AP Chem Lab Vulgate: Determination of Iron by Redox Titration

Equipment & Chemicals:

Equipment	Chemicals Used per group
ring stand	potassium permanganate
Buret	ferrous ammonium sulfate
buret clamp	6 M H_2SO_4
2-250 mL flasks	iron unknown sample
100 mL graduated cylinder	

Procedure:

Part 1: Preparation of the Buret and Flasks

□ 1. The buret must be scrupulously clean. Water should run down the inside of the buret in sheets and should not bead up anywhere on the interior of the glassware. Rinse the buret with tap water.

□ 2. Rinse the buret with distilled water.

□ 3. Rinse the buret with several small (~5 mL) portions of potassium permanganate. Discard the rinsings.

□ 4. Fill the buret with potassium permanganate solution

□ 5. Wash two 250 mL Erlenmeyer flasks with soap. Rinse well with tap water.

□ 6. Rinse the two flasks with 10 mL of distilled water. Discard the rinsings.

Part 2: Preparation of the Iron Standard

□ 1. Using the analytical balance, add approximately 0.5000 to 0.8000 grams of ferrous ammonium sulfate to each of your two flasks. Record the exact amount of FAS taken.

□ 2. Add 25mL of distilled water to each flask. Swirl to dissolve the FAS

□ 3. Add 15mL of 6M sulfuric acid to each flask. Sulfuric acid is added to provide H^+ ions required for the reduction of the permanganate ion.

Part 3: Standardization of the Potassium Permanganate Solution

□ 1. Since potassium permanganate solutions are so intensely colored, it is generally impossible to see the curved meniscus that the solution surface forms in the buret. In this case, it is acceptable to make your liquid level readings at the point where the top surface of the permanganate solution comes in contact with the wall of the buret.

□ □ 2. Record the initial volume of the permanganate solution. Begin to add the permanganate solution to the iron sample. Red streaks may be visible in the sample until the permanganate has had a chance to mix with and react with the iron(II) present.

□ □ 3. Continue to add permanganate solution *slowly* while swirling your flask. The endpoint is the first appearance of a permanent, pale pink color. Record the buret volume at the endpoint.

□ 4. Repeat steps 2 and 3 with your second iron sample.

□ 5. Using the mass of FAS titrated and the volume of $KMnO_4$ used, determine the molarity of the potassium permanganate solution. If your two trials have a significant difference in molarity, do a third trial.

Part 4: Analysis of the Iron Unknown

□ 1. You will be given an unknown iron sample. The sample will contain enough iron salt to complete two trials. Record the sample ID number.

□ 2. Put half of your iron sample in each of your two clean Erlenmeyer flasks. Using the analytical balance, record the mass of the sample used in each flask.

□ 3. Add 25mL of distilled water and 15mL of 6M sulfuric acid to each flask. Be sure to remember to add the distilled water and sulfuric acid to the iron unknown samples.

□ □ 4. Titrate the two iron unknown samples as you did earlier. Record initial and final volume of your buret.

□ □ 5. From the volume of potassium permanganate required to titrate each unknown sample, and from the average molarity of the $KMnO_4$ solution, calculate the number of moles of potassium permanganate required for each iron sample.

□ □ 6. From the number of moles of potassium permanganate required for titration of each iron sample, calculate the number of moles of iron present in each sample.

□ □ 7. From the number of moles of iron present in each sample and the molar mass of iron, determine the mass of iron present in each unknown sample.

□ □ 8. From the mass of the iron present in each sample, and from each sample's mass, calculate the percent iron in each sample.

□ 9. Calculate the average percent iron by mass in your unknown sample.

Data Tables:

Standardization of the $KMnO_4$ Solution

	Sample 1	Sample 2
Mass of FAS taken, g		
Moles of FAS present, mol		
Moles of $KMnO_4$ present, mol		
Initial $KMnO_4$ volume, mL		
Final $KMnO_4$ volume, mL		
Volume $KMnO_4$ used, mL		
Molarity of $KMnO_4$ solution, M		
Mean molarity of the $KMnO_4$ solution, M		

Analysis of Unknown

	Sample 1	Sample 2
Sample ID		
Mass of unknown taken, g		
Initial $KMnO_4$ volume, mL		
Final $KMnO_4$ volume, mL		
Volume $KMnO_4$ used, mL		
Moles of $KMnO_4$ present, mol		
Moles of iron present, mol		
Mass of iron present, g		
% of iron present, %		
Mean % iron present, %		

Name _____ AP Chem ____/____/____

AP Chem Virtual Lab Addendum to Redox Titration Lab

The following is a virtual lab of a redox titration. Each student must complete

1. Enter the following web address: http://www.chem.iastate.edu/group/Greenbowe/sections/projectfolder/flashfiles/redoxNew/redox.html
2. Click on Select the Reaction (in the upper left hand corner)
3. Choose $K_2Cr_2O_7$ as your oxidizing agent.
4. You will be given the standardized molarity of your titrant.
5. Add titrant using the slider on the right side of the page.
6. As the color change from peach to light blue begins to occur, add the titrant drop-wise.
7. If you add too much titrant, click repeat and you will be able to start again using the same molarity.
8. Once the blue color remains, stop adding titrant.
9. The total volume of titrant added is shown to the left of the burette.
10. Using the concentration of $Cr_2O_7^{2-}$, the total volume of $Cr_2O_7^{2-}$ used and the volume of the Sn^{2+} solution, calculate the concentration of the Sn^{2+} solution.
11. Enter your answer using the proper number of significant figures.
12. Click OK
13. If your answer is correct it will say so.
14. If you are correct, print the page, write your name on it and hand in at the end of class. If you are incorrect, try again until you enter a correct result.
15. Close your browser.
16. Staple your on-line results to this paper and hand it in by the end of the period. All info MUST be completed.

Each student must do his/her own virtual lab. Multiple printings of the same virtual lab will not be given credit. This assignment will count for 10 points. (6 points for the correct result. 4 points for the correct answers to the questions below.) You can earn an additional 5 extra credit points if you complete the assignment for a second time using either of the other two oxidizing agents. The virtual lab and extra credit is due at the end of the period. I encourage you to do this assignment prior to the lab.

1. Write the balanced oxidation half reaction:

   Required:

   Extra Credit:

2. Write the balanced reduction half reaction:

   Required:

   Extra Credit:

3. Which substance is oxidized?

   Required:

   Extra Credit:

4. Which substance is reduced?

   Required:

   Extra Credit:

Pre-lab Questions:

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

The mass percent of iron in a soluble iron(II) compound is measured using a titration based on the balanced equation above.

(a) What is the oxidation number of manganese in the permanganate ion, MnO_4^- (aq)?

(b) Identify the reducing agent in the reaction represented above.

The mass of a sample of the iron(II) compound is carefully measured before the sample is dissolved in distilled water. The resulting solution is acidified with H_2SO_4(aq). The solution is then titrated with MnO_4 (aq) until the end point is reached.

(c) Describe the color change that occurs in the flask when the end point of the titration has been reached. Explain why the color of the solution changes at the end point.

(d) Let the variables g, M, and V be defined as follows:

g = the mass, in grams, of the sample of the iron(II) compound

M = the molarity of the MnO_4^-(aq) used as the titrant

V = the volume, in liters, of MnO_4^- (aq) added to reach the end point

In terms of these variables, the number of moles of MnO_4^- (aq) added to reach the end point of the titration is expressed as M x V. Using the variables defined above, the molar mass of iron (55.85 g mol^{-1}), and the coefficients in the balanced chemical equation, write the expression for each of the following quantities.

(i) The number of moles of iron in the sample

(ii) The mass of iron in the sample, in grams

(iii) The mass percent of iron in the compound

(e) What effect will adding too much titrant have on the experimentally determined value of the mass percent of iron in the compound? Justify your answer.

Post-lab Questions

Oxalic acid, $H_2C_2O_4$, is a primary standard used to determine the concentration of potassium permanganate, $KMnO_4$, in solution. The equation for the reaction is as follows.

$2\ KMnO_4(aq) + 5\ H_2C_2O_4(aq) + 3\ H_2SO_4(aq) \rightarrow 2\ MnSO_4(aq) + 10\ CO_2(g) + 8\ H_2O(l) + K_2SO_4(aq)$

A student dissolves a sample of oxalic acid in a flask with 30 mL of water and 2.00 mL of 3.00 M H_2SO_4. The $KMnO_4$ solution of unknown concentration is in a 25.0 mL buret. In the titration, the $KMnO_4$ solution is added to the solution containing oxalic acid.

(a) What chemical species is being oxidized in the reaction?

(b) What substance indicates the observable endpoint of the titration? Describe the observation that shows the endpoint has been reached.

(c) What data must be collected in the titration in order to determine the molar concentration of the unknown $KMnO_4$ solution?

(d) Without doing any calculations, explain how to determine the molarity of the unknown $KMnO_4$ solution.

(e) How would the calculated concentration of the $KMnO_4$ solution be affected if 40 mL of water was added to the oxalic acid initially instead of 30 mL? Explain your reasoning.