no effect on the calculated molarity of permanganate? Justify your answer.