HYDROGEN PEROXIDE ANALYSIS

INTRODUCTION

Description

Students determine the percent of hydrogen peroxide in store-bought hydrogen peroxide by titration with potassium permanganate. The potassium permanganate is standardized by students with titration of solid sodium oxalate.

Goals for This Experiment

The goals of this experiment are to have students:

1. perform titrations;
2. work with the concepts of mean, standard deviation and relative standard deviation;
3. report data to the proper number of significant figures;
4. consider the concept of *precision* in measurements;
5. experience quality control concepts in industry; and
6. read and use information from MSDSs.

Recommended Placement in the Curriculum

The Hydrogen Peroxide Analysis lab would be best implemented after the students have had experience with the use of a buret (as a volume measuring device), as well as general statistics. This is a good lab to introduce the skill of titrating.

ANALYSIS OF HYDROGEN PEROXIDE SOLUTION

SCENARIO

You are a recent graduate of Miami University Middletown's associate degree program in Chemical Technology and you have just begun a $29,000-job in quality control for HYPER Corporation, a local manufacturer of hydrogen peroxide solutions. Your job at HYPER is to join a team of technicians who monitor the hydrogen peroxide concentrations on the "3%-line" to ensure that the solutions continuously fall within the company specifications for concentrations of 2.8%–3.2%. (The industrial standard concentration range is 2.5%–3.5%, but your company has established a reputation for meeting even higher standards than the industry as a whole. It will be your job to help them continue to do this.) To monitor the solution concentration, you and your team members must routinely extract samples from the production line and perform analyses similar to that described below.

BACKGROUND

Hydrogen peroxide in aqueous solution can act either as a strong oxidizing agent or as a reducing agent. In moderate to high concentrations it can cause severe burns to skin and mucous membrane. HYPER Corporation markets aqueous hydrogen peroxide in concentrations between 3–90% by weight. For example, 90% solution is used in rocket propulsion, some professional hair bleaching agents contain 6% hydrogen peroxide, and 3% solutions are widely used for bleaching flour, straw, gelatin, and textile fabrics including silk. A 3% hydrogen peroxide solution also has a therapeutic use as a topical anti-infective, antiseptic, and cleansing agent for both humans and animals. HYPER Corporation sells its 3% solution to the plastics industry, to home renovation specialists who use it to remove old paint, to tanners for processing hides, to photography supply houses, to wine makers, and to pharmaceutical companies.

YOUR TASKS

Your tasks will be to perform the analysis of samples of hydrogen peroxide solution from the production line (provided by your instructor), to evaluate the accuracy and precision of the method, and to determine the quality of the manufactured product sold to the company's customers.

In this experiment you will work individually. The other members of the class represent the other members of your team at HYPER. Each team member will do some statistical analysis of his/her own data and some analysis of the pooled data for the team.

SAFETY CONCERNS

Sodium oxalate is an antioxidant, potassium permanganate is an oxidizing agent, hydrogen peroxide can act as either, and sulfuric acid is a strong acid. **Before you begin work on this project**, find out what these terms mean and what special precautions must be taken while using them. The Material Safety Data Sheets (MSDS) will be available on the class bulletin board for you to use or you may them on the following Web sites: http://www.fisher1.com/ or gopher://atlas.chem.utah.edu/11/MSDS. (If you use the Web to find material, be sure to include that in your written record for the experiment.)

PROCEDURE

I. Standardization of Potassium Permanganate Solution

*Potassium permanganate solution can not be prepared accurately to a fixed concentration. Thus, you must first titrate the solution with a known mass of pure, solid sodium oxalate (for which the number of moles can be calculated to four significant figures). This process is called **standardization**. The reaction equation is given below.*

$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$

Procedure for standardization

- Obtain approximately 125 mL permanganate solution from the stock bottle.
- Fill a buret with permanganate solution. Record the initial volume.
- Weigh two 0.7 g samples of dry sodium oxalate ($Na_2C_2O_4$) to the closest 1 mg, and transfer them to clean, labeled 250-mL Erlenmeyer flasks. Record the mass of the samples.
- Add 70 mL 0.75 M sulfuric acid to each flask and swirl to dissolve the solid.
- Use a hot plate to heat one flask's contents to 80-90°C. DO NOT BOIL. You can place a thermometer in the flask to determine the temperature. Rinse the thermometer with a small stream of distilled water from your wash bottle (aimed into your flask) as you remove the thermometer just before titrating. This is necessary to prevent loss of reactant.

CAUTION: Handle the flask with care. **It will be HOT!** Liquid in this temperature range can cause burns. Use tongs or have your instructor show you how to fold a paper towel for use as a protector.

- While the solution is hot, titrate with the permanganate solution. The temperature should not fall below 60°C during the titration. (If it does, the mixture will turn brown and cloudy instead of decolorizing as expected.) Record the final volume of permanganate solution.
- Heat the other sample solution and repeat the titration.

Calculation of the concentration of potassium permanganate solution

⇒ Prepare a table to record answers to the following:

1. Calculate the number of moles sodium oxalate in each sample. The molar mass of sodium oxalate is 134.00 g.
2. Calculate the number of moles of potassium permanganate at the endpoint of each titration.
3. Calculate the molarity of the potassium permanganate solution from each titration (moles permanganate/liter solution).
4. Find the mean of the two molarities.
5. **If** the difference between the two calculated molarities is greater than 5% of the mean, you should titrate another sample and do the necessary calculations. Average all values.
6. Once you have values for the permanganate concentration that you believe are valid, add the individual values to the class data list on the computer. Do this before you continue so that the information will be ready when you are ready to do your data analysis.

II. Hydrogen Peroxide Analysis

In acid solution, the dark purple MnO_4^- oxidizes hydrogen peroxide, forming oxygen gas and very pale pink Mn^{2+} ions. Thus, when a solution of $KMnO_4$ is added dropwise to an acidified solution of hydrogen peroxide, each drop is decolorized until all the H_2O_2 is used up. The next drop of permanganate solution added colors the solution and signals the end-point of the titration.

$$2MnO_4^- + 6H^+ + 5H_2O_2 \rightarrow 5O_2 + 8H_2O + 2Mn^{2+}$$

Procedure for analysis

- Weigh a clean, dry (on the outside) 250-mL Erlenmeyer flask on the balance. Add about 10 mL of the hydrogen peroxide solution provided and weigh it again. Record the mass of the sample solution. Add about 50 mL distilled water. Slowly add 15 mL 3 M sulfuric acid, swirling the flask after each addition.

CAUTION: 3 M sulfuric acid is sufficiently concentrated so that this mixture might become warm. Add the acid to the solution slowly, while swirling. If any acid should spill, wash the area with plenty of water.

- Fill your buret with potassium permanganate solution. Record the initial volume.
- Titrate the acidic hydrogen peroxide solution you just made, swirling continuously. Continue until one drop produces a pink color that lasts for at least 1 minute. The color is more readily seen if there is a piece of white paper under the flask. Record the buret reading.
- Refill the buret. Repeat the titration with a duplicate sample.

Calculation of Peroxide Concentration

⇒ 1. Calculate

- the number of moles of hydrogen peroxide in the first sample,
- the number of grams of hydrogen peroxide in the first sample, and
- the mass percentage of hydrogen peroxide in the first sample of hydrogen peroxide solution.

⇒ 2. Repeat the above calculations for the second sample.

Before you leave the laboratory, make sure you have a copy of the class data for the concentration of potassium permanganate solution, including the mean and standard deviation for the set.

EVALUATING THE QUALITY OF YOUR DATA

Before you start the following tasks, review the section "A Look at *Quality* in Calculations Made From Measurements" in the front part of your lab book. You will find the necessary methods, equations, and an explanation of terms.

In this task you will

- interpret the information given by the mean and standard deviation of the class data for the permanganate concentration,
- evaluate the quality of your values in relation to the class mean,
- judge the quality of the information obtained by the class about the peroxide concentrations in light of the statistical information known, and
- judge the quality of your data as a technician for HYPER Corporation.

Procedure

1. Obtain a copy of the team set of data for the permanganate solution. Record this information as "mean plus/minus standard deviation."
2. Calculate the relative standard deviation of the team mean in percentage notation.
3. Decide whether or not any of the individual points seem to be too different from the remaining values to be considered valid. Make sure you can defend your decision.

Now take a deep breath, stretch, and think about the numbers you have in terms of what they <u>mean</u> to you as a technician working for HYPER Corporation. Reread the introductory material and use the statistics you have calculated to help you decide the answers to the following questions.

1. Would you say the class data set is <u>accurate</u>? <u>precise</u> ? What is your reasoning?

2. Where do your individual results fall with respect to the mean of the class set?

3. Does the hydrogen peroxide concentration you determined for your individual sample fall within the range specified by HYPER Corporation? If not, what reasons could account for this?

4. Let's suppose that HYPER manufactures two H_2O_2 products: one containing 30% hydrogen peroxide by mass and the other containing 3.0% hydrogen peroxide by mass. You are responsible for quality assurance of the 30% product line. Your analysis of a particular lot gives you a mean of 29.5% and standard deviation of 1.8%. A coworker whom you have been training is responsible for the 3.0% line. His analysis of a single lot gives a mean of 2.8% and a standard deviation of 0.4%. Whose analysis indicates the better precision? On what evidence do you base your choice?

HYDROGEN PEROXIDE ANALYSIS

INSTRUCTOR NOTES

Time Required

The laboratory experiment should take between 2–2$^1/_2$ hours. You should check the students' calculations and require more titrations if precision is poor. Students need to be encouraged to perfect their titration technique since it is a basic laboratory skill and will be used throughout the course.

Group Size

Students should work individually during this experiment. This will ensure that each student gains enough experience with titrating.

Materials Needed

per class (20 students):

- 4 L of 0.100 M $KMnO_4$: weigh 63.2 g of potassium permanganate in a 2 L container and bring to volume with distilled water. The solution should then be standardized using the same procedure the students will use (dry sodium oxalate in dilute sulfuric acid). The concentration needs to be determined to at least three significant figures. Students will need this concentration to answer question 1. **DO NOT** label the bottle with the concentration. Tell the students the concentration after they have completed the experiment.
- 75 g of dry sodium oxalate ($Na_2C_2O_4$ should be placed in an oven for 2–3 hours at ~110°C, removed just prior to the beginning of lab and stored in a dessicator.) Label the bottles containing the sodium oxalate as poisonous.
- 1 L of 3 M H_2SO_4: from concentrated H_2SO_4, add 166 mL of concentrated acid to 750 mL of distilled water. Bring to volume with distilled water.
- 2 L of 0.75 M sulfuric acid:
  - ➢ If using concentrated H_2SO_4, add 83 mL of concentrated acid to 1.5 L of distilled water. Bring to volume with distilled water.
  - ➢ If using 3 M H_2SO_4, add 500 mL of 3 M acid to 1,200 mL of distilled water. Bring to volume with distilled water.
- 3 pint-sized bottles of commercial hydrogen peroxide solution (purchased from a supermarket or drug store—this is a 3.0% solution). You may want to combine the bottles before testing to minimize small concentration differences that are possible.
- 8–12 hot plates

per student:

- 50-mL buret
- two 250-mL Erlenmeyer flasks
- 100-mL graduated cylinder
- 10-mL graduated cylinder
- 150-mL beaker

Safety, Disposal, and Special Handling

Review the Material Safety Data Sheets of any chemical used in the experiment for information regarding safety. Dispose of waste according to your local ordinances.

Points to Cover in Pre-lab

- Discuss the scenario with students to make sure they understand the task. You may want to talk about quality control in industry. Permanganate titration of peroxide solutions **is** a standard method of quality analysis in the pharmaceutical industry.
- Check to make sure students know the meanings of **antioxidant** (reducing agent), **oxidizing agent,** and **strong acid** as they pertain to safety. (We use the term *antioxidant* because it is lay language but not usually used in class.) ***Make sure*** students understand safe handling and disposal of materials and what to do if a spill should occur. Warn them about brown spots that appear on the skin if the permanganate solution is spilled on it. Assure students that the spots are not a health problem and will wear away.
- Show students how to do a titration. It may be a good idea to demonstrate one so they can see the entire process. This is not a discovery experiment from this perspective. It is intended to teach students the fundamental process of titrating.
- Because of the dark color of the permanganate, students will be able to read the buret only to the nearest 0.1 mL.

Likely Play-Out of Lab

Part I: The Standardization of Potassium Permanganate Solution

- Because of good reproducibility, students usually need to do only two titrations for standardization. Students will use about 22–25 mL of solution for a titration, depending on the concentration of the permanganate solution.
- You will probably have to help students with the calculations. They will have had them in lecture class but will not recognize the connection. The average molarity is approximately 0.1 M. (In 1995 the mean molarity for one section was 0.0989 ± 0.0008 M for 30 data points. The range for the set was 0.0910–0.1164 M.)
- Spend time checking the students' titrating techniques.
- The class data can be collected on a spreadsheet showing the mean and standard deviation. Set up the spreadsheet so that the students' data is collected in column A, the mean is calculated, and the deviations of each datum from the mean are printed in column B. The standard deviation is reported in a cell near the value for the mean.

Part II: The Hydrogen Peroxide Analysis

- Students will use about 35 mL of permanganate solution for each titration of 10 g H_2O_2.
- The percentage hydrogen peroxide is approximately 3%, depending on the age of the solution used. The percent concentration may vary among different brands or even different lots of the same brand.

Part III: Evaluating the Quality of the Data

Students should calculate the mean and standard deviation of their titrations by hand. Each student should enter their data into the class spreadsheet (or report their data to the instructor). Collection of the concentrations on a spreadsheet is easiest. The mean and standard deviation can be calculated by the computer, and copies can be printed for each student or posted. You may need to point out to the students that, for example, a concentration of 0.1014 ± 0.0088 M means that any concentration reported between 0.0926 M and 0.1102 M represents the same value. Students can then see that a high standard deviation results in a wider range and thus, a less precise concentration.

Possible Answers to Questions

1. **Would you say the team data set is <u>accurate</u>? <u>Precise</u>? Why?**
   Students should show that they understand the terms *accurate* and *precise* in their answers to this question. They will probably think the data set has both characteristics. To describe accuracy, they should compare the data to the concentration of the solution made (approximately 0.100 M) and what you tell them the concentration is. The precision of the data statement should relate to the size of the standard deviation and the relative standard deviation (RSD) values. Whatever they say, the support should be with values (not "close," etc.).

2. **Where do your individual results fall with respect to the mean of the team set? Does this information help you have more/less confidence in the accuracy of your own concentration value? Why?**
   The students' answers will depend on their data. Students should recognize that if their points are relatively close to the team mean, they should have more confidence than if the points are far away. The repeatability of their calculations should give them confidence in their titrating technique.

3. **Does the hydrogen peroxide concentration you determined for your individual sample fall within the range specified by HYPER Corporation? If not, what reasons could account for this?**
   Answers will vary. They should make sense relative to the values given. Reasons should revolve around technique (theirs or the Corporations') when making the product. Examples include the following:
   - overtitration (This would increase the concentration of hydrogen peroxide. If students overtitrated, their concentration should be higher than expected. Check to see if this is the case.)
   - inaccurate reading of the buret

   There are also reasons the students cannot control such as the following:
   - oxalate not completely dry

4. **Let's suppose that HYPER manufactures two H_2O_2 products: one containing 30% hydrogen peroxide by mass and the other containing 3.0% hydrogen peroxide by mass. You are responsible for quality assurance of the 30% line. Your analysis of a particular lot gives you a mean of 29.5% and standard deviation of 1.8%. A coworker whom you have been training is responsible for the 3.0% line. His analysis of a single lot gives a mean of 2.8% and an standard deviation of 0.4%. Whose analysis indicated the better precision? On what evidence do you base your choice?**
   30% line: RSD = (1.8/29.5) * 100 = 6%
   3.0% line: RSD = (0.4/3.0) * 100 = 14.3%

   The 30% line is more precise (RSD = 6%) than the 3.0% line (14.3% RSD). Students should recognize that even though the numbers are larger for the 30% line, it is the *relative* values of mean and standard deviation that make for greater precision.