11

Potentiometric Titration of Unknown Hydrogen Peroxide (Buret)

One method of determining the concentration of a hydrogen peroxide, H_2O_2, solution is by titration with a solution of potassium permanganate, $KMnO_4$, of known concentration. The reaction is oxidation-reduction and proceeds as shown below, in net ionic form.

$$5\ H_2O_2\ (aq) + 2MnO_4^-\ (aq) + 6\ H^+\ (aq) \rightarrow 5\ O_2\ (g) + 2\ Mn^{2+}\ (aq) + 8\ H_2O\ (l)$$

In this experiment, you will use an ORP (Oxidation-Reduction Potential) Sensor to measure the potential of the reaction. Your data will look like an acid-base titration curve. The volume of $KMnO_4$ titrant used at the equivalence point will be used to determine the concentration of the H_2O_2 solution. Your sample of H_2O_2 will be of unknown concentration. You will only have 2 hours to complete this experiment with calculations, so come prepared.

OBJECTIVES

In this experiment, you will

- Conduct the potentiometric titration of the reaction between unknown concentration of hydrogen peroxide and potassium permanganate.
- Measure the potential change of the reaction.
- Determine the concentration of the hydrogen peroxide solution.

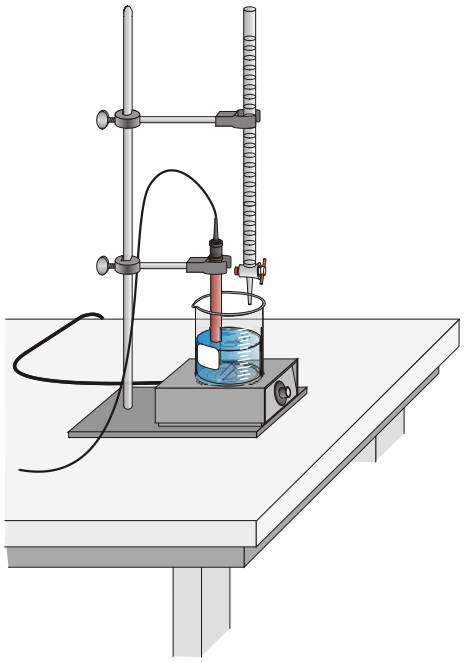

Figure 1

You will conduct the titration in a conventional manner. You will deliver volumes of MnO_4^- titrant from a buret. You will enter the buret readings manually to store and graph each potential-volume data pair.

MATERIALS

LabQuest
LabQuest App
Vernier ORP Sensor
UK hydrogen peroxide, H_2O_2, solution
0.020 M potassium permanganate, $KMnO_4$, solution in H_2SO_4
4.5 M sulfuric acid, H_2SO_4, solution
18M H_2SO_4
50-mL buret
Buret clamp
250 mL beaker
magnetic stirrer
stirring bar or Microstirrer
wash bottle
distilled water
ring stand
utility clamp
two 10 mL pipets and pump
two 100 mL graduated cylinders

Measuring Volume Using a Buret

1. Obtain enough 0.02 M KMnO4 for about 6 total trials and then standardize it with approximately 0.135 g of oxalic acid dihydrate. Hints: (1) How many trials do you need to do to standardize? (2) Write the balanced redox reaction between oxalate and permanganate. Record your results in your lab notebook.

a. Dissolve each acid sample in about 25 mL of distilled water. Take one 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.***

b. 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.

c. 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.

2. Prepare an acidified and diluted hydrogen peroxide, H_2O_2, solution for the titration.
   a. Measure out precisely 10.0 mL of the hydrogen peroxide solution and add 90.0 mL of distilled water. Mix the dilute H_2O_2 thoroughly.
   b. Measure out precisely 10.0 mL of the dilute H_2O_2 solution. Add 25 mL of distilled water and 10 mL of 4.5 M sulfuric acid, H_2SO_4, solution. **CAUTION:** *H_2SO_4 is a strong acid, and should be handled with care.*
   c. Transfer the solution to a 250 mL beaker.
3. Place the beaker of H_2O_2 solution on a magnetic stirrer and add a stirring bar. If no magnetic stirrer is available, stir the mixture with a stirring rod during the titration.
4. Connect the ORP Sensor to LabQuest and choose **New** from the **File** menu. If you have an older sensor that does not auto-ID, manually set up the sensor.

5. Set up a ring stand, a buret clamp, and a buret to conduct the titration (see Figure 1). Rinse and fill the 50 mL buret with 0.020 M MnO_4^- solution. **CAUTION:** *Handle the$KMnO_4$ solution with care; it has been mixed with H_2SO_4, which can cause painful burns if it comes in contact with the skin.*

6. Use a utility clamp to suspend the ORP Sensor on the ring stand, as shown in Figure 1. Position the ORP Sensor so that its tip is immersed in the H_2O_2 solution but does not interfere with the movement of the magnetic stirring bar. Gently stir the beaker of solution.

7. Set up the data-collection mode.
   a. Change the data-collection mode to Events with Entry.
   b. Enter the Name (Volume) and Units (mL). Select OK.

8. The objective of your first trial is to determine the region of the titration curve near the equivalence point, and not to precisely determine the equivalence point. At the equivalence point, you will see a faint pink color of unreacted MnO_4^- solution.
   a. Start data collection.
   b. Before you have added any of the MnO_4^-solution, select **Keep** and enter **0** as the buret volume in mL. Select OK to store the first data pair.
   c. Add 1 mL of the MnO_4^- titrant. Stir the solution gently at all times. When the potential stabilizes, select **Keep** and enter the current buret reading. Make this reading as precise as possible. Select **OK** to store the second data pair.
   d. Add MnO_4^- solution in 1 mL increments and enter the buret reading after each increment. Continue adding MnO_4^- solution until the potential value remains constant.
   e. **Stop** data collection to view a graph of potential *vs.* volume. Examine the titration curve and estimate the volume of MnO_4^- solution used to reach the equivalence point of the titration. Record this value in your data table for Trial 1.

9. Dispose of the reaction mixture as directed. Rinse the ORP Sensor with distilled water in preparation for the second titration.

10. Repeat the necessary steps to conduct a second titration with a new sample of H_2O_2 solution. You may draw your H_2O_2 sample from the remaining 90 mL of H_2O_2 that you diluted in Step 2a.

11. When you conduct the second titration, carefully add the MnO_4^- solution drop by drop in the region near the equivalence point, so that you can precisely identify the equivalence point of the reaction.

12. Examine your titration data to identify the region where the potential made the greatest increase. The equivalence point is in this region.
    a. To examine the data pairs on the displayed graph, select any data point.
    b. As you move the examine line, the potential and volume values of each data point are displayed to the right of the graph.
    c. Identify the equivalence point as precisely as possible and record the volume of MnO_4^- solution used to reach the equivalence point of the titration for Trial 2.

13. At the direction of your instructor, conduct a third trial. Print a copy of the titration curve for the trial that you intend to use in your data analysis. Print the graph directly from LabQuest, if possible. Alternately, transfer the data to a computer, using Logger *Pro* software.

REPORT SHEET NAME:_____

Code for Unknown Peroxide: _____

Average Molarity of $KMnO_4$: _____

Write the balance chemical equation for the reaction of hydrogen peroxide and potassium permanganate below:

SAMPLE DATA TABLE

	Trial 1	Trial 2	Trial 3
Volume of H_2O_2 solution			
Volume of MnO_4^- solution used at equivalence point (mL)			

DATA ANALYSIS

1. Calculate the moles of MnO_4^- used to reach the equivalence point of the reaction for each trial.

2. Use your answer to question 1, along with the balanced redox equation in the introduction, to calculate the moles of H_2O_2 in the sample of solution for each trial.

3. Calculate the molar concentration of the H_2O_2 solution for each trial.

4. The hydrogen peroxide solution that you tested is sold as a commercial product with a concentration, as described on the label of the container, as 3%. A 3% H_2O_2 solution converts to a molarity of 0.88 M. Compare your experimentally determined molarity of H_2O_2 to commercial hydrogen peroxide. What percentage is your unknown solution?