Experiment 4

Redox Reactions

Introduction

Redox is the term given to reactions that involve a transfer of electrons from one reactant to another. The reactant that gains electrons is said to be **"reduced"** and the reactant that loses the electrons is said to be **"oxidized"**. Reduction and oxidation are combined into the term "redox" because you can never have one without the other.

Reduction is a gain of electrons.
Oxidation is a loss of electrons.

At the end of a chemical reaction, the net gain of electrons must be equal to the loss of electrons. If one reactant loses electrons, another reactant must gain them. Therefore, you must always have at least 1 reduction reaction and at least 1 oxidation reaction. In electrochemistry, these two reactions are called **"half-reactions"** and they occur simultaneously in a redox reaction.

Oxidation Numbers

Oxidation numbers (also called **oxidation states)** are the theoretical "charges" on an atom in a molecule or ion or on the ion itself. The oxidation number of any atom can be determined by following the rules below.

Rules for Determining Oxidation Numbers

These are the rules that must be followed when determining an oxidation number. They must be followed in order, with the rule higher up on the list taking precedence over a rule lower in the list when there are conflicts. In general, work from the outside atoms first going towards the center atoms last.

1. Oxidation number = 0 for elements (not in a compound) and compounds
    a. In diatomic elements (e.g. N_2, O_2, the halogens, H_2) each individual atom is treated as an element and is assigned an oxidation number of zero.
    b. The oxidation numbers of all elements in a neutral compound must add up to zero.
2. Oxidation number = the ionic charge on an ion (written as a superscript).

For elements within a compound: Apply the following rules to each atom individually and in order:

3. Oxidation number = +1 for group IA elements and H (if with another 1A element, H is -1)
4. Oxidation number = +2 for group 2A elements.
5. Oxidation number = -2 for each oxygen atom.
6. Oxidation number = -1 for group 7A elements
    a. If both elements in the compound are in group 7A, then the one higher in the list is assigned an oxidation number of -1.
7. Oxidation number = -2 for group 6A elements.
8. Oxidation number = -3 for group 5A elements (very shaky!!!)

Example 1: Assigning oxidation numbers for the atoms in potassium permanganate, $KMnO_4$

Rule 1: Oxidation number = 0 for elements, molecules and salts.
$KMnO_4$ has an overall oxidation number of zero since it is a salt.

Rule 3: Oxidation number = +1 for group IA elements and H.
Potassium, **K**, is a group 1 atom, so it must have an oxidation number =+1.

Rule 5: Oxidation number = -2 for each oxygen atom.
There are 4 **oxygen**, **O**, atoms, so 4 x -2 = -8 for the total charge for all atoms together.
Note: The oxidation number for each oxygen atom is -2, not -8.

We used the rules for assigning 2 out of the 3 atoms in potassium permanganate but notice that there are no hard and fast rules for transition elements. Transition elements can have varying oxidation numbers depending on the other elements present in the compound. To find the oxidation number of a transition element (or the last element in any compound), use the information you already have from the rules and the fact that all of the oxidation numbers in a neutral compound must add up to zero. The oxidation number of the remaining element is whatever value is needed to make the overall oxidation number of the compound zero. In the case of a polyatomic ion, the oxidation number of the remaining element is whatever value is needed to make the overall oxidation number of the polyatomic ion equal to its charge.

$KMnO_4$ Overall charge on compound= 0 (Rule 1)
1K : +1 (Rule 3)
4O : −8 (Rule 5)
$0 = K + 4O + Mn$
$0= (+1) + (-8) + Mn$ so $Mn=+7$

MnO_4^- Ionic charge = −1 (Rule 2)
4O : −8 (Rule 5)
$-1 = 4O + Mn$
$-1 = (-8) + Mn$ so $Mn=+7$

If you are given the name, you can determine the oxidation number of a transition element from its roman numeral. For example, the iron atom in iron (II) oxide will have an oxidation number of +2 and the iron atom in iron (III) oxide will have an oxidation number of +3.

Half-Reactions and Balancing Charges

For the example, $Fe^{2+} + Mn^{7+} \rightarrow Fe^{3+} + Mn^{2+}$, the elements are balanced, but the charges are not. There is an overall +9 charge on reactant side but only a total of +5 on product side. You cannot have unbalanced charge between products and reactants, so the charges must be balanced as well. The easiest method of balancing charge is to use half-reactions, which are written to show the number of electrons transferred in the oxidation and reduction processes. The two half-reactions, 1 reduction and 1 oxidation, are balanced separately and then multiplied by a whole number, if necessary, to ensure that both half-reactions have the same number of electrons. The two half-reactions are then added together to cancel the electrons and give the overall redox reaction with balanced charges.

The two half reactions for the previous example are as follows:

Reduction: $Mn^{7+} + 5e^- \rightarrow Mn^{2+}$ Electrons are reactants
Adding 5 electrons to the reactant side balances the charge (+7 - 5 = +2).
Now there is a +2 charge on each side of the equation.
The oxidation number of the element, Mn, decreases from +7 to +2.

Oxidation: $Fe^{2+} \rightarrow Fe^{3+} + 1e^-$ The electrons are on the product side
Adding 1 electron to the product side balances the charge (+2 = +3 - 1).
Now there is a +2 charge on each side of the equation.
The oxidation number of Fe increases from +2 to +3.

The reduction half-reaction requires 5 electrons, but the oxidation half-reaction only requires 1 electron, so the oxidation half-reaction must be multiplied by 5 to make the number of electrons involved in both processes equal:

$$\begin{array}{lcl} 1 \times (Mn^{7+} + 5e^- \rightarrow Mn^{2+}) & = & Mn^{7+} + \cancel{5e^-} \rightarrow Mn^{2+} \\ \underline{5 \times (Fe^{2+} \rightarrow Fe^{3+} + 1e^-)} & = & \underline{5Fe^{2+} \rightarrow 5Fe^{3+} + \cancel{5e^-}} \\ & & 5Fe^{2+} + Mn^{7+} \rightarrow 5Fe^{3+} + Mn^{2+} \end{array}$$

Now, each side of the balanced equation has a charge value of +17 and a total of 5 electrons are transferred between the iron and manganese ions. Note that the value for the total charge on each side of the chemical equation does not have to equal zero, but it must be the same for both sides. The balanced chemical equation is written as: $5Fe^{2+} + Mn^{7+} \rightarrow 5Fe^{3+} + Mn^{2+}$.

Oxidizing and Reducing Agents

Reduction never occurs without oxidation and oxidation never occurs without reduction. However, often only one of these half-reactions is of interest. For example, chlorine bleach works by oxidizing substances in stains so we say chlorine bleach is an "**oxidizing agent**". Any reactant that drives a redox reaction is considered an "agent". If the reactant causes oxidation, it is an oxidizing agent, if it causes a reduction the chemical is referred to as a **reducing agent**.

Oxidizing agents cause oxidation. If a chemical causes oxidation, then the substance it works on must have lost electrons in the oxidation process. That means that the oxidizing agent must have gained those electrons. Therefore, the oxidizing agent is reduced. To remember where oxidizing agents are in the periodic table, look for the element "oxygen". Thus, the nonmetals, located on the right side of the periodic table, are good oxidizing agents. They want to gain electrons to become more stable. Oxidizing agents get these electrons by pulling them away from another chemical, thus causing oxidation.

Reducing agents cause reduction. Reducing agents force other substances to receive electrons. Thus, the reducing agent is oxidized. To remember where reducing agents are in the periodic table, look to groups I and II. These groups want to form ions with positive charges and thus lose electrons. To rid themselves of these electrons, they must force another substance to take them, thus causing a reduction to occur.

In Your Experiment

There are 2 parts in this experiment. First you will manipulate the oxidation states of manganese by performing a series of redox reactions that cause the oxidation number of manganese to change. The color and phase changes observed are indicative of the transfer of electrons that takes place as the manganese becomes increasingly reduced from the +7 oxidation state towards the +2 oxidation state.

The second part of the experiment is a redox titration. You will use potassium permanganate to oxidize a solution containing Fe^{2+}. The iron (II) ion is oxidized to iron (III). The manganese is reduced from Mn(VII) to Mn(II).

$$5Fe^{2+} + Mn^{7+} \rightarrow 5Fe^{3+} + Mn^{2+}$$

This portion of the experiment is a practical application of redox theory and includes calculations that are commonly performed in titration experiments based on the ratios of the reactants and products in the balanced redox reaction. You will also review mass percent calculations and percent error in this exercise.

Part 1: Oxidation States of Manganese:

Manganese is a transition metal that can exist in ionic form with a variety of oxidation numbers. The states that are most commonly found are Mn^{7+}, as seen in the permanganate ion, MnO_4^- and Mn^{4+} in solid form as MnO_2. By controlling the pH and type and amount of the reducing agent in the redox reactions, the less stable oxidation states of Mn^{6+}, Mn^{5+} and Mn^{3+} and Mn^{2+} can also be observed. Some background on each of these oxidation states is given below. You do not need to be able to write the balanced redox reactions on your own, but you do need to be able to determine the oxidation numbers of all atoms as well as balance simple half-reactions based on oxidation number rules.

Mn^{7+}

The +7 ion is usually found in the form of permanganate, MnO_4^-. In an aqueous solution, MnO_4^- has a dark purple-pink color. It is extremely reactive and oxidizes other compounds easily due to the very high positive charge. If it gets on your skin or clothing, it will cause a brown discoloration as the manganese is reduced to an oxidation state of +4 and MnO_2 is formed. (**Do not try this reaction on your clothes or skin, the stain is often permanent**.)

Mn^{6+}

The +6 ion is found in complex form as MnO_4^{2-}. It is not stable, but can be prepared by adding a reducing agent, sodium bisulfite ($NaHSO_3$), to a basic solution of permanganate. A dark blue-green solution is indicative of the MnO_4^{2-} ion. Note the color quickly – it will disappear as the Mn^{6+} reduces easily to Mn^{4+}.

$$2e^- + \underset{(Mn^{7+})}{2MnO_4^-} + 3OH^- + \underset{(S^{4+})}{1HSO_3^-} \rightarrow \underset{(Mn^{6+})}{2MnO_4^{2-}} + \underset{(S^{6+})}{1SO_4^{2-}} + 2H_2O + 2e^-$$

Mn^{5+}

The unstable +5 ion is found in complex form as MnO_4^{3-}. It exists only under very basic conditions. This ion can only be prepared if the permanganate solution is first made extremely basic by adding a pellet of sodium hydroxide. Note that the reactants are the same as in the previous example, but the ratios in the chemical reaction are different. The solution must be extremely basic to form the Mn^{5+} ion rather than the Mn^{6+} ion. A pale blue-green solution is indicative of the presence of the MnO_4^{3-} ion. If you get the dark green-blue solution from the previous example, your solution was probably not basic enough and you will need to start over.

$$2e^- + \underset{(Mn^{7+})}{1MnO_4^-} + 3OH^- + \underset{(S^{4+})}{1HSO_3^-} \rightarrow \underset{(Mn^{5+})}{1MnO_4^{3-}} + \underset{(S^{6+})}{1SO_4^{2-}} + 2H_2O + 2e^-$$

Mn^{4+}

The +4 oxidation state is naturally present in the solid salt, manganese dioxide, MnO_2. It is a stable salt that forms easily when permanganate reacts. When formed from an aqueous solution containing permanganate ion, manganese dioxide is a brown precipitate that is seen as soon as the permanganate is reduced, leaving a colorless solution and a brown precipitate containing the manganese. The solution needs to be very slightly basic to force the reaction to completion.

$$1OH^- + \underset{(Mn^{7+})}{2MnO_4^-} + \underset{(S^{4+})}{3HSO_3^-} \rightarrow \underset{(S^{6+})}{3SO_4^{2-}} + \underset{(Mn^{4+})}{2MnO_2} + 2H_2O$$

Mn^{3+}

The +3 oxidation state is fairly unstable and very difficult to obtain. Formation of the +3 ion must take place in an acidic solution, so that the O^{2-} ions released by the permanganate can immediately attach to two H^+ ions to form water. To provide a source of H^+ ions, the Mn^{2+} solution is prepared in 3M H_2SO_4. The Mn^{7+} in the MnO_4^- pulls electrons from the Mn^{2+} ions converting both the Mn^{7+} and the Mn^{2+} to the same ion, Mn^{3+}. Thus, the product of both half-reactions is Mn^{3+}.

$$4e^- + 8H^+ + \underset{(Mn^{7+})}{MnO_4^-} + \underset{(Mn^{2+})}{4Mn^{2+}} \rightarrow 4H_2O + \underset{(Mn^{3+})}{5Mn^{3+}} + 4e^-$$

Mn^{2+}

The +2 oxidation state is stable in an acidic aqueous solution. The salts, $MnSO_4$ or $Mn(NO_3)_2$, are a very pale pink. When dissolved in water at high enough concentrations, the solution will also be a very pale pink. This color is difficult to see at dilute concentrations, so you will need to hold a piece of white paper under the test tube to discern the color.

Part 2: Reaction of Potassium Permanganate with Iron (II) Ammonium Sulfate

In this section, you will perform a redox titration that involves the reaction of potassium permanganate ($KMnO_4$) with iron (II) ammonium sulfate hexahydrate, $Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O$. Potassium permanganate ($KMnO_4$) has a very dark pink color in an aqueous solution and is a good oxidizing agent. Since the oxidizing agent is reduced during a redox reaction, the permanganate ion (MnO_4^-) will be reduced to manganese ion (Mn^{2+}) in the presence of the reducing agent, Fe^{2+}.

Reduction: $MnO_4^-{}_{(aq)} + 5e^- \rightarrow Mn^{2+}{}_{(aq)}$

(dark pink) (colorless or very pale pink)

When dissolved in water, $Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O$ ionizes and produces Fe^{2+}. The Fe^{2+} is oxidized by the MnO_4^- to Fe^{3+}, which is a pale yellow color. Each Fe^{2+} ion loses 1 electron that is gained by the manganese during the reduction of MnO_4^- to Mn^{2+}.

Oxidation: $Fe^{2+}{}_{(aq)} \rightarrow Fe^{3+}{}_{(aq)} + 1e^-$

(colorless) (light yellow)

To balance the charge, 5 Fe^{2+} ions will be needed for every Mn^{7+} that is reduced.

Redox Reaction: $5Fe^{2+}{}_{(aq)} + Mn^{7+}{}_{(aq)} \rightarrow 5Fe^{3+}{}_{(aq)} + Mn^{2+}{}_{(aq)}$

Redox Titrations

Like acid-base titrations, redox titrations are essentially just stoichiometry problems. In this titration, you will gradually add the dark pink permanganate solution to a colorless iron (II) solution and watch as the permanganate oxidizes the Fe^{2+} to Fe^{3+}. The dark pink color of the permanganate disappears as it is reduced to form Mn^{2+} and Fe^{3+} ions. Once all of the Fe^{2+} is gone, there is nothing left to reduce the permanganate and the last drop of permanganate remains purple since it cannot be reduced to Mn^{2+}. Thus, your solution will remain very slightly pink due to the diluted permanganate ion that remains in the solution. This is your endpoint.

Once you have achieved your endpoint, you will record the volume of titrant that was needed by reading the value from the buret. You will need to use stoichiometry to ultimately determine the mass of iron in the mass of salt used. As usual, the steps needed to perform this calculation are in the calculations section of the experiment but you will need to reproduce them on your own in the concept review.

Once you have determined the mass of iron in your sample, you will calculate a mass percent iron in the sample of $Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O$ that you used in your experiment as a source of Fe^{2+}. You will then compare your calculated percent iron to the theoretical value for percent iron in the same compound. Your experimental value should be within 5% of the theoretical value. Remember that mass percent is simply a comparison of the mass of an element or ion in a compound to the total mass of the compound:

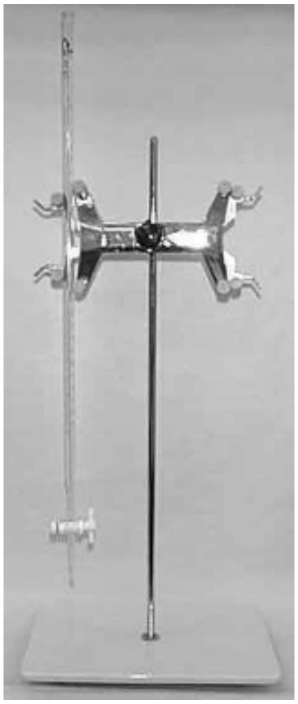
$$\text{Percent Mass} = \left(\frac{\text{Mass of Element (or ion) in compound}}{\text{Total Mass Compound}}\right) \times 100$$

Equipment Procedures

Using a Buret

A **buret** is a precisely measured piece of glassware used to measure volume as it is delivered. It is often used in titration reactions.

Pre-rinsing the buret



1. Check to see that the stopcock is closed.
2. Pour approximately 2 mL of the solution being used into the buret.
3. Remove the buret from the clamp and gently roll the buret nearly horizontally so that the solution coats the inside of the buret.
4. Replace the buret in the buret clamp.
5. Place a waste beaker below the buret and allow the solution to drain into the waste beaker.

Filling the buret

1. Check to see that the stopcock is closed.
2. Use a funnel to fill the buret with the solution to be used in the titration. This solution is called the "titrant". Be careful not to overfill the buret.
3. Place a waste beaker below the buret.
4. Carefully open the stopcock to allow solution to flow into the tip of the buret and remove any air that may remain.
5. Check for bubbles remaining in the buret. Tapping gently on the side of the buret will often cause them to move to the top of the solution. If you have trouble removing bubbles, contact your TA. Any bubbles present in either the tip of the buret or in the main body of the buret will cause errors in your volume measurements, so all air must be removed before starting the titration.

Titrating with a buret

1. Record the volume of the liquid at the top of the buret. **It does not have to be exactly zero** but it does need to be recorded accurately based on the lines of the buret.
2. Place the flask containing the solution below the tip of the buret.
3. If you are using a magnetic stirrer, set it up at this time.
4. Slowly open the **stopcock** to allow the titrant to begin to drip into the flask. Be sure to keep swirling the liquid in the flask to keep the titrant continually mixing with the solution in the flask.
5. As you approach the endpoint, (solution remains colored for longer period of time), slow down the drip rate.
6. Close the stopcock and record the volume when you think the reaction is complete.
7. If you overshoot the endpoint (solution remains dark pink) you will need to re-do the titration, so work carefully.

Lab Precautions

Chemical Hazards

2M Sulfuric Acid Solution

NFPA RATING: HEALTH: 2 FLAMMABILITY: 0 REACTIVITY: 0
DERMAL EXPOSURE:
Wash off with soap and plenty of water.
EYE EXPOSURE:
Flush with copious amounts of water for at least 15 minutes. Contact your TA immediately.

Sodium Hydroxide: Solid

NFPA RATING: HEALTH: 3 FLAMMABILITY: 0 REACTIVITY: 2
ORAL EXPOSURE
Caustic. If swallowed, wash out mouth with water provided person is conscious. Do not induce vomiting. Contact your TA immediately.
DERMAL EXPOSURE
Caustic. In case of extensive skin contact, flush with copious amounts of water for at least 15 minutes. Remove contaminated clothing and shoes. Contact your TA immediately.
EYE EXPOSURE
Caustic. In case of contact with eyes, flush with copious amounts of water for at least 15 minutes. Assure adequate flushing by separating the eyelids with fingers. Contact your TA immediately.
REACTIVITY
Will react strongly with water and acids, use caution when handling

50% Sodium Hydroxide Solution

NFPA RATING: HEALTH: 2 FLAMMABILITY: 0 REACTIVITY: 1
ORAL EXPOSURE
Caustic. If swallowed, wash out mouth with water provided person is conscious. Do not induce vomiting. Contact your TA immediately.
DERMAL EXPOSURE
Caustic. In case of extensive skin contact, flush with copious amounts of water for at least 15 minutes. Remove contaminated clothing and shoes. Contact your TA immediately.
EYE EXPOSURE
Caustic. In case of contact with eyes, flush with copious amounts of water for at least 15 minutes. Assure adequate flushing by separating the eyelids with fingers. Contact your TA immediately.
REACTIVITY
Will react with strong acids, work with caution.

0.020M Potassium Permanganate Solution

NFPA RATING: HEALTH: 1 FLAMMABILITY: 0 REACTIVITY: 0
DERMAL EXPOSURE:
Will stain clothing and skin. Flushing with water will remove permanganate solution, but will not remove stain.
EYE EXPOSURE:
Flush with copious amounts of water for at least 15 minutes. Contact your TA immediately.

0.02M Sodium Bisulfite Solution

NFPA RATING: HEALTH: 1 FLAMMABILITY: 0 REACTIVITY: 0
DERMAL EXPOSURE:
Flush with water.
EYE EXPOSURE:
Flush with copious amounts of water for at least 15 minutes. Contact your TA immediately.

2M Manganese Sulfate Solution

NFPA RATING: HEALTH: 0 FLAMMABILITY: 0 REACTIVITY: 0
DERMAL EXPOSURE:
Will stain clothing and skin. Flushing with water will remove permanganate solution, but will not remove stain.
EYE EXPOSURE:
Flush with copious amounts of water for at least 15 minutes. Contact your TA immediately.

Iron Ammonium Sulfate Hexahydrate

NFPA RATING: HEALTH: 2 FLAMMABILITY: 0 REACTIVITY: 0
DERMAL EXPOSURE
Skin irritant. In case of extensive skin contact, flush water for at least 15 minutes.
EYE EXPOSURE
In case of contact with eyes, flush with copious amounts of water for at least 15 minutes. Assure adequate flushing by separating the eyelids with fingers. Contact your TA immediately.

Concentrated Phosphoric Acid Solution

NFPA RATING: HEALTH: 2 FLAMMABILITY: 0 REACTIVITY: 0
DERMAL EXPOSURE:
Skin irritant. Caustic. Flush with water for at least 15 minutes.
EYE EXPOSURE:
Flush with copious amounts of water for at least 15 minutes. Contact your TA immediately.

Equipment Hazards

There are no hazards associated with the equipment used in this experiment.

Chemical Disposal

Dispose of all chemicals in waste container.

Experiment 4: Procedures and Data Sheet

(Submit as part of your informal report)

Name: ____ Date: _______ Section: _______

TA Signature: ___________________________________

All data must be written in pen at the time it is collected. **Pencil is not allowed!!**
Record all measurements with the correct number of significant figures and units.
TA signature & TA initials on any changes made to the data are required or the data is invalid.

Part 1: Mn^{7+} in the form of MnO$_4^-$

1. Add 4 drops of 0.02 M $KMnO_4$ solution to a clean, dry 4" test tube
2. Add 4 drops of distilled water to dilute the permanganate to better see the **dark purple color.**
3. Set the test tube in a test tube rack for comparison to the other solutions.

Part 2: Mn^{6+} in the form of MnO$_4^{2-}$

1. Add 5 drops of 0.02 M $KMnO_4$ to a second clean dry 4" test tube.
2. Add 1 drop of 50% NaOH to the $KMnO_4$. Swirl to mix.
3. Add 0.02 M $NaHSO_3$ dropwise while swirling until a **dark green-blue solution** forms.
4. Set the test tube in a test tube rack for comparison to the other solutions.

Part 3: Mn^{5+} in the form of MnO$_4^{3-}$

1. Ask your TA to add 1 pellet of sodium hydroxide to a clean dry 4" test tube.
2. Add 1 drop of 0.02 M $KMnO_4$ to the test tube. Swirl to partially dissolve the pellet.
3. Add 1 drop of 0.02 M $NaHSO_3$ to the mixture. Swirl to mix.
4. **A pale blue solution** indicative of MnO_4^{3-} is formed above the pellet.
5. Add 2-3 mL of distilled water to dissolve the pellet. Swirl until pellet is fully dissolved.
6. Set the test tube in a test tube rack for comparison to the other solutions.

Part 4: Mn^{4+} in the form of MnO$_2$

1. Add 2 drops of 0.02 M $KMnO_4$ to a clean dry 4" test tube
2. Add 5 drops of 0.02 M $NaHSO_3$ to the solution. Swirl to mix.
3. Add 1 drop of 50% NaOH. Swirl to mix. **An opaque brown solution will form.**
4. Place the test tube in the test tube rack and allow the precipitate to settle out.

Part 5: Mn^{3+}

1. Add 10 drops of 2.0M manganese sulfate solution, $MnSO_4$, to a 4" test tube.
2. Add 5 drops of sulfuric acid and swirl to mix.
3. Add 0.02 M $KMnO_4$ slowly while swirling until the color remains a **transparent reddish-brown**.
4. Set the test tube in a test tube rack for comparison to the other solutions.

Part 6: Mn^{2+} This solution was prepared by dissolving solid $MnSO_4$ in 3M H_2SO_4 to form Mn^{2+}.

1. Add 10 drop $MnSO_4$ solution to a 4" test tube to better see the color.
2. There is no reaction associated with this ion.
3. Hold the test tube over a clean white piece of paper to see the very pale pink color.
4. Set the test tube in a test tube rack for comparison to the other solutions.

Part 7: Clean-up

1. Pour all your used solutions into the waste container.
2. Wash all test tubes very well with water and air dry. You will be charged for dirty test tubes.

Have your TA initial that you have completed the first 6 parts of the experiment.

TA initials: __________

Laboratory Procedure (II): Performing a redox titration

1. Zero the balance.
2. Weigh out about 0.150-0.200g of $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$ and record the **exact mass.**
3. Transfer the solid $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$ to a 125 ml Erlenmeyer flask.
4. Use your wash bottle to add about 10 mL of distilled water to the Erlenmeyer flask.
5. Add about 10 drops of 2M sulfuric acid to the Erlenmeyer flask.
6. Swirl the flask to gently dissolve the crystals. The solution will be colorless. If the crystals do not fully dissolve, add another few drops of sulfuric acid.
7. Add the stir bar to the flask.
8. Record the exact molarity of the potassium permanganate solution. Get this value from your TA.
9. Fill a buret to the 10mL mark with the potassium permanganate solution, making sure that you fill the tip by draining some permanganate all the way through the buret into a 250mL waste beaker.
10. Place the Erlenmeyer flask on the hotplate/stirrer, insert your stir bar and turn on the stirrer to gently stir the contents of the flask. You will continue to stir while titrating.
11. Position the buret tip above the center of the flask.
12. Record the volume of the liquid in the buret with 2 decimal places.
13. Slowly start to titrate the $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$ / acid mixture with the permanganate.
14. Stop the titration when you see the $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$ / acid mixture turn a pale yellow color with no traces of the pink permanganate.
15. Record the buret reading at this point.
16. Add 1 drop of concentrated phosphoric acid to remove the yellow color. (This does not affect the titration, it just makes it easier to see the endpoint.)
17. Add 1 drop of potassium permanganate from the buret. If your solution remains pale pink or brownish you have reached the endpoint. If not, add more permanganate drop by drop until you see a pale pink color.
18. Record the volume reading of the buret once the endpoint is reached. You should need approximately 3-6mL for the titration. If it takes more than this amount, tell your TA.
19. When you are finished with the titration, dispense the remainder of the potassium permanganate solution from the buret into the waste beaker from procedure 1.
20. Use your spatula to retrieve your stirbar and dispose of all used solution in 250 mL waste beaker.
21. Dispose of all waste solutions and unused $KMnO_4$ in the waste container.
22. Fill the buret with water and allow the water to run out through the tip of the buret into a beaker. Rotate the stopcock repeatedly until no trace of permanganate solution is seen in the buret.
23. Rinse your buret twice more and dispose of waste water in the sink.
24. Refill the buret with clean water and add 2 drops of sulfuric acid.
25. Have your TA initial that the buret & all test tubes are clean.

Mass of $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$: ____________________

Molarity of $KMnO_4$ solution: ____________________

Initial Buret Reading: ____________________

Buret Reading when solution is pale yellow: ____________________

Buret reading at endpoint after additional $KMnO_4$ ____________________

Buret & all test tubes are clean: TA Initials: __________

Experiment 4: Data Rubric (20pts)

Points

Data are neat and legible	5pts	__________pts
Significant figures (>80% correct)	3pts	__________pts
Units (>80% correct)	2pts	__________pts
All data are present and make sense	10pts	__________pts

Deductions (sliding scale based on TA discretion)

Lab area left unclean	-20pts	__________pts
Improper waste disposal	-20pts	__________pts
Disruptive behavior	-20pts	__________pts
Lab coat or safety glasses removed while in lab	-20pts	__________pts
Data sheet is missing TA signature	-20pts	__________pts

Other: ____ __________pts

Comments: ____

Grade for Data Sheet __________pts

Experiment 4: Results Table
(Submit as part of your informal report)

Name: ____ Date: _______ Section: _______

Record all results with the correct number of significant figures and units.

Part 2: Performing a redox titration

Volume of $KMnO_4$ solution	
Moles of $KMnO_4$	
Moles of Fe^{2+} in sample	
Mass of Fe^{2+} in sample	
Experimental percent mass of Fe^{2+} in sample	
Actual percent mass of Fe^{2+} in $Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O$	
Percent error of mass percent in $Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O$	

Experiment 5: Results Table Rubric (20pts)

Points

Tables are neat and legible	5pts	__________pts
Significant figures (>80% correct)	3pts	__________pts
Units (>80% correct)	2pts	__________pts
All results are present and make sense	10pts	__________pts

Deductions (sliding based on TA discretion)

Results to not match data	-20pts	__________pts
Plagiarism!!! Results are identical to another student	**-100pts**	**__________pts**
Other: ____		__________pts

Comments: ____

Grade for Results Table **__________pts**

Experiment 4: Calculations

Submit as part of your informal report

Volume of $KMnO_4$

Subtract the initial buret volume from the final buret volume.

Volume of $KMnO_4$: ________________

Moles of $KMnO_4$

Use the molarity and volume of the $KMnO_4$ solution to calculate the moles of $KMnO_4$ used in the titration. Make sure to perform any necessary conversions.

Moles of $KMnO_4$: ________________

Experimental Moles of Fe^{2+} in sample

Use the Fe^{2+} to MnO_4^- mole ratio from the balanced chemical equation to calculate the number of moles of Fe^{2+} in the sample.

$$8H^+_{(aq)} + 5Fe^{2+}_{(aq)} + MnO_4^-{}_{(aq)} \rightarrow 5Fe^{3+}_{(aq)} + Mn^{2+}_{(aq)} + 4H_2O_{(l)}$$

Moles of Fe^{2+}: ________________

Mass of Fe^{2+} in sample

Use the moles of Fe^{2+} and the molar mass of Fe to calculate the mass of Fe^{2+} in your sample. (The loss of the 2 electrons is negligible compared to the mass of an iron atom, so the mass of an ion is experimentally the same as the mass of the atom.)

Mass of Fe^{2+} in sample: ________________

Experimental percent mass of Fe^{2+}

Use your experimental mass of Fe^{2+} and the mass of $Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O$ that you weighed out in step 2 of the procedure to calculate the experimental percent mass of Fe^{2+} in your sample.

$$\text{Percent Mass} = \left(\frac{\text{Mass of Element (or ion) in compound}}{\text{Total Mass Compound}}\right) \times 100$$

Experimental percent mass of Fe^{2+} in sample: ________________

Actual percent mass of Fe^{2+} in $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$:

Use the molar masses of Fe^{2+} and $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$ to calculate the actual percent mass of Fe^{2+}.

$$\text{Percent Mass}=\left(\frac{\text{Mass of Element (or ion) in compound}}{\text{Total Mass Compound}}\right)\text{ x }100$$

Actual percent mass of Fe^{2+} in $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$: _______________

Percent error in mass percent of Fe^{2+} in $Fe(NH_4)_2(SO_4)_2{\bullet}6H_2O$:
Calculate the percent error (see experiment 1).

Percent error: _______________

Experiment 4 Questions:

Submit as part of your informal report

1. (a) What is the oxidation number of manganese at the start of the titration? _______________

   (b) What is the oxidation number of manganese at the end of the titration? _______________

   (c) Is manganese oxidized or reduced during the titration? Briefly explain your answer.

2. In acid/base titrations, an indicator is often added which changes color to indicate the endpoint of a titration. Why is it not necessary to add an indicator for this titration?

3. If you add too much titrant and overshoot your endpoint, would this mistake make your experimental mass of Fe too high or too low? Briefly explain your answer.

Experiment 4: Prelab Worksheet

(Submit through Brightspace before coming to lab)

Name: ______________________________ Date: ________ Section: ________ Grade: ________

All information needed to complete this worksheet can be found in the pre-lab information and calculations sections of the lab manual. Read this introductory material first!

- Record all values with the correct number of significant figures and units.
- Place all answers on the line when provided.
- Show calculations for any numerical answers; **work must be shown to receive credit**.
- See any 102 TA via webex before your prelab is due if you have any questions.
- Each question is worth 2 points.

Use the following redox reaction to answer questions 1-4:

$$MnCl_4\,(aq) + 2Fe(s) \rightarrow 2FeCl_2(aq) + Mn(s)$$

1) What are the oxidation numbers for the Mn and Fe on the reactant side of the equation?

Mn ________________________

Fe ________________________

2) Identify the oxidizing agent in the equation. Oxidizing agent:________________________

3) Write the half-reaction associated with the reduction.

____

4) How many electrons are transferred in the reaction?

____

5) Calculate the molar mass of the $MnCl_4$.

____

6) Calculate the mass percent of the cation in the $MnCl_4$.

____

7) An excess of solid iron is reacted with 0.25g $MnCl_4$ according to the following reaction:

$$MnCl_4\,(aq) + 2Fe(s) \rightarrow 2FeCl_2(aq) + Mn(s)$$

How many moles of solid manganese can be produced from the given amount of salt in the reaction above? Assume the salt is the limiting reagent! Show **<u>all</u>** steps in this calculation!

8) What is the term for the piece of equipment used to measure volume in a titration?

9) Based on the SDS information given in the lab manual, briefly explain the hazards of phosphoric acid.

10) If you return dirty equipment to your drawer
   (a) Can the next person using your drawer report it and get a replacement from the stockroom?
   (b) **Will you be charged for this equipment?**

(a) ________________________

(b) ________________________