Experiment 18 Chemistry 101

Redox Titration:
Determination of the Percentage of Iron in a Sample

INTRODUCTION

The concentrations of redox-active species can be determined by redox titrations. In a redox titration, a measured sample of the unknown is titrated against a standard solution of a substance that will oxidize or reduce the unknown. In the present experiment you will take a sample containing iron, add acid to dissolve it [thereby converting all the iron to iron(II)], then use a solution containing permanganate ion, MnO_4^-, to oxidize this Fe^{2+} to Fe^{3+} ion. The percent of iron in the sample will be calculated from the amount of permanganate needed to oxidize fully all the Fe^{2+} ions.

A solution of permanganate ion in sulfuric acid efficiently oxidizes Fe^{2+} to Fe^{3+}

$$MnO_4^- + 5\ Fe^{2+} + 8\ H^+ \rightarrow Mn^{2+} + 5\ Fe^{3+} + 4\ H_2O$$

The permanganate ion acts as its own indicator, as MnO_4^- is highly colored while Mn^{2+} is essentially colorless. The product of oxidation, the Fe^{3+} ion, is itself, slightly colored. To avoid any possible interference with the equivalence point determination a little phosphoric acid, H_3PO_4, is added so as to complex Fe^{3+} to a completely, colorless ion. You will have 3 hours to complete this assignment; lab and calculations, so come prepared!

PROCEDURE

I. Standardization of permanganate solution

Use distilled water at all times throughout the experiment.

(1) Weigh three clean dry labeled 125 mL Erlenmeyer flasks on an analytical balance. Place about 0.135 grams of oxalic acid dihydrate, $H_2C_2O_4 \cdot 2H_2O$, into each of the three separate flasks and reweigh the flasks containing the acid.

(2) Set up a buret with $KMnO_4$ solution to be standardized by titration.

(3) Dissolve each acid sample in about 25 mL of distilled water. Again don't mix up the samples. Take <u>one</u> flask and add 1-2 mL of concentrated sulfuric acid.

- **CAUTION! Concentrated sulfuric acid is dangerous; don't spill or splash any. *Always slowly add acid to water,* never *the other way around.***

(4) The solution to which the acid has been added should get quite warm, but, since the titration is to be done at elevated temperatures to prevent side reactions, this is desirable. Heat the solution further to 70°C; during the titration the solution should be kept between 60 and 80°C.

(5) Read the level in the permanganate buret (to hundredths of an mL) -- the initial reading -and then add the solution slowly from the buret into the flask with the warmed acid sample *with constant stirring.* The equivalence point is the first appearance of a pink color (excess MnO_4^-) that lasts, with stirring, for 30 seconds. When this is obtained, read the buret again -- the final reading.

(6) Take a second flask with oxalic acid in it and add 1-2 **mL** of concentrated sulfuric acid. Repeat steps 7 and 8 above with this sample. Do a third trial with the third flask in the same manner.

II. Determination of iron

Have at least 10 mL of 85% H_3PO_4 ready for use in step (5).

(1) Weigh out accurately three samples of your unknown mixture containing iron and put each sample in a separate, clean (but not necessarily dry) Erlenmeyer flask. The amount of the sample should be around 1g. *NOTE: new weighing method:* First weigh the vial containing the unknown iron sample. Use the centigram balance to approximate the weight of iron sample in the flask, then, on the analytical balance, re-weigh the vial.

(2) Fill a buret with standardized $KMnO_4$ solution.

(3) Dilute 3 M H_2SO_4 (called dilute sulfuric acid) to 1 M H_2SO_4 (acid into water, never the other way). Stir well.

(4) Put one-third of this 1 M H_2SO_4 solution (50 mL) into *only one* of your Erlenmeyer flasks and dissolve the iron sample quickly and completely. Take an initial reading of the buret and, with stirring, as speedily as possible, start titrating by slowly adding the permanganate solution to the acidified sample.

(5) When the solution turns a light yellow color, add 3 mL of the previously readied 85% H_3PO_4***, and continue immediately***.

(6) Continue adding the permanganate with stirring. The equivalence point of the titration is the first appearance of a pink color (excess MnO_4^- that lasts, with stirring, for 30 seconds). When this occurs take the final reading of the buret.

(7) Repeat steps (4), (5) and (6) for the second sample.

(8) Repeat steps (4), (5) and (6) for the third sample.

CALCULATIONS

I. Standardization

(1) From the measured mass of the oxalic acid samples that you used, calculate the number of moles of oxalic acid in each case. Remember the oxalic acid was weighed out as a dihydrate.

(2) Write a balanced oxidation-reduction equation for the reaction of oxalic acid with potassium permanganate in an acidic solution then, from the indicated molar ratio, calculate how many moles of MnO_4^- must have been used in each of the three titrations. The products are carbon dioxide and manganese(II) ion.

(3) With the calculated number of moles and the measured volume of solution used, calculate three values for the molarity of the permanganate solution. Report an average molarity.

Table 1 Weighings for Oxalic Acid

	Trial 1	Trial 2	Trial 3
Mass of flask			
Mass of flask + oxalic acid			
Mass of oxalic acid			
Moles of oxalic acid used			

Table 2 Standardization of permanganate

	Trial 1	Trial 2	Trial 3
Final buret reading (mL)			
Initial buret reading (mL)			
Volume (in mL) of permanganate used			
Molarity of permanganate (mol/L)			
Average molarity (mol/L)			

II. Determination of iron **UK # _____**

(1) With the known molarity of the permanganate solution and the measured volumes used in the titration, calculate the number of moles of permanganate used in each of the trials.

(2) Write a balanced oxidation-reduction equation for the reaction of iron(II) with permanganate in an acidic solution and, from the indicated molar ratio, calculate how many moles of iron were in each of your weighed out samples.

(3) Calculate how many grams of iron were in each of your weighed out samples and then what mass percent of iron was present in each case. Report an average mass percent of iron in the unknown mixture.

Table 3 Weightings for iron sample

	Trial 1	Trial 2	Trial 3
Mass of vial (start)			
Mass of vial after iron sample is removed			
Mass of iron sample			

Table 4 Titration of unknown iron sample

	Trial 1	Trial 2	Trial 3
Final buret reading (mL)			
Initial buret reading (mL)			
Volume (in mL) of permanganate used			
Moles of $KMnO_4$ used			
Moles of iron in sample			
Theoretical mass of iron in sample			
% of iron in sample			

Average % of iron in UK # _____ is _____.