Determination of Oxalate Ion by Permanganate Oxidation

In this experiment you will use a volumetric titration to determine the weight percent oxalate ion in an unknown compound containing oxalate ion. The reduction of permanganate ion to manganese (II) follows the reaction below:

\[
(1) \quad \text{MnO}_4^- \rightarrow \text{Mn}^{2+}
\]

The oxidation of oxalate ion to carbon dioxide gas is as follows:

\[
(2) \quad \text{C}_2\text{O}_4^{2-} \rightarrow \text{CO}_2
\]

The complete, balanced redox reaction between permanganate and oxalate in acidic solution can be determined by adding the two above reactions and balancing. Do this and write the equation below.

\[
(3)
\]

The underlying principle behind a titration is that an equivalence point can be reached at which the number of moles of one reactant is equal to the number of moles of the other reactant. Since this is a redox reaction, the number of electrons must be equivalent. Hence, at the equivalence point, sufficient electrons will have been added to permanganate to completely reduce all of the Mn$^{7+}$ to Mn$^{2+}$, and those electrons would have been produced by oxidizing carbon from +3 in oxalate to +4 in carbon dioxide.

Using the balanced redox equation in (3) above, we can calculate the molar ratio of permanganate to oxalate. This will allow us to determine the number of moles of oxalate in the unknown. This gets around having to deal with electrons (they are accounted for in the redox reaction in #3).

**Materials:**

- Ring stand/ring
- Bunsen burner
- 3 400 mL beakers
- Thermometer
- 0.1 M KMnO$_4$ soln.
- Unknown oxalate compound
- Buret/buret clamp
- Distilled water

**Procedure:**

1. Into 3 separate, clean, dry 400 mL beaker weigh out (accurately!) approximately 0.20 g of unknown oxalate compound. Use caution: oxalate salts are toxic and must be cleaned up immediately.
2. To each beaker add approximately 250 mL of 1.0 M H$_2$SO$_4$.
3. Fill your buret with the 0.1 M KMnO$_4$ solution and note the initial volume in your notebook. Read the volumes from the top of the meniscus each time, and read to 0.01 mL.
4. Warm the oxalate mixture on a ring stand and stir frequently using a thermometer. Warm the mixture to 80-90°C and keep it above 70°C at all times. Once the mixture is warm, begin titrating, stirring frequently.
5. The end point will be reached when an extremely faint pale pink color persists in the solution for 15 seconds. The presence of a deep purple color indicates you have overshot the end point. Note: this purple color is not permanent; if a pink color remains after 15 seconds, use this as the end point.
6. Repeat steps 3-5 for each beaker.
7. Calculate the % oxalate ion by mass in the unknown sample. Report your average mass %. Repeat if the standard deviation of your trials is more than 0.3.

**Questions:**

1. Why is the oxalate solution acidified?
2. If 0.5468 g of sodium oxalate requires 33.23 g of a KMnO$_4$ solution to reach the end point, what is the molarity of the KMnO$_4$ solution?
3. Calculate the percent oxalate in the following: K$_3$C$_2$O$_4$ and K$_3$[Al(C$_2$O$_4$)$_3$]·3H$_2$O
4. How many g of KMnO$_4$ are needed to prepare 3 L of a 0.05 M solution?
5. Write a balanced redox reaction for the reaction between permanganate ion and iron (II) in acidic solution. Iron is oxidized to iron (III).