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
- Tiration
- Half-reactions
- Equivalence point
- Standardization
- 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 MnO₄⁻ ion is reduced according to the following unbalanced half-reaction:

\[ \text{H}^+ (aq) + \text{MnO}_4^- (aq) \rightarrow \text{Mn}^{2+} (aq) + \text{H}_2\text{O}(l) \]

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⁺.

\[ \text{Fe}^2+ (aq) \rightarrow \text{Fe}^3+ (aq) \]

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 to determine the concentration by titration with the potassium permanganate solution standardized. The endpoint occurs when the pink color of the MnO₄⁻ ion persists. The unbalanced half-reaction for the oxidation of hydrogen peroxide is:

\[ \text{H}_2\text{O}_2(aq) \rightarrow \text{O}_2(g) + \text{H}^+ (aq) \]

Equation 3

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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 optimal extension activities.

Pre-Lab Questions (Answer questions on separate sheet of paper.)

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 Fe$^{2+}$ and MnO$_4^-$ in acidic solution.

2. How many moles of Fe$^{2+}$ ions can be oxidized by 0.067 moles of MnO$_4^-$ ions?

3. A sample of oxalic acid, H$_2$C$_2$O$_4$, is titrated with a standardized solution of KMnO$_4$. A 25.00 mL sample of oxalic acid required 12.70 mL of 0.0206 M KMnO$_4$ to achieve a pink-colored solution. The balanced equation for this reaction is shown below.

   \[6\text{H}^+(\text{aq}) + 2\text{MnO}_4^-(\text{aq}) + 5\text{H}_2\text{C}_2\text{O}_4(\text{aq}) \rightarrow 10\text{CO}_2(\text{g}) + 8\text{H}_2\text{O}(\text{l}) + 2\text{Mn}^{2+}(\text{aq})\]

   a. What does the pink color signify in this reaction?

   b. What is the mole ratio of H$_2$C$_2$O$_4$ molecules to MnO$_4^-$ ions?

   c. How many moles of MnO$_4^-$ ions reacted with the given amount of H$_2$C$_2$O$_4$?

   d. How many moles of H$_2$C$_2$O$_4$ were present in the sample?

   e. What is the molarity of the H$_2$C$_2$O$_4$ 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
- Ferrous ammonium sulfate, Fe(NH$_4$)$_2$(SO$_4$)$_2$.6H$_2$O, 3 g
- Potassium permanganate solution, KMnO$_4$, 0.02 M, 80 mL
- Sulfuric acid solution, H$_2$SO$_4$, 3 M, 50 mL
- Water, distilled or deionized
- Balance, 0.001-g precision
- Beakers, 100-mL, 2
- Buret, 50-mL
- Buret clamp
- Erlenmeyer flasks, 250-mL, 3
- Graduated cylinders, 10-mL, 2
- Pipet bulb
- Pipet, serological, 1-mL
- Support stand
- Wash bottle
- Wax pencil
- Weighing dishes, 2

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 chemical-resistant 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)₂·6H₂O) 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 acid 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 acid 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.

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 the sample. The concentration of 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 whiteners and mouth rinses. The labels for such products may list the percent hydrogen peroxide containing hydrogen peroxide and confirm its use with your teacher. Design an experiment to determine hydrogen peroxide in each product.

_AP Chemistry Review Questions_

_Integrating Content, Inquiry and Reasoning_

Examine the five reactions shown below and identify those that can be classified as oxidation-reduction reactions:

1. \(2H_3PO_4 + 3Ca(OH)_2 \rightarrow Ca_3(PO_4)_2 + 6H_2O\)
2. \(2Cr + 3Cl_2 \rightarrow 2CrCl_3\)
3. \(C_6H_12O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O\)
4. \(Na_2CO_3 \rightarrow Na_2O + CO_2\)
5. \(2VO^{2+} + Zn + 4H^+ \rightarrow 2V^{3+} + Zn^{2+} + 2H_2O\)

The decomposition of a compound into simpler substances by means of an electrical current is an example of a redox reaction. Write the balanced chemical equation for the electrolytic decomposition of water to include all charges.

6. Write the balanced chemical equation for the electrolytic decomposition of water to include all charges.

7. Balance the following oxidation and reduction half-reactions for the decomposition of water:

\[
\begin{align*}
\text{Oxidation:} & \quad \text{____H}_2\text{O} \rightarrow \text{____O}_2 + \text{____H}^+ + \text{____e}^- \\
\text{Reduction:} & \quad \text{____H}_2\text{O} + \text{____e}^- \rightarrow \text{____H}_2 + \text{____OH}^- 
\end{align*}
\]

8. Explain how the oxidation and reduction half-reactions may be combined to give the balanced equation for the decomposition of water. What happens to the electrons and to the \(H^+\) and \(OH^-\) ions?

The usefulness of metals in structural applications depends on their physical and chemical properties. Metals and metalloids are common materials for metal structures.