Unit 4 – Chapter 4: Types of Reactions **Name _____**

Permanganate Determination of the Iron Sample **Period _____**

Lab Hints:

1. Sodium oxalate sample must be oven-dried for 24 hours before using and then placed in a tightly stoppered vial.
2. All buret readings should be estimated to the 0.01 mL so all calculations can be done with 4 significant figures.
3. Use the analytical balance to mass your samples.

Theory: The standardization of the potassium permanganate solution will be carried out by reacting it with a known mass of sodium oxalate, a substance which serves to bleach out the purple color of the permanganate solution. As long as the oxalate ion is present, the purple ion will be bleached out. As soon as the purple color persists, one has reached the **equivalence point** in the titration: all of the oxalate ion has been used up.

The experiment will be divided into two parts: the first part will consist of determining the exact concentration of the permanganate solution and the second part will be to use this standardization solution to titrate an unknown sample containing Fe and to determine the % Fe.

Procedure:

Part 1: Standardization of the $KMnO_4$ solution

1. Weigh out to the nearest 0.1 mg about 0.15 g of $Na_2C_2O_4$ into a clean and rinsed 250 mL flask. Dissolve the sodium oxalate in 75 mL of 0.75 *M* H_2SO_4. Heat the solution to 70.0^0 C. Keep it above 60.0^0C.
2. Rinse a previously-cleaned buret with distilled water followed by **three** separate 3-4 mL rinsings of the $KMnO_4$ solution. Fill the buret with distilled water above the zero mark and drain some through the tip to ensure it too is rinsed. Be sure to keep the stopcock which has been slightly lubricated pushed all the way in.
3. Since the purple color of the solution obscures the meniscus, all readings should be made from the flat, top surface of the liquid. Sight from eye level by raising or lowering the buret and use a white background.
4. Titrate the oxalate and acid solution with the permanganate, adding the solution from the buret to the flask slowly and with constant swirling. Stop the titration when the first pink color persists for 5 seconds. Try to titrate until one drop is just sufficient to give the solution a light pink color which persists upon swirling. Record the final buret reading.
5. Repeat until two consistent concentration values are obtained or a reasonable average is obtained. (We will do this by finding the average for the class).
6. Four significant digits are required in your calculations.

Part 2: Determination of % Fe in an Unknown Sample

1. Mass accurately two samples of your Fe unknown, about 0.3 to 0.4 g each for the two trials.
2. Prepare the titration mixture as follows:
    a. Pour about 75 mL of distilled water in a 250-mL flask and add 9.0 mL of 6 *M* HCl, 4 mL of concentrated H_2SO_4, and 5 mL of 6 *M* H_3PO_4. (Use a graduated cylinder).
    b. Add about as much $NaHCO_3$ as you could cover a dime with to the reaction mixture, a little at a time. This serves to blanket the reaction mixture with CO_2 and minimize the oxidation of Fe^{2+} by oxygen in the air.
3. Add one of the massed samples to the titration mixture, stir it thoroughly and titrate to the endpoint. When the pink color remains for 5 seconds, the Fe^{2+} has been used up according to the reaction: $\mathbf{MnO_4^- + Fe^{2+} \rightarrow Mn^{2+} + Fe^{3+}}$
4. Repeat for the second trial.
5. Drain the buret, fill it with distilled water and drain, and fill it with sodium oxalate solution and drain again. The purpose of the sodium oxalate solution is to bleach out any traces of the purple solution which may be in the buret. Rinse again with tap water and distilled water.