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The Potentiometric Titration of Hydrogen Peroxide

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 come from a bottle of ordinary, over-the-counter hydrogen peroxide purchased at a grocery or a drug store. The concentration of this product is labeled as 3% mass/volume, which is ~0.9 M.

OBJECTIVES

In this experiment, you will

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

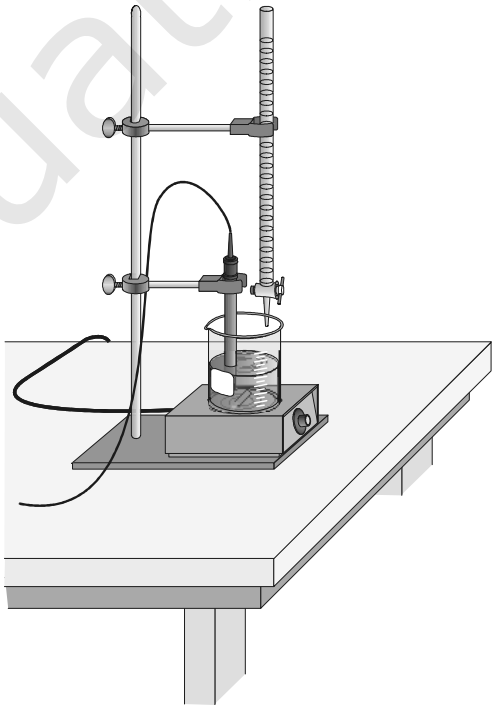

Figure 1

CHOOSING A METHOD

If you choose **Method 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.

If you choose **Method 2**, you will use a Vernier Drop Counter to conduct the titration. MnO_4^- titrant is delivered drop by drop from the reagent reservoir through the Drop Counter slot. After the drop reacts with the reagent in the beaker, the volume of the drop is calculated and a potential-volume data pair is stored.

MATERIALS

Materials for *both* Method 1 (buret) *and* Method 2 (Drop Counter)

Vernier computer interface
computer
Vernier ORP Sensor
3% 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
magnetic stirrer
stirring bar or Microstirrer
wash bottle
distilled water
ring stand
utility clamp
250 mL beaker

Materials required *only* for Method 1 (buret)

50 mL buret
buret clamp
two 10 mL pipets and pump or bulb
two 100 mL graduated cylinders

Materials required *only* for Method 2 (Drop Counter)

Vernier Drop Counter
60 mL reagent reservoir
5 mL pipet and pump or bulb
10 mL, 25 mL and 50 mL graduated cylinders
100 mL beaker
2 small beakers for preparing solutions
a second 250 mL beaker

METHOD 1: Measuring Volume Using a Buret

1. Obtain and wear goggles.

2. Prepare an acidified and diluted hydrogen peroxide, H_2O_2, solution for the titration.
   a. Measure out precisely 10.0 mL of a 3% 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 hydrogen peroxide 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 an ORP Sensor to Channel 1 of a Vernier computer interface. Connect the interface to the computer with the proper cable.

5. Start the Logger *Pro* program on your computer. Open the file "32a Peroxide" from the *Advanced Chemistry with Vernier* folder.

6. 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.*

7. Place a utility clamp on the ring stand to hold the ORP Sensor in place during the titration. 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.

8. You are now ready to begin the titration. 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. Before adding MnO_4^- titrant, click Collect. Once the displayed potential reading has stabilized, click Keep. In the edit box, type **0** (for 0 mL added). Press the ENTER key to store the first data pair.
   b. Add 1 mL of the MnO_4^- titrant. Stir the solution gently at all times. When the potential stabilizes, again click Keep. In the edit box, type the current buret reading. Press ENTER to store the second data pair.
   c. 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.
   d. Click Stop when you have finished collecting data.
   e. 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. When you have completed the titration, 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 trial 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. Use your titration data from the second trial to determine the equivalence point of the reaction. At the direction of your instructor, conduct a third trial.

13. Follow the steps below to find the *equivalence point*, which is the largest increase in potential upon the addition of a very small amount of MnO_4^- solution. A good method of determining the precise equivalence point of the titration is to take the second derivative of the potential-volume data, a plot of $\Delta^2 pH/\Delta vol^2$.
   a. View a plot of the second derivative on Page 3 by clicking on the Next Page button, ⊡.
   b. Analyze the second derivative plot and record the volume of MnO_4^- at the equivalence point.

14. Print a copy of the titration curve for the trial that you intend to use in your data analysis.

METHOD 2: Measuring Volume with a Drop Counter

1. Obtain and wear goggles.

2. Prepare an acidified and diluted hydrogen peroxide, H_2O_2, solution for the titration.
   a. Measure out precisely 5.0 mL of a 3% hydrogen peroxide solution and add 45.0 mL of distilled water. Mix the dilute H_2O_2 thoroughly.
   b. Measure out precisely 5.0 mL of the dilute H_2O_2 solution. Add 15 mL of distilled water and 5 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 100 mL beaker, and set the beaker aside until Step 7e.

3. Connect the ORP Sensor to CH 1 of the computer interface. Lower the Drop Counter onto a ring stand and connect its cable to DIG/SONIC 1.

4. Start the Logger *Pro* program on your computer. Open the file "32b Peroxide (Drop)" from the *Advanced Chemistry with Vernier* folder.

5. Obtain the plastic 60 mL reagent reservoir. Close both valves by turning the handles to a horizontal position. Follow the steps below to set up the reagent reservoir for the titration.
   a. Rinse the reagent reservoir with a few mL of the 0.020 M MnO_4^- solution and pour it into an empty 250 mL beaker. **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.*
   b. Use a utility clamp to attach the reservoir to the ring stand.
   c. Fill the reagent reservoir with slightly more than 60 mL of the MnO_4^- solution.
   d. Place the 250 mL beaker, which contains the rinse MnO_4^- solution, beneath the tip of the reservoir.
   e. Drain a small amount of the MnO_4^-solution into the 250 mL beaker so that it fills the reservoir's tip. To do this, turn both valve handles to the vertical position for a moment, then turn them both back to horizontal.
   f. Discard the drained the MnO_4^- solution in the 250 mL beaker as directed.

6. Calibrate the drops that will be delivered from the reagent reservoir. **Note**: If you are using the stored calibration (28 drops per mL), then skip this step.
   a. On the top row of the Logger *Pro* toolbar, from the Experiment menu, choose Calibrate ▸ DIG 1: Drop Counter.
   b. Proceed by one of these two methods:
      - If you have previously calibrated the drop size of your reagent reservoir and want to continue with the same drop size, select the Manual button, enter the number of Drops/mL, and click OK. Then proceed directly to Step 7.
      - If you want to perform a new calibration, select the Automatic button, and continue with this step.
   c. Place a 10 mL graduated cylinder directly below the slot on the Drop Counter, lining it up with the tip of the reagent reservoir.
   d. Open the bottom valve on the reagent reservoir (vertical). Keep the top valve closed (horizontal).

e. Click the Start button.
f. Slowly open the top valve of the reagent reservoir so that drops are released at a slow rate (~1 drop every two seconds). You should see the drops being counted on the computer screen.
g. When the volume of the MnO_4^- solution in the graduated cylinder is between 9 and 10 mL, close the bottom valve of the reagent reservoir.
h. Enter the precise volume of MnO_4^- solution (read to the nearest 0.1 mL) in the edit box. Record the number of Drops/mL displayed on the screen for possible future use.
i. Click OK. Discard the MnO_4^- solution in the graduated cylinder as directed and set the graduated cylinder aside.

7. Assemble the apparatus.
   a. Place the magnetic stirrer on the base of the ring stand.
   b. Insert the ORP Sensor through the large hole in the Drop Counter.
   c. Attach the Microstirrer to the bottom of the ORP Sensor. Rotate the paddle wheel of the Microstirrer, making sure that it does not touch the bulb of the ORP Sensor.
   d. Adjust the positions of the Drop Counter and reagent reservoir so they are both lined up with the center of the magnetic stirrer.
   e. Lift up the ORP Sensor, and place the 100 mL beaker containing the H_2O_2 solution onto the magnetic stirrer. Lower the ORP Sensor into the beaker.
   f. Adjust the position of the Drop Counter so that the Microstirrer on the ORP Sensor is just touching the bottom of the beaker.
   g. Adjust the reagent reservoir so its tip is just above the Drop Counter slot.
8. Turn on the magnetic stirrer so that the Microstirrer is stirring at a fast rate.
9. You are now ready to begin collecting data. Click Collect. No data will be collected until the first drop goes through the Drop Counter slot. Fully open the bottom valve. The top valve should still be adjusted so drops are released at a rate of about 1 drop every 2 seconds. When the first drop passes through the Drop Counter slot, check the data table to see that the first data pair was recorded.
10. Continue watching your graph to see when a large increase in potential takes place—this will be the equivalence point of the reaction. When this jump in potential occurs, let the titration proceed for a few more milliliters of titrant, then click Stop. Turn the bottom valve of the reagent reservoir to a closed (horizontal) position.
11. Dispose of the reaction mixture as directed.
12. Follow the steps below to find the *equivalence point*, which is the largest increase in potential upon the addition of a very small amount of MnO_4^- solution. A good method of determining the precise equivalence point of the titration is to take the second derivative of the potential-volume data, a plot of $\Delta^2 potential/\Delta vol^2$.
    a. View a plot of the second derivative on Page 3 by clicking on the Next Page button, .
    b. Analyze the second derivative plot and record the volume of MnO_4^- at the equivalence point.
13. Return to the original titration graph. Print a copy of the graph and the data set. If you wish to save the results of the first titration, choose Store Latest Run from the Experiment menu.

14. Repeat the necessary steps to conduct a second titration with a new solution of H_2O_2. Analyze the titration results in a manner similar to your first trial and record the equivalence point. At the direction of your instructor, conduct a third trial.

15. Print a copy of the titration curve of the trial that you intend to use in your data analysis.

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 a commercial product with a concentration, as described on the label of the container, as 3%. As stated in the introductory remarks, a 3% H_2O_2 solution converts to a molarity of 0.88 M. Compare your experimentally determined molarity of H_2O_2 to the label description.

Vernier Lab Safety Instructions Disclaimer

THIS IS AN EVALUATION COPY OF THE VERNIER STUDENT LAB.

This copy does not include:

- **Safety information**
- **Essential instructor background information**
- **Directions for preparing solutions**
- **Important tips for successfully doing these labs**

The complete *Advanced Chemistry with Vernier* lab manual includes 35 labs and essential teacher information. The full lab book is available for purchase at:
http://www.vernier.com/cmat/chema.html

Vernier Software & Technology
13979 S.W. Millikan Way • Beaverton, OR 97005-2886
Toll Free (888) 837-6437 • (503) 277-2299 • FAX (503) 277-2440
info@vernier.com • www.vernier.com