Name _____ Block: _____ Date: _____

Chemistry 12

IRON DETERMINATION BY REDOX REACTIONS INVOLVING PERMANGANATE ION

A common laboratory oxidizing agent is the permanganate ion, MnO_4^-, which is usually provided by the compound potassium permanganate, $KMnO_4$. It is especially useful for quantitative redox reactions because the permanganate in solution is an intense purple colour, but when reduced all the way to the 2+ state in Mn^{2+} becomes virtually colourless, thereby acting as its own indicator. A sample of a reducing agent can therefore be titrated with $KMnO_4$ solution, and the faint purple colour that remains even after the solution is swirled makes the completion of the reaction apparent. You may think that $KMnO_4$ will not be highly accurate in showing the completion of the reaction, since it must be left over to be seen as a purple colour. However, its colour is very intense; the 0.020 M solution you will be using is a very dark purple, and its colour can still be detected when 1 mL of the solution is added to 2 L of water, that is, when $[MnO_4^-] = 1.0 \times 10^{-5}$ M. This figure represents only 0.05% of the original concentration, which is certainly accurate enough for most situations.

Potassium permanganate is obtainable in analytical reagent quality, so a solution of it can be made up to an accurate concentration from a known mass of crystals. However, the solution should be freshly prepared because over time, any potassium permanganate solution decomposes to a certain extent, and a brown colouration of MnO_2 appears on the side of the container.

The Mn in the permanganate ion has an oxidation number of 7+; in the Manganese (II) ion it has an oxidation number of 2+. It has other common oxidation states; 6+ in the Manganate ion (MnO_4^{2-}), which is green, and 4+ in manganese dioxide, which is brown. In order to make sure that all the permanganate ion is reduced completely to Mn^{2+} and not some other state, you must follow the instructions in the experiment carefully.

In this titration reaction, the Fe^{2+} ions react with permanganate ions, MnO_4^- and acid H^+.

PURPOSE:

1. To determine the mass of Fe^{2+} in an iron supplement.
2. To perform a redox titration involving $Fe^{2+}(aq)$ and $MnO_4^-(aq)$

MATERIALS:

Equipment:

50 mL buret
Buret clamp & stand
2 x 250 mL beakers
400 mL waste beaker
Funnel
Safety goggles
Lab apron
250 mL volumetric flask
250 mL Erlenmeyer flask
25 ml pipet
Pipet pump
Mortar and pestle

Reagents:

50 mL - Standard solution of $KMnO_4$ (0.0200M)
50 mL - 1.0 M H_2SO_4
1 tablet of iron supplement (ferrous gluconate = $C_{12}H_{24}FeO_{14}$)

CAUTION: Potassium permanganate ($KMnO_4$) solution is a strong irritant, and will stain skin and clothing. Wash any spills with plenty of water.
CAUTION: Sulfuric acid is very corrosive. Do not get any on your skin, in your eyes, or on your clothing. Wash any spills with plenty of water, and call your teacher.

PROCEDURE:

1. Put on your lab apron and safety goggles.
2. Obtain **two** iron supplement tablets.
3. The iron tablet contains chalk powder, sucrose, and other minor ingredients. In order to prepare the tablets for analysis you will need to grind them with a mortar and pestle until it is **finely** ground.
4. Dissolve the powdered tablet in 1.0 M H_2SO_4 in the mortar. Transfer the powdered iron tablet and acid to a 250 mL volumetric flask using a funnel. Use the acid to help you transfer your tablet to the flask. Make up the final volume to 250.0 mL using tap water.
5. Using the funnel, pour about 15 mL of $KMnO_4$ solution into your buret. Rinse and discard.
6. Fill up the buret with the $KMnO_4$, and allow some to drain in order to fill the tip. Read the volume.
7. Using a pipet pump, withdraw about 5 mL of the iron supplement solution, and rinse inside the pipet with it. Discard. Transfer 25.00 mL of the solution to a 250 mL Erlenmeyer flask.
8. Titrate the iron (II) solution with potassium permanganate solution until the mixture has just turned pink.
9. When the purple colour starts to take a longer time to disperse, slow down the addition of the $KMnO_4$ until you add it a drop at a time. On standing, the pink colour will disappear because of secondary reaction; do not add any more $KMnO_4$. Record the volume in the buret when the faint purple colour first stays in the flask.
10. Repeat once or twice if necessary to obtain consistent results (within 0.5 mL). Record your observations in the table below.
11. **Reagent Disposal: Place any unused solutions of $KMnO_4$ and Fe^{2+} in the designated waste containers. Solutions left in the flask after the titrations may be safely rinsed down the sink with copious amounts of water.**
12. Clean all glassware and put away equipment.
13. Wash your desk top.
14. Wash your hands with soap and water!

OBSERVATIONS:

$[KMnO_4]$ = _____

Table 1: Volume of $KMnO_4$ to React with 25.00 mL of Supplement Solution

	Trial 1	Trial 2	Trial 3	Trial 4 (if necessary)
Initial buret reading (mL)				
Final buret reading (mL				
Volume $KMnO_4$ required (mL)				
Average Volume $KMnO_4$ (mL)				

QUESTIONS:

1. Combine the two half-reactions to write the equation for the reaction from this lab.

$$Fe^{2+}(aq) \rightarrow Fe^{3+} + e^-$$

$$MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$$

2. Determine the number of moles of MnO_4^- used to react with 25.00 mL of iron supplement solution from the average volume and concentration of MnO_4^-.

3. Calculate the number of moles of Fe^{2+} that reacted (use the reaction equation).

4. Calculate the mass (in milligrams) of iron in the original two iron tablets and then the mass in one tablet. **Remember that you used only a proportion of the tablet in each titration.

CONCLUSION:

What did you determine the mass of iron in the original tablet to be? Compare your value for the mass of iron with the information from the supplier about the composition of each iron tablet. Explain any why these may be different. What are some sources of error that could have contributed to the difference you see? What is the relevance of the technique used in this lab? Are there other situations in which it could be used?