Experiment 2
Analysis of an Iron Coordination Complex

Introduction:

In the previous experiment you synthesized what you believe to be $K_3[Fe(C_2O_4)_3]{\cdot}3H_2O$. In this laboratory your goal is to obtain some evidence that the complex that you made was in fact this. The analysis is based on a series of oxidation/reduction reactions from which the Fe^{3+}:$C_2O_4^{2-}$ mole ratio will be determined. The analysis makes use of deep color of $KMnO_4$ .The Mn^{7+} in MnO_4^- is reduced in the titration to Mn^{2+} (a colorless complex), thus no additional indicator is needed to determine the equivalence point.

Analysis of $K_3[Fe(C_2O4)_3]{\cdot}3H_2O$:

The purpose of this part of the laboratory is to determine the molar ratio of $C_2O_4^{2-}/Fe^{3+}$, from which the number of moles of K^+ can readily be determined. We shall not determine directly that this compound is a trihydrate.

In this volumetric analysis, the oxalate ion ($C_2O_4^{2-}$) in the compound is titrated with the ~0.02M $KMnO_4$ solution. The chemical reaction that takes place is shown in Equation 1.

$$2\ KMnO_4 + 5\ K_2C_2O_4 + 8\ H_2SO_4 = 2\ MnSO_4 + 10\ CO_2 + 8\ H_2O + 6\ K_2SO_4 \qquad \text{Eq. 1}$$

After the $C_2O_4^{2-}$ has been titrated with MnO_4^-, the resulting solution contains, among other species, Fe^{3+}. In order to determine the moles of Fe^{3+} ion in solution using the standard $KMnO_4$, it is first necessary to reduce the Fe^{3+} to Fe^{2+}. This is accomplished by using zinc metal. The chemical reaction that takes place is:

$$\begin{array}{rcll} (Fe^{3+} + e^- & = & Fe^{2+}) & \times 2 \\ Zn & = & Zn^{2+} + 2e^- & \\ \hline 2\ Fe^{3+} + Zn & = & 2\ Fe^{3+} + 3\ OH^- & \end{array} \qquad \text{Eq. 2}$$

The moles of Fe^{2+} formed in Equation 2 are now determined by further titration of this same solution with the standard $KMnO_4$. The chemical reaction that now takes place is:

$$\begin{array}{rcll} (Fe^{2+} & = & Fe^{3+} + e-) & \times 5 \\ MnO_4^- + 8\ H^+ + 5\ e^- & = & Mn^{2+} + 4\ H_2O & \\ \hline 5\ Fe^{2+} + MnO_4^- + 8\ H^+ & = & 5\ Fe^{3+} + Mn^{2+} + 4\ H_2O & \end{array} \qquad \text{Eq. 3}$$

Thus using Eq. 3, the moles of Fe^{2+} can be determined which in turn is equal to the moles of Fe^{3+} (Eq. 2) in the complex sample.

Experimental Procedure

Analysis of $K_3[Fe(C_2O_4)_3]{\cdot}3H_2O$:

1. Weigh two samples (0.200 - 0.240g) of your $K_3[Fe(C_2O_4)_3]{\cdot}3H_2O$ into two clean 125-mL Erlenmeyer flask.

2. Dissolve each sample in 35mL of distilled water. Add 10mL of 6M H_2SO_4 and stir thoroughly. Warm gently to 80°C while preparing the buret for titration.

3. Obtain ~150mL of ~0.02M $KMnO_4$ solution. Record the exact molarity in your notebook. Rinse the buret with a small portion of the solution and then fill the buret, being sure the tip is filled and there are no trapped air bubbles. Record the initial volume reading. Remember to read the top of the meniscus since you cannot see through the solution.

4. Start adding the $KMnO_4$ solution to the flask with constant swirling. As the pink color remains longer, slow the addition of $KMnO_4$ to dropwise. The endpoint has been reached when a pinkish orange coloration persists. Be careful! One drop may give you the endpoint. ***Save this solution,*** and repeat the titration using the second sample. (The color of this solution at the endpoint is due to the pink color of MnO_4^- and the yellow color of Fe^{3+})

Save both solutions after the endpoint has been reached.

5. ***In the fume hood***, add five small spatula of zinc dust to each solution. Gently heat one of the solutions to boiling while stirring constantly with a glass rod to prevent the reaction from becoming too vigorous. If the solution boils over then your analysis to date is rendered useless. Continue heating for ten minutes by which time all of the yellow should have disappeared.

6. Set aside and let cool while repeating step 5 with your second sample.

7. Gravity filter this solution into a fresh 125-mL Erlenmeyer flask. Rinse the original flask with approximately 10-mL of distilled water and gravity filter this into the same Erlenmeyer. This should yield a quantitative transfer of the Fe^{2+} ions.

8. Heat the solution to 80°C and titrate immediately with the $KMnO_4$ solution. **CAUTION!** Since this titration does not require a large volume of the $KMnO_4$ solution, start dropwise, swirl the flask constantly. Repeat steps 6 - 8 with the second titration flask.

9. Calculate the moles of $C_2O_4^{2-}$ present from the molarity and volume of $KMnO_4$ solution. See Equation 1.

10. Calculate the moles of Fe^{3+} present. See Equation 3.

11. Calculate the ratio of moles of $C_2O_4^{2-}$ to moles of Fe^{3+}.

12. Rinse your buret using distilled water from your wash bottle. With the stopcock open (over the sink of course!) continue rinsing until the water flowing from the tip is clear. Have your buret checked by your TA before returning it to the bubble wrap by the window in your lab.

Some Useful Molar Masses

$K_2C_2O_4$: 166.2 g.mol^{-1} $Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$: 392.1 g.mol^{-1} $K_3[Fe(C_2O_4)_3]{\cdot}3H_2O$: 491.1 g.mol^{-1}

Write Up:

For this experiment the focus of the laboratory report is on the 'Data Collection', 'Calculations' and 'Discussion' portion of a laboratory notebook. There is no need this week to write an actual procedure, we will assume that it went according to the given procedure with no deviations.

Data Collection:

Whenever possible data should be recorded in tables. This makes it much easier for a colleague to get to the actual measurements made. In this experiment your are determining the moles of $C_2O_4^{2-}$ and Fe^{3+} that is in a sample of your complex. A good way to record this is to use two tables one for each analysis similar to that shown below:

$C_2O_4^{2-}$ Analysis	**Sample 1**	**Sample 2**
Molarity of $KMnO_4$		
Weight of Sample		
Final Buret Reading		
Initial Buret Reading		
Volume of $KMnO_4$ dispensed.		
Moles of $KMnO_4$		
Moles of $C_2O_4^{2-}$		

Calculations:

In your 'Calculations' you should show one clear sample for each calculation that you do. For the above table one sample of how you determined the moles of $KMnO_4$ that you added, and one for how you determined moles of $C_2O_4^{2-}$. The same holds for your other table. Finally you should determine the mole ratio of $C_2O_4^{2-}$:Fe^{3+} for both samples and the average mole ratio.

Discussion:

Finally in your discussion you should address whether the analysis that you performed is consistent with your complex being $K_3[Fe(C_2O_4)_3]\cdot 3H_2O$.