Analysis of Hydrogen Peroxide AP* Chemistry Big Idea 3, Investigation 8 An Advanced Inquiry Lab

Introduction

Hydrogen peroxide is typically regarded as an "environmentally friendly" alternative to chlorine for water purification and wastewater treatment. Hydrogen peroxide readily decomposes in the presence of heat, light and catalysts. The quality of a hydrogen peroxide solution must be regularly checked to ensure its effectiveness. The concentration of hydrogen peroxide can be analyzed by an oxidation–reduction titration with potassium permanganate.

Concepts

- Oxidation-reduction Titration Standardization
- Half-reactions Equivalence point Percent composition

Background

Titration is a method of volumetric analysis—the use of volume measurements to analyze the concentration of an unknown. The most common types of titrations are acid—base titrations, in which an acid, for example, is analyzed by measuring the amount of standard base solution required to neutralize a known amount of the acid. A similar principle applies to oxidation—reduction reactions. If a solution contains a substance that can be oxidized, then the concentration of that substance can be analyzed by titrating it with a standard solution of a strong oxidizing agent.

Oxidation-reduction reactions occur by electron transfer. The balanced chemical reaction can be written as the combination of two *half-reactions*, representing the oxidation reaction and the reduction reaction, respectively. In an overall, balanced redox reaction, the number of electrons lost by the species being oxidized is always equal to the number of electrons gained by the species being reduced.

Potassium permanganate, KMnO4, is a common oxidizing agent used as a titrant in redox titrations. In an acidic solution, the MnO4ion is reduced according to the following unbalanced half-reaction:

 $H_{+}(aq) + MnO_{4-}(aq) \rightarrow Mn_{2+}(aq) + H_{2}O(l)$ Equation 1

Potassium permanganate is not considered a primary standard for analytical purposes. Common impurities include chlorine in the form of chloride and chlorate ions, nitrogen compounds, and sulfur as sulfate. In order to accurately determine the concentration of a KMnO4 solution, it may be titrated against a solution containing a known concentration of iron(II) ions, Fe₂₊. Ferrous ammonium sulfate, Fe(NH4)₂(SO4)₂, serves as a primary standard to titrate the unknown KMnO4 solution. In the corresponding half-reaction, the Fe₂₊ ion is oxidized to Fe₃₊.

$Fe_{2+}(aq) \rightarrow Fe_{3+}(aq)$ Equation 2

For this redox titration, the *equivalence point* occurs when the exact number of moles of MnO₄- ions has been added to react completely with all the Fe₂₊ ions in the solution of the primary standard. The indicator for this titration is the MnO₄- ion itself. The MnO₄- ion is purple in solution and its reduction product, Mn₂₊, is almost colorless. At the endpoint of the titration, the solution changes from colorless to light pink as the last drop of MnO₄- added does not react and keeps its color.

A solution of hydrogen peroxide will be titrated with the standardized potassium permanganate solution to determine the H₂O₂ concentration. The endpoint occurs when the pink color of the MnO₄- ion persists. The unbalanced half-reaction for the oxidation of hydrogen peroxide is:

 $H_2O_2(aq) \rightarrow O_2(g) + H_+(aq)$ Equation 3

Experiment Overview

The purpose of this advanced inquiry investigation is to determine the percent composition of a common "drug store" bottle of hydrogen peroxide through an oxidation–reduction titration with potassium permanganate. The lab begins with an introductory activity to standardize a solution of potassium permanganate by redox titration against a primary standard, ferrous ammonium sulfate. This standardization procedure provides a model for guided-inquiry design of an experiment to determine the percent hydrogen peroxide in a sample. Additional products containing hydrogen peroxide may be analyzed as part of optional extension activities.

Pre-Lab Questions

- 1. Balance the oxidation and reduction half-reactions for the iron(II) ion and permanganate ion, respectively, and write the balanced chemical equation for the overall reaction between Fe2+ and MnO4- in acidic solution.
- 2. How many moles of Fe2+ ions can be oxidized by 0.067 moles of MnO4- ions?

3. A sample of oxalic acid, H₂C₂O₄, is titrated with a standardized solution of KMnO₄. A 25-00 mL sample of oxalic acid required 12.70 mL of 0.0206 M KMnO₄ to achieve a pink-colored solution. The balanced equation for this reaction is shown below.

 $6H_{+}(aq) + 2MnO_{4-}(aq) + 5H_{2}C_{2}O_{4}(aq) \rightarrow 10CO_{2}(g) + 8H_{2}O(1) + 2Mn_{2+}(aq)$

- a. What does the pink color signify in this reaction?
- b. What is the mole ratio of H2C2O4 molecules to MnO4- ions?
- c. How many moles of MnO4- ions reacted with the given amount of H2C2O4?
- d. How many moles of H2C2O4 were present in the sample?
- e. What is the molarity of the H2C2O4 solution?
- f. If the density of the oxalic acid solution is approximately 1.00 g/mL, what is the percentage by mass of oxalic acid in the solution?

Materials

Hydrogen peroxide solution, 3%, 4 mL Erlenmeyer flasks, 250-mL, 3

Ferrous ammonium sulfate, Fe(NH4)2(SO4)2z6H2O, 3 g Graduated cylinders, 10-mL, 2

Potassium permanganate solution, KMnO4, 0.02 M, 80 mL Pipet bulb or pipet filler

Sulfuric acid solution, H2SO4, 3 M, 50 mL Pipet, serological, 1-mL

Water, distilled or deionized Support stand

Balance, 0.001-g precision (shared) Wash bottle

Beakers, 100-mL, 2 Wax pencil

Buret, 50-mL Weighing dishes, 2

Buret clamp

Safety Precautions

Sulfuric acid is corrosive to eyes, skin, and other tissue; always add acid to water, never the reverse. Notify your teacher and clean up all acid spills immediately. Dilute potassium permanganate solution is a skin and eye irritant and strong stain—it will stain skin and clothing. Avoid contact of all chemicals with eyes and skin. Wear chemical splash goggles, chemical-resistant gloves, and a chemicalresistant apron. Wash hands thoroughly with soap and water before leaving the laboratory. Please follow all laboratory safety guidelines.

Introductory Activity

Standardizing Potassium Permanganate Solution

- 1. Obtain approximately 80 mL of the potassium permanganate solution in a 100-mL beaker. Label the beaker.
- 2. Set up a clean 50-mL buret in a buret clamp on a support stand.
- 3. Rinse the buret with approximately 10 mL of distilled or deionized water and then with two 5-mL portions of the potassium permanganate solution, KMnO4 (MnO4-).
- 4. Close the stopcock and fill the buret to just above the zero mark with the MnO4- solution.
- 5. Open the stopcock to allow any air bubbles to escape from the buret tip. Close the stopcock when the liquid level is between the 0- and 10-mL marks.
- 6. Record the precise level of the solution in the buret in an appropriate data table. This is the initial volume of the MnO₄-solution.
- 7. Obtain a mass between 0.4–0.5 g of ferrous ammonium sulfate [Fe(NH4)2(SO4)2z6H2O] in a clean weighing dish using a milligram balance. Record the precise mass in an appropriate data table.
- 8. Measure 10 mL of the 3 M H₂SO₄ solution into a clean 10-mL graduated cylinder. Measure 10 mL of distilled or deionized water into a separate, clean 10-mL graduated cylinder. Add these to a clean 250-mL Erlenmeyer flask. Swirl to mix.
- 9. Add the solid ferrous ammonium sulfate to the flask. Swirl the flask to dissolve the solid. *Hint:* Be sure to transfer all of the solid to the flask. Rinse the weighing dish with distilled water into the flask to capture any lingering solid.
- 10. Position the flask under the buret so that the tip of the buret is within the flask but at least 2 cm above the liquid surface.
- 11. Titrate the ferrous ammonium sulfate solution with the MnO4- solution until the first trace of pink color persists for 30 seconds. Remember to swirl the flask and rinse the walls of the flask with distilled water before the endpoint is reached.

- 12. Record final buret reading as the final volume of the MnO4- solution in an appropriate data table.
- 13. Repeat the standardization titration one more time.

Analyze the Results

Use the collected volume data to determine the precise (accurate) concentration of potassium permanganate. This should be done before moving on to *Guided-Inquiry Design and Procedure*.

Guided-Inquiry Design and Procedure

Determination of Percent Hydrogen Peroxide

Form a working group with other students and discuss the following questions.

- 1. Balance the oxidation-reduction half reactions for hydrogen peroxide and permanganate ion, respectively. Write the net ionic equation for the reaction between MnO4- ions and H2O2 in acidic solution.
- 2. Assuming the density of 3% H2O2 is 1.00 g/mL, what mass of H2O2 is in a 1.00-mL sample? How many moles is this?
- 3. Based on the stoichiometry of the reaction between MnO₄- ions and H₂O₂ in acidic solution, estimate the volume of 0.02 M MnO₄- solution required to titrate a 1-mL sample of 3% H₂O₂.
- 4. In the *Introductory Activity* section, the redox titration was performed in an acidic solution. Research the products of the redox reaction between MnO4- ions and H2O2 in a basic solution. Would these products affect your ability to accurately judge the endpoint of the titration?
- 5. Is it necessary to know the exact volume of: (a) hydrogen peroxide sample added to the flask? (b) rinse water added to the flask during the titration? (c) potassium permanganate solution added to the flask?
- 6. Write a detailed step-by-step procedure for a titration experiment to determine the percent H₂O₂ in a sample. Include all the materials, glassware and equipment that will be needed, safety precautions that must be followed, the concentrations of reactants, etc.
- 7. Review additional variables that may affect the reproducibility or accuracy of the experiment and how these variables can be controlled.
- 8. Carry out the experiment and record the results in an appropriate data table.

Analyze the Results

Use the collected volume data to determine the percentage of hydrogen peroxide in your sample. Assume the density of the hydrogen peroxide solution is 1.00 g/mL.

Opportunities for Inquiry

Hydrogen Peroxide in Household Products

Hydrogen peroxide is found in many common household products, including teeth whitening strips, contact lens cleaner, hair dyes and mouth cleansers. The labels for such products may list the percent hydrogen peroxide in the product. Research a product containing hydrogen peroxide and confirm its use with your teacher. Design an experiment to determine the actual percentage of hydrogen peroxide in each product.